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RETROSPECTIVE STUDY OF SELECTED DoD MATERIALS AND STRUCTURES RESEARCH AND DEVELOPMENT PROGRAMS
PHASE I: Case History Data Collection

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March 1979

Prepared for
Office of the Under Secretary of Defense for Research and Engineering

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Pertinent studies of the past, such as HINDSIGHT and TRACES, are reviewed along with the IR&D programs of DoD. Funding histories in materials and structures technology are presented. Some outstanding research programs (6.1 category) sponsored by the Navy, Army, and Air Force are traced from their initial concept to their eventual contribution to the technology base.

Selected case histories include the Navy's C4 Missile System program, which exemplifies the careful utilization of materials and structures technology advances by the designers. Other case histories discussed include the history and user acceptance of advanced fibers and composites as primary structural components; the development history of titanium and its uses; the Navy's development and sustained utilization of high-yield strength steels; the development of special aluminum alloys for aircraft and ships, and the growth of the graphitic (graphite and carbon-carbon) materials technology and their high temperature applications. The disappointing results of refractory metals for military applications are also discussed.

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The DoD recognizes a continuing need to examine and, if possible, improve its investment strategy rationale in all of its R&D. Based on a request of USDR&E(R&AT/ET), the Institute for Defense Analyses undertook a study of R&D planning in the limited area of materials and structures. The objective of the study is to determine if the current R&D planning procedures in materials and structures can be improved. This Phase I report is an orderly compilation of pertinent case histories. Evaluation and analysis will be the subject of a future Phase II paper.

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SUMMARY

This paper presents the results of Phase I of a two-phase study of past Department of Defense Research and Development programs in the areas of materials and structures. The objective of the overall program is to examine and to possibly improve DoD R&D investment strategy rationale. The materials and structures areas were selected for study because of their specific scope yet broad application base. Phase I is an orderly compilation of pertinent case histories that could be gathered within the manpower and time constraints of the study. This paper, therefore, is meant to be a nonanalytic source book that will be used in the preparation of the Phase II report.

This paper reviews related past studies such as "Project HINDSIGHT" and "TRACES" to take advantage of the work of others in the area of development planning. Funding trends were compiled in as much detail as was feasible so that the impact of program funding levels can be taken into account in the Phase II analysis. A comparison is drawn between industry's significant IR&D program funding and DoD funding for R&D.

Basic Military Science (6.1 category) programs presented are those submitted by each Service. Comments on "successful" 6.1 programs were requested from the Office of Naval Research (ONR) (including NRL), the Air Force Office of Scientific Research (AFOSR), and the Army Research Office (ARO). The broad field of subjects covered include material and structural behavior, chemistry of materials, and programs concerning metal alloys, ceramics, composites, etc.

The Navy's C4 Fleet Ballistic Missile (FBM) System development is covered in detail in regard to the material and structural innovations required to achieve the required performance. The information was obtained through personal contacts with personnel from the Strategic Systems Project Office (SSPO) and the Lockheed Missile and Space Company (LMSC). The history of the FBM series of missiles is traced from the early A1 Polaris system to the C4 Trident system with its sophisticated post-boost control system that was made possible through the advanced state of refractory metal technology.

The paper discusses the advancement of composite materials in the last 20 years with the availability of high performance fibers, such as Kevlar, graphite fibers, boron fibers, etc. The graphitic materials, missile-grade graphite and graphite fiber reinforced graphite (carbon-carbons), are discussed in the primary context of their use in strategic systems.

The long development of titanium is discussed from its days as a laboratory curiosity to its use on present-day aircraft and ships. The history is presented of DoD sponsored developments in steel and aluminum, necessary to make these materials compatible with special Service needs.

CONTENTS

I. INTRODUCTION AND OBJECTIVE

In February 1978, the Office of the Under Secretary of Defense for Research and Engineering (Research and Advanced Technology/Engineering Technology) requested the Institute for Defense Analyses to undertake a study of DoD materials and structures research and development planning. The DoD recognizes a continuing need to examine and, if possible, to improve its investment strategy rationale in all of its R&D. The limited area of materials and structures was picked for this task in order to provide a viable specific study and also because, with the exception of electronics, it is probably the technology area of widest application to weapons systems.

The broad objective of this task is to determine whether, and in what ways, the current R&D planning procedures in materials and structures can be improved. To be more specific, an historic retrospective examination is to be made of selected applications and programs to search for patterns that might lead to a better understanding of how future planning should be approached. The task is divided into two parts. In Phase I, the subject of the present paper, information and comments on selected case histories are gathered and compiled in an orderly way. Thus, this paper is meant to be a nonanalytic source book. In Phase II, which will be the subject of a future final paper, the data will be evaluated, refined, and expanded as necessary, and analyzed to search out rational and useful conclusions.

While the task may superficially appear to be a mini-HINDSIGHT (see Chapter III-A) study, its major objective is considerably different. HINDSIGHT was specifically meant to demonstrate that defense-sponsored R&D (excluding 6.1 research) was both militarily necessary and desirable from a return-on-investment standpoint. Perhaps not surprisingly, it accomplished both of these ends. It is not the intent, however, of the present task to determine whether or not the past materials and structures R&D monies was well spent. No statistical analysis of return-on-investment or any other kind of overall report card will be attempted. Rather, attention will be concentrated on motivational influences and the impacts (i.e., the actual use of results in applications) of selected R&D programs. By thus considering those programs that can generally be considered "successful," it is hoped that guidelines might be derived that could enhance the "success" of future programs. These, of course, will frequently go beyond the area of pure technical planning and encounter facets of management and technology transfer.

II. SCOPE AND ORGANIZATION

The present (Phase I) portion of the task entails the collection of information relating to past materials and structures R&D programs. The study is a retrospective examination of such programs emphasizing origins of technology, stimuli that began the programs, whether alternate R&D paths were (or should have been) followed, and the impact on military systems, whether or not they were the planned applications. While the major interest is not on whether the R&D monies were efficiently used (i.e., a return-on-investment report card), financial data are included wherever possible. In any case, overall funding patterns are shown as far back as available.

It should be reemphasized that this paper (for Phase I) is meant only as a source book, a compilation of gathered data and information. All evaluation, analyses, and conclusions will be the subject of a future paper (Phase II) of the study. Brief discussions, however, are given in the present paper of some previous retrospective studies (and their conclusions) of technology events; the best known of these prior studies are HINDSIGHT (Department of Defense) and TRACES (National Science Foundation) which are summarized in Chapters III-A and III-B. Also, Chapter III-C contains abstracts of opinions stated (in previous studies or communications) concerning areas of obvious concern to the present study, such as information dissemination and technology transfer.

In any study, such as this, there is always a problem about how to handle basic research, known as the 6.1 Category in DoD parlance. While this category, is not totally undirected, it is defined as "scientific study ... related to long-term national security needs." As a practical matter, it becomes difficult to find any research area that somehow does not fit this definition. Thus, efforts to sharpen its visibility and planning, regarding useful DoD applications, have historically run into trouble. However, the issue cannot be completely ignored for the present study since, in the last few years, the annual funding of the Military Service 6.1 materials and structures programs has been comparatively large, about 25 percent of the total materials and structures technology base. For this reason, the authors requested each of the Service research agencies (Office of Naval Research including the Naval Research Laboratory, Air Force Office of Scientific Research, and the Army Research Office) to compile a representative list of its past materials and structures projects which resulted in practical military applications. The results are summarized in Chapter V.

Within the resource limitations of the IDA study (a total of about one man-year has been spent on Phase I), it was clearly impossible to attempt an historical review of all areas of DoD materials and structures R&D, and thus highly restricted selections had to be made. Two general approaches are possible: one is to make detailed analyses of the newer weapons systems that are deployed or in engineering development and search for materials and structures advances that contributed to making the system better than its predecessor system (this was the HINDSIGHT approach); the other is to examine generic advances in materials and structures technology and attempt to follow the R&D process into its various applications.

The authors adopted a combination of these approaches (which inevitably overlap). One weapon system was examined, the TRIDENT I C-4 missile, with collateral attention being paid to the MINUTEMAN missile developments. This system was picked because it appeared, *ab initio*, critically dependent upon materials and structures technology for its performance. In addition, data and information were gathered on the following generic classes of materials and structures:

1. Advanced composites and fibers
2. Missile-grade graphites
3. Carbon-carbon composites
4. Titanium development
5. High-yield strength steels for Navy use
6. Aluminum alloys
7. Navy R&D on molybdenum and other refractory metals

These areas are not mutually exclusive, of course, and generally overlap the technology advances of the three weapons systems evaluated. This overlap is unavoidable and the text contains cross-referencing where appropriate. Also, it is clear that many important areas of materials and structures have been left out, such as lightweight armor, coatings, lubricants, ceramic radomes, and windows, etc. Thus, in Chapter VI, Section I, entitled "Miscellaneous Information on Materials and Systems," is included to at least mention some of the ignored items.

III. PAST STUDIES

Except for those documents that can best be described as serving public relations or salesmanship needs (e.g., the celebration of the fiftieth anniversary of some organization or other), there have been relatively few critical historical examinations of how national agencies spend their R&D dollars and how technological advances originated. The best known, and certainly the largest, such study is the DoD HINDSIGHT project, started in 1965 as an in-house effort after a trial contract study (by Arthur D. Little) of a single system, the BULLPUP. Early results of HINDSIGHT apparently indicated that, over the 18 years or so that was considered, few if any 6.1 research results had any impact on weapons systems. ["What we have not been able to do is to demonstrate value for undirected science."] The HINDSIGHT report (Ref. 1) pointed out that its time frame was too short to make this observation meaningful. Partly as a result of this, the National Science Foundation (NSF) contracted a study called Technology in Retrospect and Critical Events in Science (TRACES) that was meant to illustrate the practical importance of undirected basic research. These two studies are briefly reviewed in the following Sections A and B. Section C delves into a variety of pertinent comments from other studies, both published and unpublished.

A review of selected Office of Naval Research projects is given in an early IDA paper by Salkowitz, Armstrong, and Howe, 1970 (Ref. 2). However, since this paper pertains more directly to the present study, it is included later in Chapter V.

A. THE HINDSIGHT STUDY

In July 1965, HINDSIGHT, the voluminous DoD in-house study of research and technology utilization in weapons systems, was initiated by the Director of Defense Research and Engineering, Dr. Harold Brown. Twenty weapons systems (something less than one-half of the inventory) were examined in detail to identify those research and exploratory development (RXD) events that were direct contributors to the technical success of each system as compared to its predecessor system. During the life of the task, some 710 RXD events were identified, most occurring from 1945 to 1963. It was estimated that this number represented between one-third and one-half of the events that might have been identified by a more exhaustive analysis. An attempt was then made to trace each RXD event to discuss the persons involved, their place and method of operation, the type and amount of funding involved, etc. A total of 1975 individuals were so identified. Detailed background information was gathered on 511 of them (which was further used to see if any common motivational threads could be perceived).

Unlike the present study, the primary purpose of HINDSIGHT was to see if the overall R&D expenditures of the DoD were both necessary (in magnitude) and wisely spent. To quote from the 1965 Harold Brown memorandum to the Services:

> "I see no other way of satisfying the Congress and the Secretary of Defense that we are getting our money's worth for RXD. Failure to satisfy these questions will almost certainly have serious consequences to those Program Categories."

Indeed, the HINDSIGHT results did indicate that some 85 percent of the utilized new scientific and technological information was directly financed by the Military Departments while the bulk of the remaining 15 percent was paid for by defense-oriented industries, probably in the form of Independent Research and Development (IR&D).

Despite the difference in objectives, many of the HINDSIGHT observations and findings still have pertinence to the present materials and structures study. Some of these are loosely paraphrased below, simply arranged in the order of appearance in the HINDSIGHT report. For the reader interested in more detail, the page number in the report is given (in parentheses) after each item.

1. "Less than 2 percent of the identified RXD events classified as research [as opposed to XD] appear to have come about because the performer was interested primarily in extending the bounds of knowledge. Most research in science whose results were found to have been used in weapon systems was undertaken as part of an organized program associated with an identifiable generic problem rather than some vague possible utility of the result." (40)

2. "The engineer appears to rely heavily on tabulated scientific information published in engineering data handbooks and text references. He does not seem concerned with the details of the underlying theory, and typically uses results that have been condensed and presented in easily handled tabular or graphical form. Thus, it is suggested that more new scientific findings would be used in weapon systems if research managers were more concerned about the form in which the new information is made available." (41)

3. "In 27 percent of the RXD Events, the identified objective of the research effort was the satisfaction of a generic technological requirement. In a few cases, particularly when the Event involved a materials or a fabrication process, the results of the research were found to have been applied to materials or applications other than those considered by the

research performer. For example, the vacuum-arc-smelting techniques developed for titanium and uranium were applied almost directly to steel production, and the plasma-deposition techniques developed for metal plating found use in the depositing of refractory materials." (43)

4. "The dominant communications path for the successful transfer of new technological information has been informal person-to-person contact. Formal papers presented at meetings typically fail to include really useful information. Perhaps because of this, one of the most used sources of information for problem solving was the category of text books and hand books even though they tend to be two to five years or more behind the state-of-the-art." (49) See Item 2 above.

5. "For practical purposes, it is impossible to apply regimented cost-effectiveness measures to most military-supported technological research planning. Generally, the synergistic consequence of hundreds of identifiable advances is the basis for a markedly superior weapon system, rather than one, two, or three important scientific or technological contributions. The real measure of the effectiveness of research management is the degree to which new knowledge is available when needed. Of the RXD Events identified, 67 percent of the requisite new knowledge was actually available before it was needed. If judged at that time, such RXD results would not have been considered cost-effective." (55)

6. "The development or application of new materials was the subject of slightly over 30 percent of the RXD Events. Of those, about three percent actually resulted in the creation of a new material. The remaining 97 percent dealt with the exploitation of a new material's characteristics (a) to permit the use of a new manufacturing process (13 percent), (b) to enable the development of a new device (16 percent), or (c) to advantageously replace older materials for reasons other than manufacturing economy (68 percent). For example, the last category

might include the replacement of germanium by silicon in solid-state devices, or the use of aluminum instead of steel in the carriage of howitzer. <u>In general, this finding clearly demonstrates the importance of materials research in the advancing of technological capability</u>. The distributions within this 30 percent of Events, however, suggest that efforts devoted to gaining a complete understanding of all characteristics of a new material and to ensuring that this knowledge was widely distributed throughout the engineering community would be more useful than attempts merely to seek another somehow-improved material." (60)

7. "Inventions, or concepts, involving approaches to system or subsystem design were the bases for only about four percent of the Events. This suggests that fundamental design concepts did not differ greatly from those on which other existing military equipment of lower performance were based. The increased performance of a new weapon system appeared to emerge from the selection and integration of many innovations from diverse technologies." (61) [Author's note: This result is surprising at first blush. It appears to indict the designers for lack of innovativeness. It might also, of course, be just a result of an inherent conservatism in the military bureaucracy.]

8. "The findings as to motivation of performers form a consistent pattern and consequently offer some guidance to management. They suggest that usable ideas are more likely to be produced if an organization deliberately establishes a reputation for skill in a limited number of problem-associated technologies instead of diversifying its talents over a wide range of technical fields." (65) "Most of the scientific and technical ingenuity and innovation leading to modern weapon systems is found in a very conservative, almost routine environment. The persons credited with the advances give evidence of extreme conservatism. They tend to remain in the technical area in which they were educated, to stay on the same job with

the same employer. They even tend to have more stable family lives. To a considerable degree, this may explain why comparatively few of the technological Events were found to be based on the results of recent scientific advances. For is it likely that individuals with such stable and conservative traits would delve into what is, to them, the unknown for the solution to a problem? Certainly not, if such a digression could be avoided." (92)

9. "Well over half the identified technological advances clearly occurred in the absence of new scientific ideas. Almost all, however, depended on the existence of new information on characteristics of materials or the operation of devices such as the transitor. The class of research that leads to new materials, to measurements of those materials' characteristics, and eventually to the publication of pertinent data has been of primary value." (98)

10. "In retrospect, the tremendous value of the NAVAHO missile development is recognized, even though it never became operational. Studies of the technological basis for modern missiles, aircraft, and nuclear submarines almost invariably find their way back to the NAVAHO program. Inertial navigation and guidance systems, stellar navigation systems, flightborne digital computers, liquid rocket engines--all have been traced to work identified as originally done for NAVAHO. This example and numerous others that have been identified through Project HINDSIGHT studies indicate the wisdom of undertaking system-development projects in a conscious attempt to focus exploratory development on programs of research in technology. Maximum freedom should be granted the project director to seek advanced technical solutions, and at the outset there should be only a limited definition of a production objective--preferably none." (104)

It should also be mentioned that the study specifically did not investigate failures. Partly, this arises from the difficulty of definition. As the report says, on page 129, "research is undertaken in the quest for knowledge and the disproof of one hypothesis may be just as important as the proof of another. Thus, perhaps, only inconclusive research may be classed as a failure. But the difficulty of discriminating between inconclusive and incomplete research is a delicate matter that is well beyond the scope of the teams identifying RXD events."

1. Systems Studied in HINDSIGHT

3.1 HOUND DOG
3.2 BULLPUP
3.3 POLARIS A-1
3.4 MINUTEMAN I and II
3.5 SERGEANT
3.6 LANCE
3.7 Torpedoes Mark 46 Mod 0 and Mark 46 Mod 1
3.8 105mm Howitzer M-102
3.9 FADAC
3.10 AN/SPS-48 Radar
3.11 Mines Mark 56 and 57
3.12 Starlight Scope
3.13 Strategic Transport Aircraft C-141A
3.14 Navigation Satellite
3.15 Nuclear Warheads M-61 and M-63
3.16 152mm HEAT-MP Cartridge XM-409

2. Pertinent RXD Events to the Present Study

Table 1 lists those HINDSIGHT RXD Events that pertain to materials and structures technology. The full list is given in Ref. 1 (starting on page 151).

TABLE 1. SUMMARY OF RXD EVENTS

Serial Number	Title	Year (Termination)	Organization Type	Funds ($1,000)	RXD Type
0016	Diffused Nickel-Cadmium Coating for Corrosion Resistance of Low Alloy Steels	1951	Profit Laboratory, Industrial	55	XD
0017	Development of a New Coating Technique for Oxidation Protection of Nickel Base Superalloys in Gas Turbine Engines	1959	Profit Laboratory, Industrial	100	XD
0018	Fabrication of Compressor Weldment from Titanium Alloys	1955	Profit Laboratory, Industrial	265	XD
0020	Development of a Vacuum Induction Melting Process	1954	Profit Laboratory, Industrial	200	XD
0021	Development of the 6Al-4V Titanium Alloy	1951	Government Laboratory Nonprofit Laboratory, Independent Profit Laboratory, Industrial	40	XD
0022	Use of Titanium Alloys for Compressor Disks and Blades	1951	Profit Laboratory, Industrial	235	XD
0023	Use of Synthetic Rubber Viton A for Sealing Rings	1957	Profit Laboratory, Industrial	43	XD
0027	Investigation of Post Buckling Behavior of Heated Skin Panels	1964	Profit Laboratory, Industrial	35	XD
0028	Development of Statistical Mechanical Property Data for 7079 Aluminum Alloys	1962	Profit Laboratory, Industrial	100	XD
0029	Development of a Redundant Structure Analysis Method	1959	Profit Laboratory, Industrial	15	XD
0030	Development of a Fail-Safe Structure Analysis Analysis Method	1959	Profit Laboratory, Industrial	28	XD
0031	Concept, Development and Demonstration of Use of Ceramics and Resins for Tooling	1960	Government Laboratory Profit Laboratory, Industrial	192	XD
0032	Shot-Peening Prior to Chromium Plating of High Strength Steel	1957	Government Laboratory	4	XD
0045	Development of Temperature Grease Thickeners	1953	Government Laboratory Profit Laboratory, Industrial	50	XD
0049	Development of a Process for Producing High Reliability Soldered Connections in Missile and Space Systems	1957	Government Laboratory, Industrial	30	XD
0050	Application of Technique Using Eddy Currents for Determining the Material Strength of Liquid Propellant Tanks	1963	Profit Laboratory, Industrial	25	XD
0051	The Development and Demonstration of a Technique To Statistically Determine the Reliability of Missile Hardware	1964	Profit Laboratory, Industrial	6	XD
0052	Development and Demonstration of a Technique Molding the Butyl Seals for Propellant Tanks	1965	Profit Laboratory, Industrial	150	XD
0053	The Development and Demonstration of an Electron Beam Welding Technique Suitable for Use with Large Systems in a Production-Type Process	1965	Profit Laboratory, Industrial	150	XD
0082	Development of UDMH (Unsymmetrical Dimethylhydrazine) for a Storable Liquid Fuel	1953	Profit Laboratory, Industrial	69	XD
0104	Recognition and Investigation of the Catastrophic Nature of Hydrogen Contamination in Titanium Alloys	1954	Government Laboratory	50	XD
0106	Thiokol Based Integral Fuel Tank Sealing Compound	1946	Government Laboratory Profit Laboratories, Industrial	15	XD
0110	Full Scale Fatigue Test Requirements	1949	Government Laboratory	54	XD

(continued)

TABLE 1. SUMMARY OF RXD EVENTS (continued)

Serial Number	Title	Year (Termination)	Orgination Type	Funds ($1,000)	RXD Type
0113	Exploratory Development of Aircraft Window Stretched Acrylic Plastic Material	1953	Government Laboratory Profit Laboratory, Industrial	51	XD
0114	Adhesive Bonded Metal Sandwich Construction for Aircraft	1955	Government Laboratory	200	XD
0115	High Strength Welds in SAE 4340 Steel Used for Structural Components	1952	Governemnt Laboratory	80	XD
0116	Investigation of the Properties of Glass	1956	Government Laboratory	45	XD
0117	Jogging of Extruded Wing Panels	1962	Profit Laboratories, Industrial	75	XD
0118	Development of High Strength, High Resilience	1965	Profit Laboratory, Industrial	75	XD
0019	Development of Small Bend Radii in Aluminum Sheet by Compressive Roll Forming	1962	Profit Laboratory, Industrial	15	XD
0120	Development of Nondestructive Tests for Adhesive Bonded Structures	1956	Government Laboratory Nonprofit Laboratory, Independent	90	XD
0128	Development of Numerically Controlled Machine Tool	1952	Nonprofit Laboratory, University Operated	450	XD
0129	Development of Automatically Programmed Tools (APT) for Numerically Controlled Machining Operation	1959	Nonprofit Laboratory, University Operated	650	XD
0130	Development of Vinylidene Fluoride-Hexafluoropropylene Elastomers for High Temperature Fluid Resistant Seals	1956	Government Laboratory	5	XD
0131	Development of Titanium Reference Alloys in Analytical Techniques for Compositional Analysis of Titanium Metal	1953	Government Laboratory Profit Laboratory, Industrial Nonprofit Laboratory, Independent Nonprofit Laboratory, University-operated	105	R
0157	Development of a Low-Emission Film Coating for Dielectric Waveguide Windows	1962	Profit Laboratory, Industrial	290	R
0168	Development of a Magnetostrictive Video Storage Device	1955	Profit Laboratories, Industrial	400	XD
0180	Printed Circuit Switch Decks	1962	Government Laboratory	18	XD
0181	Dual Voltage Thermal Battery	1965	Government Laboratory Profit Laboratory, Industrial	30	XD
0188	Device for Determining the Occurrence of Stress Corrosion of Materials in Long Term Storage	Continuing	Government Laboratory	30	XD
0200	Development of an Economical Process for Producing a High Permeability Magnetic Material with a Rectangular Hysteresis Loop	1950	Government Laboratories	75	XD
0243	Protective Coating System for Missile Silos, Wing VI	1965	Government Laboratory	7	XD
0244	Microelectronics	1965	Government Laboratory Profit Laboratories, Industrial	4,000	XD
0245	Beryllium Spacer Assembly	Continuing	Profit Laboratory, Industrial	571	XD
0246	Development of High Strength Glass Fiber Reinforcements (AF-994), for Structural Plastics	1961	Government Laboratory	180	XD

(continued)

TABLE 1. SUMMARY OF RXD EVENTS (continued)

Serial Number	Title	Year (Termination)	Organization Type	Funds ($1,000)	RXD Type
0249	Development of Microminiature Multilayer	1962	Profit Laboratory, Industrial	211	XD
0250	Development of Production Processes for Ceramic Printed Circuits	1962	Profit Laboratory, Industrial	100	XD
0251	Beryllium Isopressings for Stable Elements	1965	Profit Laboratory, Industrial	379	XD
0258	The Use of LASER To Simulate Transient Radiation Effects in Semiconductor Devices	Continuing	Profit Laboratory, Industrial	16	XD
0259	Electrochemical Machining of Beryllium	1965	Profit Laboratories, Industrial	40	XD
0282	Investigation of Materials for Solid Delay Line	1944	Government Laboratory, University Operated	10	XD
0284	Development of Indium Bond Techniques for Attaching Quartz Crystals to Fused Quartz	1949	Government Laboratories	20	XD
0291	Development of a Welding Technique for Attachment of Integrated Circuits	1963	Profit Laboratory, Industrial	200	XD
0292	Use of Sintered Copper Tungsten as Electrodes for Electrical Surge Arrestors	1963	Profit Laboratory, Industrial	100	XD
0297	Development of Solid-State Computer Circuitry	1957	Profit Laboratory, Industrial	2,000	XD
0299	The Conception and Invention of Etched (Printed) Circuits	1949	Government Laboratory	8	XD
0300	Development of a Plated Interconnection for Circuits Boards	1957	Profit Laboratory, Industrial	75	XD
0304	The Invention and Demonstration of Electron Beam Welding	1956	Profit Laboratory, Industrial	3	XD
0316	Conception and Feasibility Demonstration of a Simple Tester for Nonmagnetic Materials	1969	Government Laboratory	0.5	XD
0326	Development of Improved Grown Junction Techniques and Their Application to a Silicon Material	1957	Profit Laboratory, Industrial	160	AD
0327	Development of Alloy Junction Transistors	1956	Profit Laboratory, Industrial	270	AD
0328	Development of Fast Switching Diodes	1957	Profit Laboratory, Industrial	90	AD
0329	Development of the Surface Barrier Transistor	1957	Profit Laboratory, Industrial	120	AD
0330	Development of Silicon Diffused Rectifier	1956	Profit Laboratory, Industrial	60	AD
0331	Development of Silicon Voltage Regulator Diodes	1957	Profit Laboratory, Industrial	75	AD
0332	Development of the Alloy Junction Power Transistor	1957	Profit Laboratory, Industrial	200	AD
0394	Development of a Lightweight Ultrastable Crystal Oscillator	1961	Government Laboratory, University Operated	50	AD
0412	Development of a Radioisotope Power Supply for Satellites	1961	Government Laboratory, University Operated	300	XD
0419	Development of Materials for Disc Memory	1958	Profit Laboratory, Industrial	50	XD
0420	The Demonstration of Improved Fragmentation for Steel Shell Through Incorporation of Carbide Network	1961	Nonprofit Laboratory,	40	R
0421	The Demonstration of Suitability of AISI 52100 Steel for Manufacture of Large-Caliber Forged Shell with Improved Fragmentation	1964	Nonprofit Laboratory, University Operated Government Laboratory	107	XD
0438	Development of Ceramic Transducer Elements	1956	Profit Laboratory, Industrial		XD
0448	Development of Special Neoprene Compound with High Tear Strength for Sleeves of Swaged Fittings	1962	Government Laboratory	10	XD

(continued)

TABLE 1. SUMMARY OF RXD EVENTS (continued)

Serial Number	Title	Year (Termination)	Orgination Type	Funds ($1,000)	RXD Type
0451	Study of Plastics for Optimum Compatibility Electrolytes used in Electrochemical Timing Devices	1953	Government Laboratory	50	XD
0452	Development of Superior Antifouling Paint for Prevention of Marine Growths on Mine Cases	1956	Profit Laboratory, Industrial Government Laboratory	75	XD
0453	Development of a Technique for Nondestructive Determination of Structural Strength by Use of Differential Hydrostatic Pressure Test	1951	Government Laboratory	15	XD
3007	Experimental Investigation of the Compressive Yield Strength of Metals	1960	Profit Laboratory, Industrial	25	XD
3014	Theory, Development and Test of Fused Salt Battery	1945	Foreign Government Laboratory	49	R
3021	Development of Thick-Metal Deposition Technique	1953	Profit Laboratory, Industrial Government Laboratory	60	XD
3037	Development of a Technique for Plasma-Jet Deposition of Rockide	1964	Profit Laboratory, Industrial	163	XD
4019	Development of Consumable Electrode Vacuum Arc Melting Process for Forgeable Refractory Metals	1944	Profit Laboratory, Industrial	60	XD
4020	Development of Pyrolytic Graphite	1957	Profit Laboratory, Industrial	150	XD
4021	Invention of Composite Silver Infiltrated Porous Tungsten Rocket Nozzle	1960	Profit Laboratory, Industrial	30	XD
4022	Conception and Development of Filament-Wound Closed-End Pressure Vessel	1949	Profit Laboratory, Industrial	100	XD
4028	Prediction of Ablative Behavior and Flight Test of Quartz Heat Shield	1959	Profit Laboratory, Industrial	1,900	XD
4030	Discovery of the Principle of Ablative Cooling	1953	Government Laboratory	100	XD
4040	Conception and Demonstration of Integrated Semiconductor Circuits	1958	Profit Laboratory, Industrial	100	XD
4071	Development of Epitaxial Deposition of Semiconductor Material	1960	Profit Laboratory, Industrial	100	R
4075	Development of Propellant Additives to Inhibit Gun Tube Erosion	1958	Foreign Government Laboratory	--	XD
4078	Development of Autofrettage Swaging	1959	Government Laboratory	300	XD
4101	Development of Planar Transistor Process Technology	1955	Profit Laboratory, Industrial	10	XD
4106	Research on a Solid-State Amplifier (Transistor)	1948	Profit Laboratory, Industrial	1,000	R
4108	Development of Method for Growing High-Purity Single Crystals of Germanium	1950	Profit Laboratory, Industrial	200	XD
4109	Development of a Germanium Transistor with Alloyed Indium Junctions	1951	Profit Laboratory, Industrial	30	XD
4110	Demonstration of Zone Melting for Purification of Metals	1951	Profit Laboratory, Industrial	3	XD
4112	Development of Silicon Transistor	1954	Profit Laboratory, Industrial	78	XD
4113	Development of an Oxide Masking Process for Delineating Diffusion Regions on Silicon Transistors	1955	Profit Laboratory, Industrial	10	XD
4114	Research on Diffusion Techniques for Transistors	1956	Profit Laboratory, Industrial	300	R

(continued)

TABLE 1. SUMMARY OF RXD EVENTS (continued)

Serial Number	Title	Year (Termination)	Orgination Type	Funds ($1,000)	RXD Type
4115	Development of Thermo-Compression Bonding for Transistors	1956	Profit Laboratory, Industrial	30	R
4116	Development of Method for Levitation of a Floating Zone of Silicon	1953	Government Laboratory	50	XD
4117	Development of Molecular Electronics Amplifiers	1961	Profit Laboratory, Industrial	300	XD
4118	Conception and Demonstration of "Molecular Electronic" Integrated Circuits	1958	Profit Laboratory, Industrial	500	XD

B. THE TRACES STUDY

In the fall of 1967, the National Science Foundation contracted a $164,000 study to the Illinois Institute of Technology Research Institute (IITRI) entitled Technology in Retrospect and Critical Events in Science (TRACES). The final report (Ref. 3) was issued in December 1968. While such specific wording was carefully avoided in the final report, it seems obvious that the study was undertaken to refute the findings of HINDSIGHT that contribution to defense needs from basic research had been small.

The IITRI and NSF personnel were said to have come up initially with a list of some 20 recent technological innovations of wide importance from which they chose five for a detailed analysis. These five are described below in a direct quote from Ref. 3.

"1. <u>Magnetic Ferrites</u> - a class of materials, widely used in computer memories, telecommunications, and small electric motors, which developed out of a broad base of research activities before and after World War II.

2. <u>Video Tape Recorder</u> - a development whose origin extends back to basic work in magnetic materials, control systems, and mechnical design, but which culminated from an obvious need created by television and the convergence of research and development shortly thereafter.

3. The Oral Contraceptive Pill - an innovation of great economic and social importance, which resulted from a long history of nonmission research in physiology, hormones, and chemical synthesis.
4. The Electron Microscope - an instrument, based on knowledge gained from fundamental research in electron physics and wave mechanics, which has had very many applications throughout the scientific and technical world.
5. Matrix Isolation - a new and promising technique, already revolutionizing certain chemical processing industries, for the study of the mechanisms of chemical reactions. Matrix isolation is an example of a scientific technique, hardly visible to the non-specialist, yet of great significance because of its key contribution to technologies of direct economic importance."

As an example of the techniques used by the authors of Ref. 3, consider their treatment of magnetic ferrites. Recognizing that mission-oriented research was starting in the late 1940s and early 1950s in hard and soft ferrites for use in such application as motors, loudspeakers, TV and recording transformers, radar modulators, and computer storage devices, they attempted to trace the supporting non-mission-oriented research. The latter was broken into crystal chemistry, telecommunications, ceramic materials, and magnetic theory. Some of the items so identified were Maxwell's electromagnetic theory (1864), the Weiss theory of ferromagnetic domains (1907), Heisenberg's quantum mechnical theory of ferromagnetism (1928), Roentgen's discovery of X-rays (1895), the Bragg laws of X-ray diffraction (1915), Goldschmidt's classification of atomic radii (1926), Pauling's bond theory (1929), Van Vleck's theories on electrical and magnetic susceptibilities (1932), and many others. The authors concluded, of course, that all of these basic research results were essential to the ultimate development of useful ferrites and, thus, non-mission-oriented research must be supported.

Similar treatments were applied to the other four "technological innovations" selected in Ref. 3. In passing, some curiosity must be expressed as to the particular choices made. Even in 1967, two of the most revolutionizing technologies were the gas turbine engine (air transportation) and various semiconductor devices (electronics as a whole). The authors could have treated both by their techniques with considerable success. The jet engine could not have been developed without superalloys which, in turn, could be traced back to Sommerfeld's theory of metals (1928) and earlier quantum theories of matter. Similarly, the authors could have had a field day with semiconductors. Instead, they picked two items like the electron microscope and matrix isolation. These are just two of many tools (albeit useful tools) for advancing basic and applied research efforts. Despite some words in the report, they were not then, nor are they today, <u>instrumental</u> in advancing any major technologies or practical systems.

Little more need be said here about the TRACES study. From its five tracings, it notes that "these benefits to our society are the return, realized through innovation, on prior investment in non-mission research." It concludes that "innovations for the next generations depend on today's non-mission research." The report's major, and almost only, message is that undirected basic research is good. No useful guidelines are deduced (nor, perhaps, can they be) as to how much funding is necessary, who should get this funding, how should they go about getting it, and generally how should funding agency programs be organized to obtain optimal results.

C. OTHER PAST STUDIES

There have been, of course, a number of committee, panel, or individual reports that touch on retrospective aspects of specific technical areas. Many of these are outlined in Chapter VI. There also have been a few formal or informal written comments which deal with more general R&D problems such as technology transfer, information storage and retrieval, and the like. Such areas are of concern to the present study but do not fit into the later format of this paper. Therefore, they will be mentioned now in the form of excerpts that appear pertinent.

1. An Approach for Systematic Evaluation of Materials for Structural Application (Ref. 4)

"The present system by which the need for property data is foreseen, the data supplied, and used, has become seriously inefficient. Because there is no systematic materials evaluation conducted:

1. The period of time between development and utilization of new materials is excessive.
2. The critical attributes of new materials are often overlooked.
3. The critical attributes of established materials are sometimes overlooked when applied in new design situations.
4. The optimum materials which best meet the performance, fabrication, and cost parameters may not be selected."

"The Air Force Materials Laboratories was one of the groups that recognized early the growing seriousness of the problem. One manifestation was the difficulty in selecting those materials which warranted inclusion in their data acquisition program, and in determining which properties to measure. Those who produce materials, and designers who specify the materials have related problems."

"The formation of the Committee at the suggestion of the Department of Defense was an attempt to explore the nature and ramifications of the general problem and to recommend an approach for its solution."

"Materials engineers are continually confronted with the need to decide how materials should be evaluated. At other times, they are asked to decide the usefulness of a new test or of a variation of an old test. The only way these and other related questions can be answered is to have an intimate knowledge of the way the materials in question must perform in the application of concern."

"In addition, the timing to be employed in developing the required data is critical. If data are developed in great detail for specific materials too far in advance of projected applications, the risk of wasted effort is great. On the other hand, a new weapon application cannot use a new material unless sufficient data are in hand at the time of design. This presents a dilemma to those charged with advancing the state of the art for national defense. The problem is twofold:

1. Information is not readily available about how a new material may be employed to take advantage of its physical and mechanical properties in the context of the operating environment. Similarly, how best to evaluate the new material may not be appreciated.
2. Present guidelines are inadequate for defining the depth of evaluation (assessment of suitability) for a new material in advance of specific end application."

"With an appreciation of the sequential nature of the decisions involved in selecting and incorporating a material into a design, the nature of the overall problem was clarified. The user of the data, the provider of the data, and the producer of the material have different interests and concerns."

Conclusions (from Ref. 4)

"1. The Committee concurred that the present manner of dealing with the materials evaluation problem is increasingly inefficient. An improved procedure could enhance the likelihood that optimum materials are, in fact, selected, and could probably also save money by reducing needless testing and by decreasing the likelihood of promoting new materials which will later be found to be unsuitable."

"2. Attempts to determine meaningful material evaluation trends or patterns from the several case histories developed in depth early in the life of the Committee were unproductive."

"3. A potentially promising approach to criteria for a material evaluation technique appeared to lie in the use of failure analyses. The Committee was unable to develop this approach successfully because of the general lack of documentation concerning actual service failures and the factors existing at failure. This approach, nevertheless, may warrant further study."

"4. Examination of the suitability and availability of material evaluation test techniques confirmed the generally well-known problems concerned with the attempts to develop useful tests that correlate well with service experience. A survey and an evaluation of all available test techniques were beyond the scope of the Committee charter."

"5. The Committee concluded that the development of guidelines for material evaluation requires the analyses of numerous case histories for specific material applications."

"6. The need for the analysis and recording of data for numerous case histories caused the Committee to address itself to the feasibility of developing a computerized approach to the data handling."

"7. The Committee concluded that not only does a computerized approach appear feasible but that, if developed, it could be useful to the Government, the using industry, and the materials producers."

"8. The full development and utilization of a computerized approach to material evaluation will require the ongoing development of a data bank relating material applications requirements and environments to specific components and systems. A second data bank containing information on the availability and adequacy of specific material test techniques will also be valuable."

"9. The present status of Government programs in support of materials information data banks and other national technology information retrieval systems (such as the Mechanical Properties of Materials Center, the ThermoPhysical Properties Research Center, and the Plastics Technical Evaluation Center) should be reviewed to determine where useful areas of cooperative effort may exist in the development of software and data management."

2. Data Analysis Centers: Custodians of the Numerical Data of Science and Technology (Ref. 5)

"The expression 'need to know' has become almost a euphemistic phrase for denoting the requirements for operating in our potentially information-rich environment. Although luck, intuition, and hunches are likely to retain a place in many endeavors, the individual, organization, or the nation having the right information at the right time in the right place has at least an initial advantage over those without this information. Thus, efforts to upgrade the quality of information and to improve access to and control of information resources must parallel the production of raw information. There is, currently, a serious imbalance between these two activities. Production has been the hare and information synthesis the tortoise. In this race, the hare can win only if the tortoise does."

"The big problem we still face involves putting together this innumerable bit of intelligence into larger, more meaningful and useful patterns. So we must make commensurate efforts to bring our understanding and activities from the level of simply generating data to organizing them into an internally consistent set of an ensemble which process I wish to refer to as the 'synthesis of knowledge.'"

"While the nation's scientific and technical community starves from lack of evaluated information, it is being smothered by an overwhelming document birth rate with the associated emission of polluted information referred to as 'research results.' The nation spends large sums of money on its Research and Development only to waste it by ignoring its results. Information discovery is indeed futile in the absence of an adequate means of information recovery. Should we decide that there shall be continued research at any level of funding, then the evaluation and proper dissemination phase of the results of this research can hardly be questioned."

"The expenditure of public funds on basic and applied research is justified by Congress, to a major extent, for maintaining the nation's scientific and technological leadership and thus makes a significant impact on our economy and commerce. In recent years Congress has been increasingly critical of many areas of our research expenditures, and there have been negative attitudes toward support of research in the physical sciences, in general, at a time when such research should have been accelerated to assist in the solutions of the manyfold problems that we face. As research scientists, where did we go wrong?"

"It is suggested tha a major part of the answer to the above question lies in the fact that the results of our research often do not reach the end-user for whom they are intended. This implies a broken link or perhaps an unfilled gap in policies for funding research."

"To illustrate the point, let us look at a specific discipline, such as material science and, in particular, at data on the properties of materials. This is especially appropriate at this time because this is an area which has and will continue to have the most direct impact on national shortages that we are already faced with, such as 'energy' and 'basic materials,' which we will be hearing about more and more in the years to come. From the vantage point of a data evaluation center, one can state without detailed elaboration at this time, that the state of the numerical data on material properties in the world's primary literature is conflicting, confusing, and generally inaccessible in a form which is useful to the end-user: the scientist, engineer, designer, planner, decision maker, etc. Even the research sponsor and his reviewers are not fully abreast of it. Therefore, the millions of dollars of research investment in public funds do not fully benefit the end-user as intended."

"Because sponsors of research have not recognized the importance of closing the gap between the generation of information and translating it into a form useful to the user, the *status quo* remains. Instead, we should see to it that research results are properly channeled to nourish our national anatomy. It suggests that this function is the full and unshirkable responsibility of the research sponsor and may not be relegated to some other agency. Evaluation, analysis, and synthesis of research results are part and parcel of research itself and not a separate function in the form of an afterthought."

"By virtue of its highly inadequate level of funding, NSRDS-NBS has not been able to make an adequate impact on the tremendous task ahead, considering the magnitude of accumulated backlog of the numerical data of science. Nevertheless, much useful ground-breaking has been achieved and selective projects have been activated on a priority basis. As a result of necessary fiscal constraints, however, the data of engineering and the applied sciences have not received the attention they deserve. On this point, the Information Analysis Centers (IACs) under DoD sponsorship, which are inherently technology-oriented, have equally fallen behind in their respective assigned missions as their funding level has essentially remained constant in recent years, while the cost of doing business has constantly increased. As a result, their effectiveness has decreased and the time will soon come when we will find our information resources totally inadequate to meet our national needs."

"In support of the various national efforts toward improved utilization of scientific and technical information, it is paramount, therefore, that universities take a double-pronged action: first, in the establishment of undergraduate curricula covering the effective use of accumulated engineering and scientific data; second, the incorporation of critical data analysis programs as an integral part of the research projects they undertake. Both of these activities are well within the charter of a university, namely, the responsibility of translating the often confused state of the results of research into a coherent information base readily usable by the nation's industry to improve production and advanced technology."

3. Headquarters, U.S. Air Force: Report of the Structures Technologies Panel (STP) (Ref. 6)

This was an in-house panel, set up by then Air Force Chief Scientist, Dr. James W. Mar, for the purpose of reviewing the posture of Air Force structures technologies. Much of the panel's report concerned the management organization of the Aeronautical Systems Division (ASD) and the various laboratories doing materials and structures work, specifically the Flight Dynamics Laboratory (AFFDL) and the Materials Laboratory (AFML). Thus, most of the panel's findings are not of interest here. Some of the comments, however, deserve quoting because they represent broad difficulties not confined to the

Air Force (in fact, it should be noted that the Air Force has subsequently alleviated many of these problems).

> "Some ... weaknesses ... are ... fragmentation of effort, divided responsibilities with key subjects falling between the chasms, ineffective transmission of ASD needs to the Laboratories and of Laboratory technology to ASD, a poor corporate memory.
>
> On the one hand, the Laboratories feel that ASD is too slow in using new technology, on the other hand, ASD feels that the methodology developed by the Laboratories is not suited to the mission of developing new airplanes.
>
> It seems to the STP that the use of Laboratory personnel on an ad hoc basis [to provide major consulting service to ASD] lacks an important principle; namely that the Laboratories bear none of the direct responsibility for their advice, i.e., ASD must decide whether or not to accept the advice.
>
> The separation of structures from materials as it exists between the AFFDL and AFML should be studied. The separation is an historic one and in certain disciplines, such as fracture and fatigue, separateness seriously impedes the inter-disciplinary approach which is required.
>
> The apportionment of the Air Force Office of Scientific Research (AFOSR) budget should, be critically examined in the light of today's world. For example, the 1972 OSR budget devotes $12 million to physics, chemistry and mathematics where as only $8.8 million is devoted to materials and mechanics .... The physicist, chemist and mathematician can obtain support from a number of Federal Agencies, whereas essentially all of the aeronautical scientist's support comes from DoD with only a minimal amount from NASA. [This argument sounds a bit weak to the present authors.]
>
> The STP firmly believes the U.S. Air Force and the nation need a national facility devoted to experimental structural mechanics .... The opportunity to develop expertise in aircraft fatigue design and

> development is not readily acquired by administering research contractors or by short-term consulting assignments.
>
> In recent years, new specification requirements such as fatigue control on the B-1 have been invented rather than developed. It would be advantageous to the Air Force to move cautiously in introducing new requirements. The STP strongly recommends that the Air Force test additional specification controls on an existing system and thus gain understanding of the possible impact before imposing the new specification. For example, the initial estimate of the impact of the fracture mechanics requirements on the B-1 was $29 million and 2900 pounds.
>
> The cut-back of resources for the materials information function (the Materials Information Centers) could be detrimental."

Concerning Manufacturing Technology, "a worthwhile change (in DoD Instructions 4200.15) would be to alter the current language, 'Its objective is to bridge the gap between prototype production and mass production ... 'to read' ... between demonstrated feasibility and quantity production ...'."

4. MAB Report on Research-Engineering Interactions

In January 1965, the Department of Defense requested the Materials Advisory Board (MAB) to undertake a study of research-engineering interactions. The study (Ref. 7) was to address three tasks.

1. Study representative examples of research-engineering interaction in order to extract common denominators, or clues resulting in success or failure.
2. Select and describe those successful principles or techniques most appropriate for adoption by the Department of Defense.
3. Recommend to DoD appropriate action for the enhancement of transferring the results of research to hardware.

The MAB appointed an Ad Hoc Committee on Principles of Research-Engineering Interaction, which included scientific and engineering representatives from industrial, nonprofit, and university organizations, as well as liaison members from DoD, Army, Navy, Air Force, NASA, and MAB itself, to conduct this study. Case histories were then recorded of selected critical events in the development of ten different materials including those of metallic, organic and inorganic, nonmetallic nature. The properties involved were electronic, mechanical, and chemical, primarily. Each subject was selected and its history written by a member of the Committee. During the study it was discovered that several of the cases turned out to consist of several subcases, and that a single research-engineering interaction (REI) might lead to the development of a number of diverging explorations. In other words, the starting point of the study "corresponds to the sprouting of a family tree of material developments and the subcases represent branches of important results from the original primary trunk."

Critical events occurred at various stages of the research and development process and could be organized into the following sequence.

- Initial scientific findings.
- Recognition of new material, or process possibility.
- Creation of the material or process.
- Demonstration of feasibility of use of the material or process in a specific equipment or product.
- Production.
- Operation.

In the various cases studied, a time span between 10 and 55 years was involved, with an average case including a time span of 25 years.

The findings dealt with the characteristics of the environment, the individuals, and the nature of the specific problem being studied. The findings were:

1. Flexibility of support in terms of allowance for changes in direction of attack or unanticipated exploration of a problem was critical to the final success of a program.
2. The achievements often required close and frequent communication between organizationally independent groups and were brought about by key individuals.
3. The critical events often involved individuals with postgraduate academic training but only infrequently did the events involve individuals whose principal interests were in basic research.
4. In several cases, an individual stood out in the achievement of success because of his extensive energies and single-mindedness in attaining the goal.
5. The recognition and thorough understanding of an important need by an individual significantly contributed to bringing about the research-engineering interactions.
6. The technical accomplishments were largely based on information and approaches that were available but had not been previously pursued.

On the basis of the experience gained in their study, the Committee made the following recommendations.

1. DoD organizations and others would find helpful to understand successful research-engineering accomplishments by a specific pattern of questions dealing with the flexibility of all parts of a program, the communications inside and outside of a program, the role of individuals in it, and the definition of the needs within a program.

2. The method of case history analysis in this subject area is useful, especially to the managers and participants in research and engineering programs.
3. A broader attack on these problems, even involving the case history approach, should be considered. Much might be learned by studying subject areas that never reached practical fruition.

Bid and Proposal costs are the costs incurred in preparing bids for contracts or in submitting unsolicited proposals to the government; these costs too can be charged against government contracts. B&P includes both the administrative costs of proposal preparation and the costs of developing supporting technical data to respond to government specifications. In practice, the latter costs are indistinguishable from those for IR&D and, for most purposes, IR&D and B&P can be considered together. The Defense Department has recognized this overlap by setting up similar rules in its procurement regulations for the two categories. In this paper we shall refer mainly to IR&D, with the understanding that the discussion is applicable to a large part of the B&P program also.

In 1974, the major defense contractors recovered $808 million from the Defense Department for IR&D/B&P costs and an additional amount, estimated at about $200 million, was paid to smaller contractors. But this money did not appear in DoD budget under the heading "Research, Development, Test and Evaluation" (RDT&E), nor were any specific results of the program listed in the Director of Defense Research and Engineering's (DDR&E's) annual report to Congress. Because DoD's share of IR&D/B&P costs is paid through overhead charges on defense contracts, the program is not identified in the budget. Most of the money for IR&D comes from the procurement section of the budget, with the RDT&E budget contributing only the proportion which is charged via overhead to R&D contracts. This anomalous situation is an important factor in explaining the relative obscurity of the IR&D program. Each year Congressional committees hold extensive hearings on the RDT&E budget of the Pentagon, yet the IR&D program was virtually unknown to the Congress until Senator Proxmire first focused attention on it in 1969. At that time, Senator McIntyre, Chairman of the Subcommittee on Research and Development of the Senate Armed

Services Committee, admitted on the floor of the Senate that he had never before heard of IR&D.

The following year, Congress held hearings on IR&D and passed legislation mandating certain procedures for DoD's management of the IR&D/B&P program. The Commission on Government Procurement took up the question, and the General Accounting Office (GAO) has made a series of studies on the implementation of the 1970 law. In September 1975, the Subcommittee on Research and Development, Senate Armed Services Committee, and the Subcommittee on Priorities and Economy in Government, Joint Economic Committee, held three days of hearings on the IR&D program.

Throughout the hearings the defense contractors have strongly supported IR&D as a normal business practice, one whose costs should be recovered from the Defense Department as from any other customer. They argue that their IR&D programs provide incentives for innovative work and a flexibility that are lacking in contract R&D. The Pentagon also supports the program, extolling it as a way of encouraging new ideas, increasing competition in the defense industry, and maintaining a healthy technology base at a relatively low cost to the government. Critics, however, claim that the IR&D program is unnecessary, given the billions spent for contract R&D each year by the Defense Department. They argue that there is no evidence that the benefits of the program have been commensurate with its costs; indeed, it is described as a virtual subsidy to the defense industry. Some companies, it is charged, have used IR&D funds to gain a technological advantage in their nondefense undertakings, or to cover cost overruns on defense contracts. But the chief criticism has been leveled at the fact that the program has been funded outside the regular RDT&E budget of the Defense Department without Congressional oversight.

Although the dollar amounts reimbursed by the Defense Department for IR&D programs appear small relative to a $100 billion defense budget, they are substantial compared to other categories of government spending. For example, funds for IR&D/B&P have exceeded the total National Science Foundation budget for every year for which data are available. Within the defense budget, IR&D/B&P funds equal about 10 percent of the RDT&E budget, and about 40 percent of the funds allocated by DoD to its own in-house laboratories. DoD payments for IR&D (not including B&P) are almost equal to the amounts spent in industry via RDT&E contracts for support of the defense technology base. Viewing these figures as an expression of a national science policy, we can ask whether this distribution of resources between different R&D programs is an optimal one.

The support of defense research and development work outside the RDT&E budget raises the question of accountability to Congress. At present, the size of the IR&D program is not subject to external budget review, but instead is the result of internal DoD decisions and negotiations with its major contractors. The only mechanisms for Congressional oversight in the program are the opportunity to ask questions during Congressional hearings on the defense budget and a requirement for an annual report on IR&D/B&P. The annual reports, however, are not very informative. They list the IR&D/B&P costs of the major defense contractors as a group and DoD's share of those costs, but there are not details on which companies receive the money and what they do with it. Nor does the annual report attempt to demonstrate the ways in which the program is responding to the formal goals set for it by DoD. The lack of this type of information effectively precludes the Congress from forming a valid judgment on the value of the IR&D program.

In addition to the question of Congressional oversight, there is also the question of the Pentagon's management of the

IR&D program. Over the years, the Defense Department had developed management procedures which are described as providing adequate control over the size and quality of IR&D programs in the individual firms without interfering in the ability of the firms to make independent decisions on the direction of their programs. These procedures include technical evaluation by DoD scientists of the IR&D programs of the major defense contractors, and the negotiation of ceilings on the dollar amounts which DoD will pay. The effectiveness of these measures, however, may be doubted. In practice DoD's evaluation of the technical quality of a company's proposed IR&D program has only a limited effect on the negotiations for its program ceiling, although such a link would seem to be a prerequisite of good management. Furthermore, there is still disagreement over some of the principles embodied in DoD's rules on IR&D. Should the Defense Department support only R&D which is relevant to the defense function or should it accept a broader definition of relevance? Should projects which are aimed at commercial markets be allowed? Are on-site reviews of IR&D programs indispensable to evaluation procedures or are they an expensive luxury which can be replaced by review of technical write-ups? How serious are the problems posed by what is politely called "creative accounting," i.e., the problem of companies classifying their costs in ways designed to enhance their cost recovery from the government? These questions are important because an evaluation of the IR&D program must consider not only the stated goals for the program but also whether or not it functions to fulfill those goals.

A final category of issues are those which may be grouped together as indirect effects on the IR&D program. The disbursement of large amounts of money, for certain activities only, and to certain firms only, inevitably has had effects on the internal structure of the firms, on the overall structure of the industry, and conceivably, on the defense posture of the United

States. IR&D projects represent an entry point into the weapons pipeline; indeed, hope of winning future contracts is the major incentive for firms doing IR&D. If, as some have argued, U.S. weapons decisions are often the result of a technological imperative rather than stemming from a rational analysis of military requirements, then IR&D represents an early step in the development of technological pressures. It is possible that the cumulative impact of these indirect effects in the areas of industry structure and strategic decisions are more important to our ultimate judgment of IR&D than the evaluation of the stated aims of the program, more narrowly defined.

1. DoD's Objectives for the IR&D Program (from Ref. 8)

The Defense Department has consistently supported the IR&D program as an important element in its total R&D program and in its relationships with military contractors. Formally, three objectives are listed for the IR&D program. First, IR&D is seen as a means of strengthening the technology base of the industry. The absence of tight DoD controls, it is argued, creates an environment conducive to real innovation. The IR&D program provides incentives to the defense firms to allocate resources to R&D in military-related areas and to explore a diversity of approaches to technical problems in addition to those chosen for funding through contract R&D. The Defense Department claims also that the IR&D program reduces the total costs of weapons procurement by allowing DoD to purchase already developed components "off the shelf."

A second related objective given for the IR&D program is to increase competition in the defense market by developing technical competence in more than one supplier for any specific requirement which the DoD may have. By this use of the word "competition" the Defense Department obviously does not intend the specialized meaning of the economist, who refers to a market characterized by many buyers and sellers, a homogeneous product, and free entry. The defense market deviates sharply from this ideal type but, by supporting more than one contractor in a technical area, the DoD hopes to gain some of the benefits of competition. In addition to the gains in innovation resulting from increased technological rivalry, an effect that properly belongs under the first objective, the presence of alternative sources of supply reduces the market power of individual contractors and protects the DoD from the risks of relying on a single firm. It may also result in lower prices, although, for a variety of reasons, price competition is not strong in the defense market.

The third objective listed by the DoD for the IR&D program views it as contributing "as appropriate" to the economic stability of the defense industry by encouraging diversification within the individual firm. Such diversification reduces the firm's vulnerability to shifts in procurement policy and, it is hoped, enhances the long-run health of the industry.

It should be noted that the objectives stated by DoD for the IR&D program are not wholly consistent. Encouraging firms to diversify may, in fact, lead to resources being pulled from the defense market. Maintaining alternative suppliers in specialized technical areas does not necessarily yield lower prices for defense procurement; the market may be shared according to some noncompetitive principle. Furthermore, the IR&D program is poorly designed to increase the numbers of alternative suppliers. Because IR&D costs are recovered through an overhead charge, the largest sums of money go to the largest, well-established defense contractors and to those technical areas for which there are already many contracts. Electronics is an obvious example: of the top 100 DoD prime contractors in Fiscal Year 1974, 19 were in the electronics industry, and other major contractors, particularly in the aircraft industry, also

IV. FUNDING TRENDS IN THE MATERIALS AND STRUCTURES TECHNOLOGY BASE

A difficulty in obtaining figures of DoD Technology Base Funding in materials and structures is that of definition. What is included, for example, in the term "materials?" There is no question about a structural metal alloy, but where do electronic materials end and electronic devices begin? Single crystal growth is clearly a materials problem, but what about epitaxial layer growth, photomasking and stripping, etc.? Hydraulic fluids and lubricants are normally considered "materials," but fuels for airbreathing engines or rockets are not. Similarly, for "structures R&D," fracture mechanics clearly qualifies but stress analysis and fatigue studies of an experimental gas turbine engine may well be categorized as "propulsion." Obviously, any definition will always be somewhat arbitrary.

Having said the above, and recognizing that the problem will continually cause some inconsistencies, this particular difficulty will now be ignored. In the overall, the total financial sums are large enough that differences of a few million dollars will not be significant. In any case, the most useful data for present purposes are the historical financial trends in the various categories and mission areas (aircraft, missiles, space, ships, ordnance, and basic research) rather than the absolute dollars.

It is impossible to obtain mission area funding patterns prior to FY 1971 for Materials and FY 1974 for Structures.

These years are the first times the respective Technology Coordinating Papers (TCPs) appeared for these two technology areas (Ref. 8). For the Materials area, an earlier estimate of total Federal R&D funding was made in August of 1971 by the Interagency Council for Materials (ICM) of the National Materials Advisory Board (NMAB). This survey (which apparently was never published in a formal fashion) breaks out the Military Service and DARPA Materials funding from FY 1966 through FY 1971 by type of effort, i.e., basic research, applied research, and experimental development that conform roughly to the 6.1, 6.2, and 6.3A Categories. No such survey seems to exist for the Structures area and it is nearly hopeless to try to even get total funding data prior to about FY 1972.

Figures 1 and 2 show the best estimates of the annual Materials and the annual Structures budgets for the fiscal years available. In each figure, the totals are broken down into the areas of Research (6.1), Exploratory Development (6.2), and Advanced Development (6.3A).* For Materials, the 6.1 plots include the appropriate DARPA funding, which historically runs about one-half of that of the combined Services (or about one-third of the 6.1 total). There is no DARPA funding that is unique to Structures Technology. Small discrepancies between the various sources of information have been glossed over in Figs. 1 and 2.

Figure 3 illustrates the historical trend in DoD total RDT&E budget for the 6.1, 6.2, and 6.3 Categories, both in actual dollars and in constant 1968 dollars. The Materials and the Structures budgets are also shown as percentages of the total; it can be seen that these percentages (especially for Materials) show a downward trend despite increases in each individual budget.

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*It is recognized that much equivalent R&D is performed directly under systems programs, but the present study considers only the identifiable Technology Base programs. Similarly, Manufacturing Technology programs are not considered.

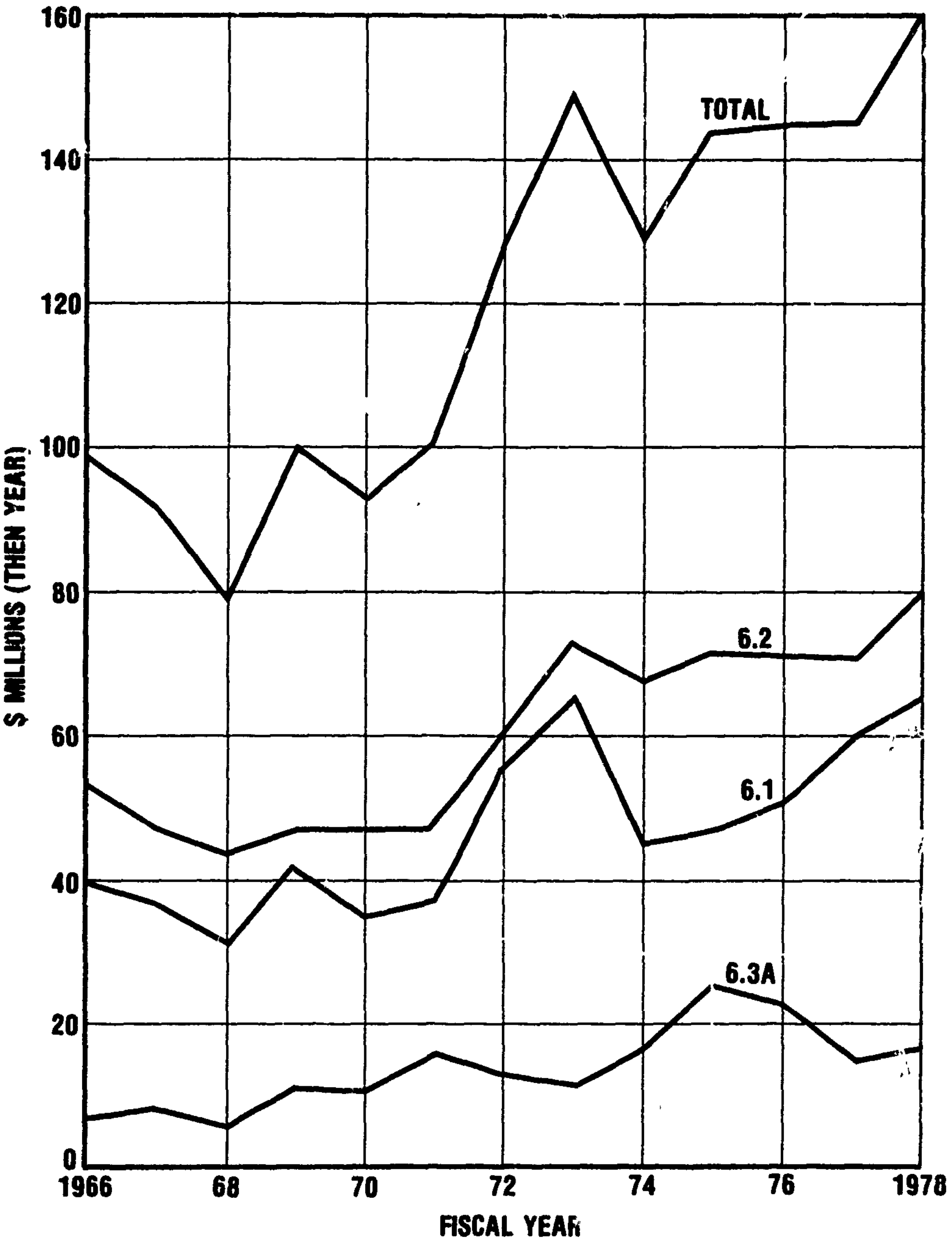

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FIGURE 1. DoD Materials R&D (estimated annual costs) (Data prior to 1972 are those estimated by the Interagency Council for Materials, see text)

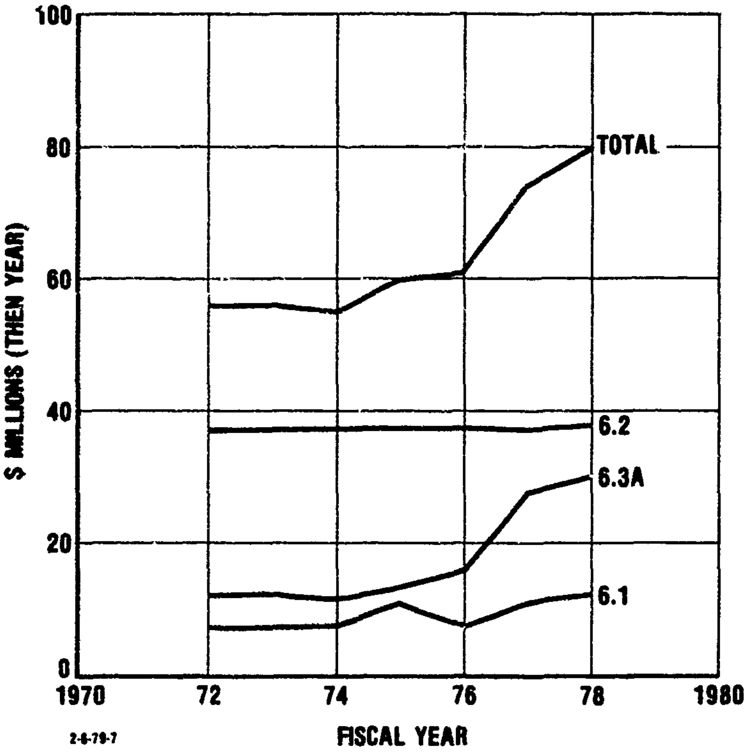

FIGURE 2. DoD Structures R&D (estimated annual costs)

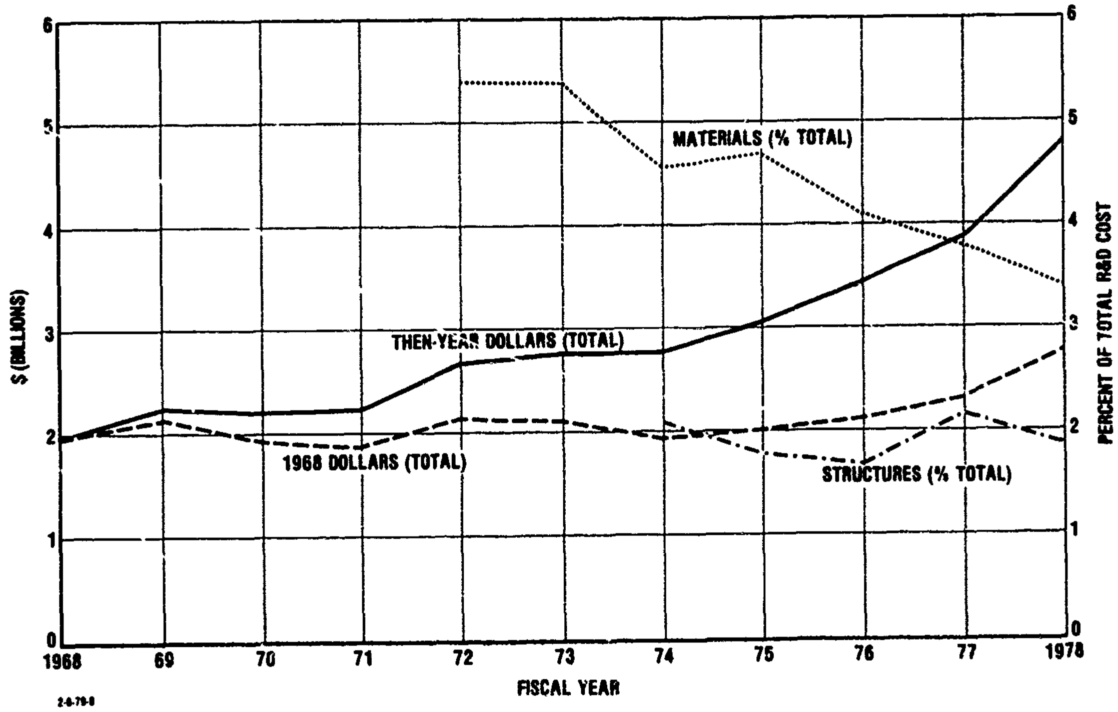

FIGURE 3. Expenditures for All DoD R&D in Categories 6.1 (Research, 6.2 (Exploratory Development), and 6.3 (Advanced Development)

Figure 3, of course, does not show a completely fair perspective since the entire 6.3 budget is included in the total, not just the 6.3A (now called "Advanced Technology Development"). Since FY 1974, the actual total figures are shown below (in millions of dollars). These, then, are the true percentages of the entire Technology Base Program devoted to Materials and Structures. Note that the trend is still down, although not as rapidly as indicated on Figure 3.

Category	FY 74	FY 75	FY 76	FY 77	FY 78
6.1	304.1	300.6	329.7	383.4	441.9
6.2	1062.4	1083.8	1184.4	1342.4	1506.0
6.3A	200.4	255.0	299.9	360.6	444.6
Total	1566.9	1639.4	1814.0	2086.4	2392.5
Materials and Structures, %	11.8	12.4	11.2	10.5	10.0

Figures 4 and 5 show the amount of 6.2 and 6.3A funding in Materials and in Structures devoted to each of the mission categories, Aircraft, Missiles, Space, Ships/Craft, and Ordnance/Vehicles. Ocean Engineering and Submarines are included under Ships/Craft. It should be noted that the area of Structures Technology has no authorized funding devoted to space missions.

Figure 6 indicates approximately the cumulative funds applied to the five technology areas shown. The authors are grateful to Col. H.S. Rubenstein (OUSDR&E/ET) for providing these data. Note that the total monies spent on these projects are about 20 to 25 percent of all monies spent on Materials and Structures from 1960 to 1978.

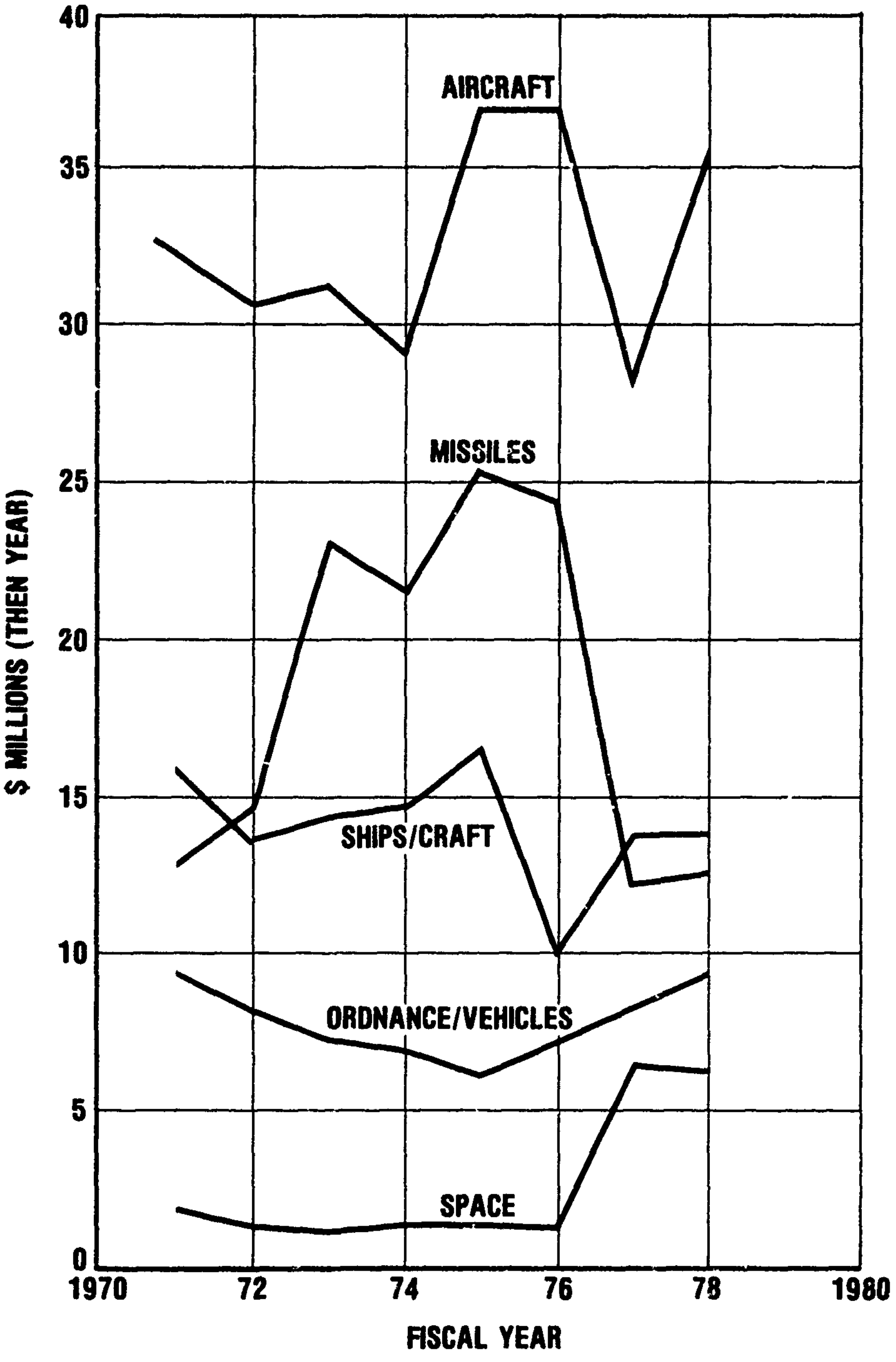

FIGURE 4. Materials 6.2 and 6.3A R&D by Mission Categories

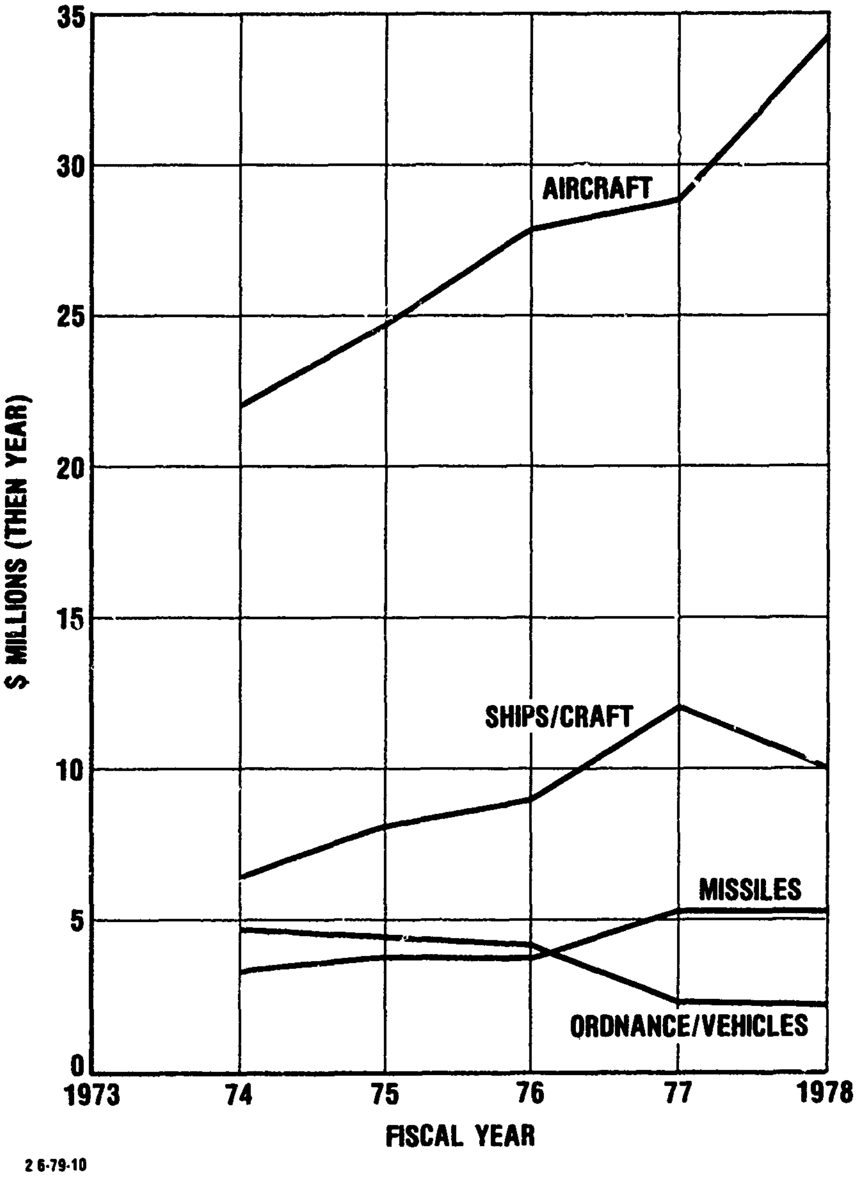

FIGURE 5. Structures 6.2 and 6.3A R&D by Mission Categories

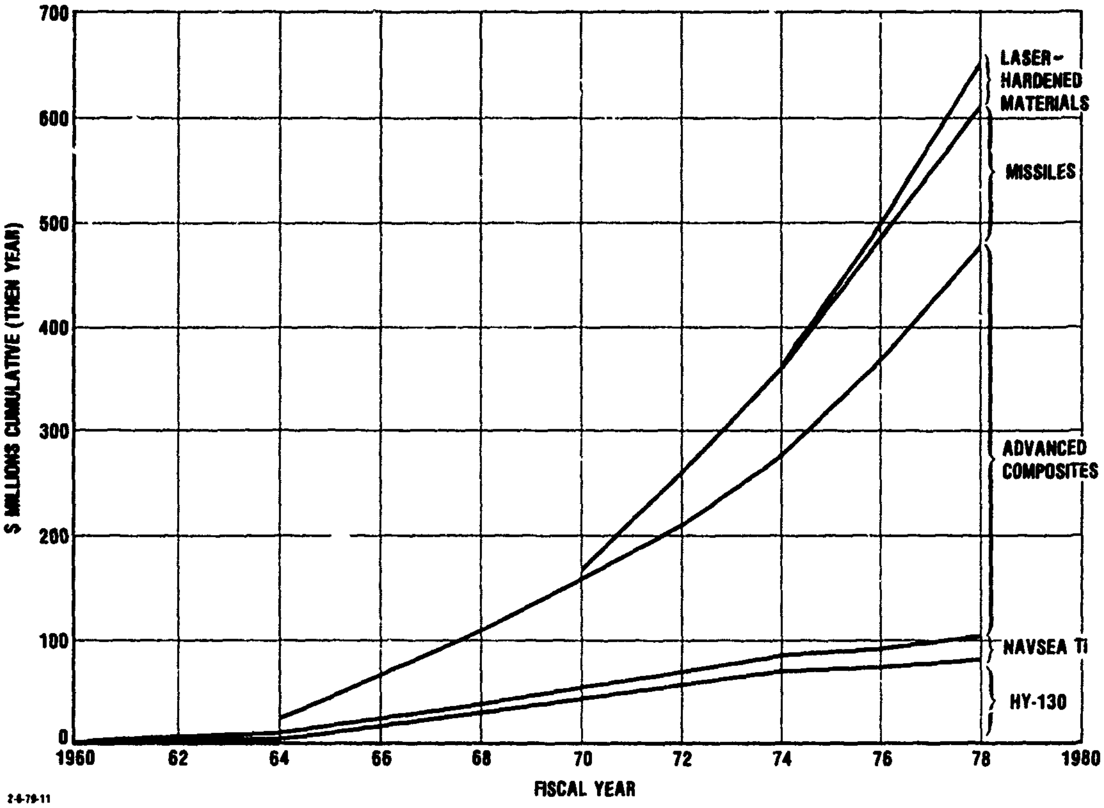

FIGURE 6. Estimate of Cumulative Costs in Materials and Structures R&D for Some Major Technologies (total cumulative Materials and Structures R&D costs over time period shown are approximately $3.1 billion). It should be noted that the NAVSEA Ti costs include expenditures on piping, pumps, etc. The cumulative (6.2) funds for submarine pressure hull R&D are about one-half the figures shown.

V. BASIC MILITARY SCIENCE (6.1 CATEGORY)

Earlier in this report, it was mentioned that it is generally difficult to examine long-range research (6.1) retrospectively by looking for its direct use in military applications. It is perhaps more profitable to seek successful 6.2 (exploratory) developments and then look back for 6.1 influences; this is attempted later in the report. The authors, nonetheless, desired to obtain some measure of "successful" 6.1 programs and requested pertinent comments directly from the Office of Naval Research (ONR), including the Naval Research Laboratory (NRL), the Air Force Office of Scientific Research (AFOSR), and the Army Research Office (ARO). The comments given below are from personal communications of E.J. Salkowitz (ONR), A.I. Schindler (NRL), L.C. Kravitz (AFOSR), and G. Mayer and J.C. Hurt (ARO).

A previous review of selected ONR-supported research should be mentioned. This was done by the Institute for Defense Analyses (IDA) in 1970 (Ref. 2). This study was the result of several meetings in the ARPA Materials Sciences Office. A list of some of the areas supported by ONR that were active for a number of years includes:

1. Molybdenum and other Refractory Metals
2. Boron Compounds and Inorganic Polymers
3. Cryogenics
4. Dislocations
5. Computers
6. Fracture
7. Magnetics
8. Propellants

9. Shock Tubes
10. Solidification and Crystal Preparation
11. Transformations
12. Surface Science
13. Field Emissions
14. Ceramics

To make the study tractable, the first four programs listed above were chosen. The study started with excellent goals and complete case histories of the few chosen topics were performed; however, toward the end of the study the effort lessened. The final recommendations made were as follows (considerably paraphrased).

1. As far as possible, uniform criteria for criticism and judgment of research programs should be clearly set forth.
2. Decision-making benchmarks should be established early in the research program.
3. Better procedures should be developed for deciding whether, and when, to terminate a program.
4. Internal DoD communication (concerning research needs) should be improved.
5. Utilize the advice of outside experts.
6. Improve communications between all parties doing research.

These recommendations sound much like rules set forth in any standard business text on research management and are unlikely to suggest any new guidelines for 6.1 managers. The details of Ref. 2, however, are of value to the present study and some are incorporated in the case studies of Chapter VI.

Simply for perspective, the approximate DoD 6.1 budget (FY 1979) officially categorized in Materials and Structures is shown below (in millions of dollars):

	Materials	Structures	Total
Navy	15.6	2.0	17.6
Air Force	4.3	5.1	9.4
Army	7.0	1.5	8.5
Sub-Total	26.9	8.6	35.5
DARPA	15.0	0	15.0
TOTAL	41.9	8.0	50.5

Additional related work (e.g., polymers, lubricants, electronic materials) would add another $23.0 million for a total of $73.5 million. At present, the Services manage their 6.1 monies somewhat differently. In the Air Force, all research projects (in-house or not) are approved and funded out of OSR. In the Navy, NRL gets about $5.5 million and the Naval Air Systems Command about $2.9 million, both of which are more or less outside the direct control of ONR. In the Army, the Army Materials and Mechanics Research Center gets about $2.2 million and the Benet Weapons Laboratory about $1.6 million without requiring the initial approval of ARO. Of course, ONR and ARO still maintain the function of coordinating all the 6.1 efforts in the two Services.

A. OFFICE OF NAVAL RESEARCH/NAVAL RESEARCH LABORATORY (ONR/NRL)

1. Structural Mechanics Program

Since the early 1960s the Structural Mechanics Program has been carrying out research on computerized sound radiation prediction models for submarines. These methods have been correlated successfully with controlled tests and have had the involvement of a team of contractors including Electric Boat. As a result of its participation in this program, Electric Boat utilized these techniques for the first time in the design and construction of the TRIDENT submarine. This submarine represents the first case where quieting techniques based on analytical considerations were carried out extensively in the design and analysis phase. Concentration in the ONR program was in the low frequency portion of the sound signature that radiates extensively into the far field and is an important factor in detection.

2. Submarine Shock Protection Techniques

Submarine shock protection techniques are currently undergoing extensive reassessment on the part of the U.S. Navy community. The major technological advances that relate to our ability to examine the effectiveness of our weapons against enemy submarines or the vulnerability of our submarines to enemy weapons have evolved as a result of ONR research. A simplified technique coming out of an ONR research program has enabled us to account for submarine hull-water interaction in a way that permits, for the first time, a tractable method for examination of the problem. The methods have been verified in both the elastic and plastic ranges by extensive correlations with controlled tests. It is also helping in the assessment of HY-130 as a follow-on next-generation submarine hull material.

3. Ferrographic Lube Oil Analysis

Naval Ship Engineering Center (NAVSEC) has used Ferrographic Lube Oil Analysis* on a production basis and is firmly convinced of its effectiveness in predicting mechanical failure. The mission of the Naval Ship Engineering Center is to perform engineering and material management functions for ship system, equipment, and material requirements in support of Naval Sea Systems Command. NAVSEA provides support of program requirements for ship design, system and equipment design, procurement, installation, material management, and maintenance engineering. NAVSEC considers Ferrographic Lube Oil Analysis a modern tool of maintenance engineering.

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*This was originally a NAVSEC development.

It is now well known that the material, size, and shape of particles of wear give an excellent indication of the wear condition. Ferrography is the only method available to NAVSEC to determine size and shape of these particles. Site analysts on the NAVSEC Ferrographic program can and do make maintenance recommendations to the engineering officers of ships in the program.

Sponsorship of further development of Ferrography at TransSonics by the Office of Naval Research led to a program by Naval Air Propulsion Center and Naval Air Engineering Center. It involved collecting a number of samples of oil from different types of equipment and having them analyzed at TransSonics. NAVSEC was asked to participate, and to provide periodic samples from main feed-pump motors from a number of ships. Results were so encouraging that in 1975, when this program was drawing to a conclusion, NAVSEC procured a set of equipment for one Intermediate Maintenance Activity (IMA) site and had personnel trained in analysis. It was decided to locate the analysis capability on-site where sampling could be controlled and feedback and follow-up with ship's operating and maintenance personnel would be facilitated. This on-site analysis capability is backed by engineering support at NAVSEC.

NAVSEC's maintenance engineering program has established ferrographic analysis at three intermediate maintenance activities, servicing three squadrons of SSBN ships, approximately ten ships per squadron. Each oil-lubricated equipment in the program is sampled on an average of once every three months, normally following the quarterly refit. Sixteen equipments periodically sampled include four main feed-pump motors, auxiliary diesel engine sump, main propulsion lube oil system, two ship service turbine generators, two refrigeration compressors, and the ship's air compressors. Air-conditioning compressor lube oil samples and the ship's hydraulic oil samples have recently been added to the program. An occasional grease sample is analyzed if it can be obtained when a bearing is being changed. Analysis and follow-up at one Intermediate Maintenance Activity (IMA) site, of approximately 160 samples per quarter, require about one-third of the trained analyst's work time. It has been found to be well worth the time and expense.

4. Coatings Research

NRL has had a coatings research group for many years. During the height of the submarine atmosphere program at the Naval Research Laboratory, several efforts were undertaken to develop new paints. NRL, for example, developed a paint that could be applied to the hull of a submarine while the hull was wet. This effort was designed specifically to answer the need to paint some of the submarines while they were docked in Holy Loch. The most recent development in the coatings area, that of the fluorourethane paints, was considered as a hull paint. There were six test patches evaluated on the USS SANDLANCE (SSN660).

5. Oxygen Generation Research

NRL has several programs dealing with oxygen generation. These range from various approaches to the electrolysis of water to produce hydrogen and oxygen, and improvements in this system, to the present classified study on what to do with some of the undesirable products produced by this approach. In addition, NRL's inorganic programs have looked very carefully at the problem of improvements in chlorate candles that are now used on submarines, and has made detailed comparisons of the various sources of oxygen and potential sources of oxygen that are now being developed in the inorganic 6.1 research program. The most recent question came from Admiral Wilson who wanted a comparison between the potential usage of potassium superoxide and the chlorate candles. NRL, because of its 6.1 research program in inorganic chemistry, was able to draw on this expertise and make a rapid authoritative comparison of the systems in answer to Admiral Wilson's question. Not all of NRL 6.1 research activities transfer to the fleet. For example, there were two research concepts that would have combined carbon dioxide removal with oxygen generation. One was the sulfate cycle and the other was the use of algae. The sulfate cycle system turned out to be sound in scientific principle but too complicated from an engineering point of view for a shipboard system. Similarly, the algae study turned out to need too much power from the light to be successfully used at sea. Nonetheless, it is new thrusts and new activities of the research program that do lead to new breakthroughs and new achievements in many areas of the fleet, including the submarine.

6. Research on Fuels Used on Submarines

NRL's basic fuel and combustion program has been applied to several problems in the submarine fleet. Only two will be cited here. Dr. Enoch Johnson headed a multilaboratory task force on the study of the use of Otto* fuel when it was first suggested that it be used for submarine torpedoes. His work led to the development, in conjunction with another laboratory, of a detector for the Otto fuel. Dr. Johnson's quick response to this problem was made possible again by his support in analytical chemistry research. In addition, the whole Chemistry Division at NRL formulated a multipronged attack for the study of the use of N_2O_4 and monomethyl hydrazine as a liquid propellant for a candidate missile. The recommendation generated from this multi-man-year program was that this fuel was too dangerous to be used on board because it presented too many

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*Author's note: This is named after its developer, Otto Reitlinger, and there is no connection with Nikolaus Otto and the Otto cycle engine.

atmospheric hazards. This recommendation was accepted and this combination of chemical is not being considered as a fuel on the submarines.

7. Electrostatic Precipitators

NRL's basic research program in the early 1960s dealing with the fundamental studies of control and removal of aerosols led to the development of the electrostatic precipitators that are now on the submarines. This basic research also allowed NRL to respond quickly to solve problems generated by these electrostatic precipitators. The most notable problem found was the generation of ozone due to the incorrect polarity of the precipitator when it was installed originally on the submarines.

8. Carbon Dioxide Removal

NRL has maintained a strong program over the years in several aspects of carbon dioxide removal. It was the NRL research that led to the MEA (monoethanol amine) scrubber now used on submarines. In addition, NRL has had several experimental programs dealing with molecular sieves that are still a potential candidate for replacing MEA scrubbers. The inorganic research program has also delved into the task of using lithium hydroxide to remove the CO_2 during emergencies. There are still many problems in getting lithium hydroxide to absorb the carbon dioxide in an amount sufficient to keep the atmosphere pure.

9. Submarine Carbon Bed Research

Early work by Dr. Vic Deitz, supported by the 6.1 program on the adsorption of materials on carbon, has led to detailed understanding of how carbon may be made a more effective adsorbent of contaminants in the submarine atmosphere. This enabled NRL to make recommendations of new specifications for the carbon used in the main filter bed on nuclear submarines. This filter bed is the primary protection of the sailors against contaminants generated in the nuclear submarines. NRL's research on carbon beds also has led to direct recommendations to the fleet on the time intervals between carbon filter bed changes.

10. Carbon Monoxide-Hydrogen Burner Developments

In the early 1960s, NRL initiated a program to find new methods of removing carbon monoxide and hydrogen from the atmosphere. The basic research in combustion chemistry, particularly the catalytic combustion research of Drs. Enoch Johnson and Frank Thomas, led to the development of the carbon monoxide-hydrogen Hopcalite catalytic burner now used on all submarines. In addition, basic research studies enabled NRL to respond with answers to rather important questions when the carbon monoxide burner ingested unusual compounds. One such event occurred with the formation of a very toxic substance from the methyl chloroform. Only the availability of a strong basic research chemistry program in the area of catalytic combustion enabled the Chemistry Division to respond quickly to this very critical fleet need. In addition, this same basic knowledge of the chemistry occurring on catalysts led to an understanding of the formation of nitrogen oxides that are produced in the burner from hydrazine used in some water treatments. Thus, basic chemistry helps to understand the problem and to recommend corrective actions. None of these efforts would be possible without the 6.1 research effort.

11. Analysis of Submarine Atmospheres

NRL has long had a program of analysis of the submarine environment. Indeed, NRL is listed in the Naval Sea Systems Command Instruction Book to Submarines to be sent samples of the atmosphere for analysis whenever an emergency arises in the submarine fleet. This leads to a whole series of interesting and challenging problems which NRL is capable of addressing because of its broad-based support and availability of experts in the field of analytical chemistry. The reason these experts and equipment are able to address the very complex needs of the submarine emergency analysis is because of the diverse and powerful 6.1 analytical chemistry research program that deals with studies in advanced mass spectrometry, gas chromatography, laser spectroscopy, and other forms of analysis. A few examples are included here.

a. Analysis of the atmosphere in a submarine after a fire. The details of this analysis can be provided in a classified document, if they are needed.

b. The analysis of the waste products in the missile launch tube of SSBN submarines. NRL routinely performs analysis of the waste materials left in launch tubes after the testing of a missile launch. This is to comply with environmental constraints placed on the fleet not to contaminate harbor waters.

c. Emergency analysis, as noted above, whenever an emergency atmosphere event occurs in the fleet while the submarines are on patrol or on station. Samples of air are taken and returned to NRL for analysis. In a period of three years, NRL had done over 30 such analyses.

d. NRL is routinely asked to analyze and assess the impact which some change in the submarine's operation may have on the quality of the atmosphere in the submarine. For example, when a new water treatment system using hydrazine was introduced into the system, NRL was asked to use its sophisticated analytical techniques to assess the atmosphere problems that may be caused by such treatment. In another example, a new paint that had been used on one of the types of sonar cabinets was thought to contain mercury being emitted to the atmosphere from these cabinets above the accepted limits. Several years ago a contractor attached "Insolite" insulation to the interiors of the submarines using a solvent containing large amounts of methyl chloroform. NRL was called upon not only to identify what materials were present, which panels contained large amounts of the trapped solvent, but also to estimate for how long the methyl chloroform would be released. This was successfully concluded because NRL had a 6.1 study on the permeability of gases through materials under way at that time in the Mass Spectrometry Section. Also, NRL had been asked to formulate a statistical evaluation of all submarine atmosphere contaminants and to relate these to crew activities during the cruise. To this end, NRL scientists take cruises, analyze the atmospheres in submarines, and draw conclusions based on the data taken from which they make recommendations to NAVSEA on what type of procedures need to be followed to give the crew the best type of atmosphere available. These analyses focused on the detection of asbestos particles in the atmosphere and developing appropriate control action.

12. Analyzer Development

NRL's research has led to the development of all the atmosphere analyzers that have been used on nuclear-powered submarines. These included the Mark I, II, III, IV, and V, the total hydrocarbon analyzer, and CAMS-I. In addition, NRL research has led to development of CAMS-II, the computer-controlled mass spectrometer designed to monitor any new contaminant introduced into the submarine atmosphere. In particular, the CAMS-I and CAMS-II developments have been the most successful of all of the NRL analyzer developments. These are described in Section 13 below.

All of the analyzer developments have come out of the general analytical chemistry 6.1 task area. The earlier units, which were primarily based on infrared detection, were developed by Dr. Frank Thomas of NRL's old Electrochemistry Branch. Dr. R.E. Kagarise and his colleague, Dr. R.A. Saunders, led the development of infrared detection and analysis of contaminants. The total hydrocarbon analyzer was also developed in this analytical area from work performed by Drs. Enoch Johnson, Homer

Carhart, and Fred Williams. The Mark V atmosphere analyzer was also based on chromatography principles and was developed out of the same group with notable contributions from Dr. Merle Umstead and Mr. Harold Eaton. All of these developments had their conception in the 6.1 analytical chemistry research in early to late 1960s. The hydrocarbon analyzer and the Mark V atmosphere analyzer were an outgrowth of the program designed to improve chromatographic techniques. The CAMS work came out of the mass spectrometry research run by Dr. F.E. Saalfeld and, as noted above, the Marks I, II, III, IV, and V came out of the research run by Drs. Kagarise and Thomas.

13. The Central Atmosphere Monitor System (CAMS)

A new system for the analysis of submarine atmospheres--the Central Atmosphere Monitor System (CAMS-I)--has been developed at NRL. With this system, submarine crews for the first time are able to reliably monitor the atmosphere aboard their ships. A follow-on program has developed a more versatile system, CAMS-II.

Navy submarines are required to have the capability to maintain submerged operation continuously for periods up to 90 days. To meet this operational requirement, submarine atmospheres must be monitored and controlled. Successful monitoring of a submarine atmosphere requires an atmosphere analyzer that can be operated and maintained by shipboard personnel and can monitor the principal gases, such as oxygen, carbon dioxide, refrigerants, and hydrogen, as well as trace contaminants (carbon monoxide, acid gases, and aliphatic, aromatic, and chlorinated hydrocarbons). Submarine atmosphere analyzers have at present only a limited capability to monitor the principal gases. Moreover, some of the available analyzers have given the Fleet serious operational problems.

CAMS-I is a combination carbon monoxide detector and fixed-collector mass spectrometer. It has solved the Navy's immediate need for a reliable submarine atmosphere analyzer. This unit has passed technical and operational evaluation tests and thus has become the first submarine atmosphere monitor to become "service approved." Prototypes of the CAMS-I were installed aboard two submarines for operational evaluation and were left on board after completion of the test. Without routine maintenance, these units operated for more than 25,000 hours without a failure. Since CAMS-I had no failures during operational evaluation, the mean-time-between-failures and the mean-time-to-repair could not be determined. Presently, CAMS-I production units are being installed in the Fleet. The first prototype CAMS was installed on the nuclear submarine USS PINTADO (SSN672) in 1972.

Although CAMS-I is a reliable monitor, which was not true of previous atmosphere analyzers, it is not sufficiently versatile. If, for example, a new system were introduced into submarines, CAMS-I would not be able to monitor pollutants peculiar to the new system without substantial alteration.

The eventual replacement of CAMS-I with CAMS-II will greatly increase the versatility of the atmosphere analysis capability aboard submarines. The unit will be programmed to monitor atmospheric contaminants unique to each submarine. Eventually, all submarine life-support equipment could be automated from the analyzer, thus providing a better quality atmosphere in the submarine and freeing the crew for other duties.

The CAMS programs have been funded by the Naval Sea Systems Command. The CAMS-II program is currently in advanced development.

14. Chemistry Program

Basic 6.1 research sponsored by the ONR chemistry program has led to a new high-energy density lithium thionyl chloride primary battery that will be valuable for a number of ocean-oriented applications. These include oceanographic instrumentation, sonar systems, undersea weapons, and small undersea vehicles. For example, as part of a 6.2 project with support from the Special Projects Office, Dr. McCartney from Naval Underwater Systems Center (NUSC) evaluated these batteries for applications in the Deep Sea Rescue Vehicle Program (DSRV) for the CNO. Continuing ONR-sponsored 6.1 research is aimed at developing a high-energy density rechargeable battery.

Basic research on the synthesis of fluorine-containing materials has been under way within the ONR contracts program and in-house at NRL for some time. This research has led to a new teflon-like flourourethane paint which was applied to a 2500-ft area in the bilge of the FORRESTAL (CV 59) during overhaul in 1977. The hull of a harbor tug, the SEA MULE, has also been coated with fluorourethane paint. The materials needed for this and other applications were produced in sufficiently large quantities by an industrial chemical company following technology transfer from NRL.

15. High-Yield Strength Steel

In the mid-1950s the Navy adopted HY-80 steel for all submarine hull construction. Influencing that choice was the fact that it could be shown unequivocally that the HY-80 steel weldment system was superior to other competing systems. This was made possible by advances in fracture theory and testing techniques developed at NRL under ONR sponsorship.*

Fracture tests used in establishing acceptance criteria and for qualifying hull materials (plates, forgings, extrusions, castings, and welds), HY-80, HY-100, HY-130, HY-180, and Ti-100, such as the drop weight test and dynamic tear test, were developed at NRL under ONR sponsorship.

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*NAVSEA (then BUSHIPS) also made key contributions to the HY-80 weld system (see pages VI-151 ff.).

16. Sonar Sensors

The majority of the fundamental research leading to the development of lead zirconate titanate (PZT) and barium titanate materials for use in sonar and hydrophone applications was sponsored by ONR. These materials are used in all Navy active sonar and passive hydrophone systems. Submarine systems include BQQ-5, BQR-2, BQR-7, BQR-14, and BQR-15.

The procedures used in effectively trouble-shooting mechanical failures in submarine sonar systems are based on research conducted by NRL under ONR sponsorship.

17. Development of Radiation Resistant Steels for Nuclear Service Applications

Metallurgical guidelines have been developed by NRL for the production of radiation resistant steels for elevated temperature nuclear service relating to light-water reactor systems. The guidelines have been successfully proven with commercially produced materials and have now been adopted industrywide in the form of new material specifications by the American Society for Testing and Materials (ASTM) and by the American Welding Society (AWS). These guidelines are clearly applicable to military as well as commercial power reactors.

Prior to this achievement, low alloy steels were observed to exhibit a significant susceptibility to embrittlement by nuclear service environments. Moreover, wide variations in steel resistance to the detrimental radiation effects had been found. The NRL work, which culminated in the development of the new material specifications, included the initial isolation of the critical factors responsible for steel-to-steel variability, the identification of the fundamental mechanism controlling this behavior, and the adaptation of this knowledge for the improvement of basic steel irradiation behavior. This research has been recognized as a primary breakthrough in the safe application of steels for reactor structures.

Program time frame: 1966 to 1977
Program personnel: Code 6390
Program support: Sponsored jointly by AEC (now NRC) and ONR
Program cost: This research was part of a multifaceted program and separate costs are not readily available.

Nuclear radiation service typically produces progressive reduction in the fracture resistance of reactor pressure vessel steels. This detrimental effect is recognized by the ASME Code (Section III) and by the Code of Federal Regulations (10CFR50) such that indirect and direct limitations are imposed on postirradiation fracture resistance for commercial reactor vessels. For current reactor vessel steels, that are made to NRL guidelines for radiation resistance, there is little concern that Code requirements can be met over projected vessel lifetimes. However, for certain older reactor vessels that did not have the benefit of current technology, a combination of factors may exist that may bar meeting the Code criteria after some period of extended vessel operation. For these situations, postirradiation heat treatment has been investigated by NRL as a possible method for the periodic relief of radiation embrittlement.

Recent results demonstrate that for 288C radiation applications, a 399 °C heat treatment of one-week duration can be highly effective for reducing radiation embrittlement. Earlier NRL studies provided the necessary guidance for the *in situ* heat treatment of the Army Sm-1A reactor. The continuing studies are critically assessing the potential of annealing for embrittlement relief for a broad range of reactor vessel conditions. The conditions of interest include those encountered by both civilian and military reactors.

Program time frame:	1965-1968 and 1975-present
Program personnel:	Code 6390
Program support:	Sponsored by AEC (now NRC)
Program cost:	This research was part of a multifaceted program and separate costs are not readily available.

18. Application of Ceramic Fractography to Sonar and Other Navy Hardware Systems

The NRL Ceramics Branch has made the study of the nature of ceramic fracture surfaces and the determination of the origin of these fractures a central part of its research program for approximately the past eight years. These ceramic fracture studies have had a number of important scientific and technological payoffs. Scientific payoffs from this work include a significant extension of our understanding of the failure mechanisms of ceramics and the applicability of fracture mechanics theory to these mechanisms. This work has had broad-scale and far-reaching impact on the utilization of ceramics in Navy and DoD systems. Besides the longer term scientific payoffs, ceramic fracture studies have had direct technological payoffs. Examples of such payoffs are:

a. The development of ceramic composite systems showing promise for new reentry nose tip and radome materials.

b. The identification of the nature and distribution of failure-causing defects in ceramics for turbine and diesel engines. This work has aided in the development and utilization of ceramics in heat engines.

c. The identification of failure-causing defects in fiber optics for communication systems. This work has aided the development of such systems to meet more demanding high-stress, long-life, underwater applications.

e. The identification of failure-causing defects and stress distributions in sonar ceramics.

The sonar ceramic fracture studies are an excellent example of the application of 6.1 research results to systems' needs. This work, which began in 1971 as part of the overall 6.1 program on ceramic fracture, first showed the range and distribution of strengths available in sonar materials from commercial suppliers and the types of defects causing the failure. This work is used by the ceramic manufacturers to determine optimum mechanical strength for their processes and products. The fundamental fracture studies have further evolved from NRL's efforts to the stage where they can be applied to ceramic fractures that occur in service or in testing. This analysis has been applied to a number of systems including Mk-12 transducer of the Mk-48 torpedo, Mk-11 transducer, DT 308 Source SURTASS, experimental hydrophones at NUSC, and two Westinghouse transducers. The most extensive and most important sonar ceramic fracture studies have been carried out on the Mk-48 system and on the Mk-11 transducer.

19. Other Research Activities

Although many areas have been covered in showing how NRL has contributed to the support of the Navy's submarine fleet, not every activity can be covered. Only two will be mentioned here. The first deals with the contamination found on the electrical contacts in the guidance system. It was reported by the Strategic Systems Project Office (SSPO) that many of the guidance systems, when tested, generated electrical noise. NRL's surface chemistry program and the analytical chemistry program joined forces to track the problem to the outgassing of contaminants from materials used in the gimbel that surrounds the slip ring which then condensed on the surface of the slip ring. It was the presence of these impurities that gave rise to the noise. Through experience developed in other of NRL's programs, they were able to recommend new cleaning techniques. The second effort dealt with the generation of discharges in the very-low-frequency (VLF) capacitors that are used in the communication link with submarines. NRL mounted a multidisciplined challenge to identify the causes of the problem and to point ways to improvements. Both of these efforts were again made possible by the strong 6.1 in-house effort. For example, NRL supports a 6.1 research program in surface analysis which provided the essential capability for analyzing the contamination of the slip rings. The support in the analytical instrumentation section made it possible to solve the problems with the VLF capacitor which were also due to contaminants.

B. AIR FORCE OFFICE OF SCIENTIFIC RESEARCH (AFOSR)

It has to be noted, in fairness, that the OSR input to the authors did not include chemistry work, which made such a heavy contribution to the ONR data. This is entirely the responsibility of the authors who did not request chemistry projects but also did not see fit to edit them out of the ONR input. In Phase II of the present study, the authors intend to look more deeply into the chemistry 6.1 projects of all three Services which seem to yield a short technology transfer time compared to materials and mechanics.

OSR noted that the timing of the present study was a bit early to include the titanium aluminides (Ti_3Al and TiAl). The Air Force predicts that these new intermetallics will be a "success" story in another two or three years, particularly as applied to gas turbine use.

Under the basic research Project 7353 on metals and ceramics, a substantial research effort was conducted from the early 1960s to the early 1970s under Task 735306, Fundamentals of Composites, in the area of metal matrix composites. This encompassed work on various types of reinforcing materials such as whiskers, filaments, wires, coated filaments, etc., metallic and ceramic matrices, particularly aluminum and titanium and on the basic interactions, processing methodologies, and properties of the composites and filaments. One of the more promising of these structures was based on B and Borsic filament reinforced aluminum composites--which led to later development of such items as engine fan blades and tubular structures for spacecraft. The latter, at present, are prime candidate materials for space applications.

Under the current Project/Task 2306/P6, Failure Prediction and Avoidance, basic research on corrosion inhibitors has been conducted for the past six years. The present program with the

University of Florida is conducted under Contract F33615-75-C-5200 with Professor Ellis D. Verink, Jr. as the Principal Investigator. The purpose of this program has been to provide a fundamental basis for the selection and development of new multifunctional inhibitors for the protection of high strength alloys to prevent degradation and failure from localized and general corrosion. Much of the effort has emphasized characterizing and inhibiting reactions at the crack tip or in pits such as occurs in stress corrosion cracking, hydrogen embrittlement, and corrosion-fatigue. Several major developments of practical significance have been derived from this program already. Rapid new electrochemical screening techniques have been developed and used to identify new promising inhibitor systems. The basis for selecting current multifunctional inhibitors for the F-4 Automated Rinse Facility at MacDill AFB, Florida, was provided. The results will also directly benefit a new FY 78 6.2 program on the development of bilge inhibitors for large cargo aircraft such as the C-141A. Furthermore, it appears that we are on the verge of a scientific breakthrough in our understanding of the competing roles of hydrogen embrittlement and rapid anodic dissolution in environmentally related catastrophic failure of high strength steels. This would be of great practical as well as theoretical significance if the results of comparative studies at Boeing Aircraft and the University of Florida validate their preliminary results.

Some of the results concerning the Metallurgy of Structural Materials Program that have had significant impact on 6.3 Advanced Development Activities (specially 6.5211F--Aerospace Structures and Materials and 7.8--Manufacturing Technology) are listed below.

Area of Work	Dates of 6.1 Activity	People	Total
Improved Al Alloys Through Powder Metallurgical Techniques	1971-79	Fine/Northwestern Starke/Georgia Tech Lawley/Drexel Lankford/Southwest Research Institute Davidson/Southwest Research Institute	$600K
Lower Cost by Substituting Steel for Ti (LOCOSST Program, AF 14-10 Steel)	1974-76	Antolovich/ Cincinnati	$ 65K
TiAl Alloys for Engine Applications	1975-78	Collings/Battelle Hren/Florida	$200K
Metal Matrix Composites B/Al Fan Blades	1977-79	Carlson/General Electric	$100K

One of the topics of sponsored research for the past several years has concerned itself with two- and three-dimensional analysis of cracks. Both Finite Element and Boundary Integral Equations (BIE) methods were used in this research effort. Specifically, new mathematical models were sought that provided improvements in accuracy and efficiency over the current state of the art. Results of these investigations have been applied in two known instances. The hybrid finite element model of two-dimensional cracks has been incorporated into the TEXGAP computer code. This code is a widely used design and analysis tool within the solid propellant industry. Also, the stress solutions for three-dimensional cracks in elastic materials using BIE have been incorporated into the engine disc-life prediction procedure currently employed by Pratt and Whitney in the design of the F-100 engine used on the F-15 and F-16.

C. ARMY RESEARCH OFFICE (ARO)

1. Case Study of Basic Research on the Thermomechanical Processing of Ceramics as Related to Army Applications in Armored Vehicles, Missiles, Gas Turbines, Armaments, and Vision Devices (1971)

Motivation

The Army has a growing need for high strength-to-weight materials for ballistic armor and for heat- and erosion-resistant components for aircraft and missiles. Refractory ceramics are of special interest for these applications because of their high potential load resistance, low density, and favorable resistance to some aggressive environments. However, highly innovative processing techniques are required to overcome the inherent brittleness and low resistance to thermal stresses of refractory ceramics. Processing is usually the major potential barrier to achieving the needed shape and size, optical and electrical properties, cost-effectiveness, and reliability in structural ceramic components.

Army Interest

Structural ceramics are required for lightweight, transparent and opaque armor, high-temperature gas turbine components, nongalling bearings, nose tips and radomes, IR and laser windows, and high strength-to-weight materials for logistic support systems.

Background

Research by T. Vasilos at Avco Corporation during the late 1950s employed hot-pressing technology (which involves the simultaneous application of heat and pressure to sinter ceramic powders into dense fine-grained bodies) to fuse silica into aerodynamic shapes for reentry nose cones. Under ARO sponsorship beginning in 1963, R.M. Spriggs and T. Vasilos extended this research at Avco to other refractory oxides, and they concentrated on establishing the effects of thermomechanical processing at high temperatures and pressures on the microstructure and properties of polycrystalline ceramics.

It was during the course of this work that Spriggs discovered in 1964 that hot-pressed sintered compacts could be deformed under compression at high temperatures to produce essentially fully dense, fine-grained, crack-free bodies with microstructures resembling those of hot-worked metals. This thermomechanical process, which was named "press-forging," not only imparted an unusually high strength and elastic modulus but also produced a high degree of transparency to certain oxides, such as Y_2O_3 and MgO. This work, along with the development by Robert Coble of translucent Al_2O_3, "Lucalox," by conventional sintering methods, pioneered the applications of transparent oxide technology to the development of transparent armor, optical windows, and laser host materials.

Since 1965 ARO has sponsored research by Professor Spriggs and colleagues at Lehigh University into understanding both strengthening mechanisms of thermomechanically processed polycrystalline ceramics and the kinetics of synergistic densification effects that occur when thermomechanical (pressure-sintering and hot-pressing) and thermochemical (decomposition, transformation, liquid phase formation) processes occur concurrently. This research has had a significant impact on the development of novel processes for structural ceramics and on the expanding industrial use of pressure-sintering and press-forging for producing oxide, carbide, nitride, and boride components of increasing size and geometrical complexity. Examples of recent important accomplishments of Professor Spriggs' research include the synthesis of single crystals (e.g., transparent Al_2O_3) by the strain-annealing of press-forged bodies, a description of pressure-sintering kinetics for Al_2O_3, and an improved understanding of the effects of thermomechanical processing on the thermal stress fracture behavior of brittle ceramics.

Since 1965 there has been a growing interest in the use of structural ceramics for ballistic protection of personnel and material, and active in-house programs and contracts involving AMMRC, the Natick Laboratories, TACOM, MERDC, and AVSCOM have been devoted to the development of lightweight, transparent, and opaque facing materials for armor systems. In the missile areas, work has been carried out at AMMRC and MICOM in the use of ceramic materials in components such as radomes, nose cones, rocket nozzles, ablative shielding, and infrared transmitting windows. The application of ceramic materials to laser rods and infrared laser windows has involved widespread participation on the part of MICOM, FA, HDL, ECOM, and AMMRC. Since 1971 AMMRC had been contract monitor for a large-scale ARPA program involving Ford Motor Company and the Westinghouse Electric Corporation to develop a ceramic gas turbine engine by 1976. These programs made use of hot-pressing and press-forging technology, and benefitted from contributions to the fundamental knowledge underlying these processes by Professor Spriggs and his associates over the previous years. Of course, many government, industrial, and university laboratories have also contributed to the fundamental knowledge.

Specific Army Applications

Transparent Armor. Transparent armor has been developed mainly for aircraft windows and viewing devices for ground-based vehicles. The development of transparent ceramic armor offers a weight reduction of 50% over transparent glass systems, with an even larger thickness reduction. Monocrystalline Al_2O_3 and polycrystalline $MgAl_2O_4$ are the primary candidate materials for this application.

Hot-pressing and press-forging techniques that offer an alternative to crystal growing for producing large transparent alumina slabs have been developed. One method, developed by Spriggs, involved the press-forging of Al_2O_3 followed by the formation of single crystals by strain-annealing techniques. The second method, developed by Dr. Vasilos of the Avco Corporation, also involved the press-forging of polycrystalline alumina. However, transparency was achieved by developing a high degree of crystallographic alignment of the grains. A third approach, developed at AMMRC, involved the production of transparent, polycrystalline magnesium aluminate* ($MgAl_2O_4$) and lithium aluminate ($LiAl_5O_8$). Since those compounds are cubic, the polycrystalline forms are transparent, and single crystals are not required. Therefore, these materials offer the promise of a potentially cheaper, lighter weight material than sapphire. Two processing methods were evaluated: press-forging, which is a contractural effort, and hot-pressing, which was developed at AMMRC. The size of the bodies that can be produced by these techniques was limited primarily by the size of the equipment and tooling. However, disks in size of 4 inches in diameter by 0.5 inch in thickness have been produced.

Since Al_2O_3 is hexagonal, polycrystalline Al_2O_3 will not be transparent because of birefringence unless special processing techniques are developed. AMMRC, therefore, over the years has concentrated on crystal-growing methods to produce transparent, single-crystal alumina disks that are subsequently assembled into a mosaic transparent armor system. Union Carbide Corporation, under contract to AMMRC, has developed processing equipment and procedures for preparing alumina crystals from the work on a production scale.

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*Frequently called Spinel.

Electromagnetic Window and Optical Materials. The requirement for high-quality transparent ceramics exists for uses such as reflex sights, high-intensity arc lamp windows, and laser windows in the 1-5μm range. High-energy transmitting laser windows require materials that have a high optical transmittance and low optical absorption coefficient ($<0.001 \times cm^{-1}$), a high resistance to radiation damage, high strength, and high resistance to thermal stress fatigue and fracture.

Although single crystals, such as sapphire, are available for many of these applications at wavelengths below about 5μm, limitations in processing and excessive optical absorption preclude their use for some applications, such as high-energy transmitting laser windows. Therefore, the development of transparent polycrystalline ceramics by the use of hot-pressing and press-forging techniques has been pursued at AMMRC and elsewhere. Additional process development for sapphire is also warranted to determine the extent to which its optical absorption is intrinsic or process-related.

Previously, the formation of transparent polycrystalline yttrium oxide (Y_2O_3) required either the use of densification aids, such as lithium fluoride, or the use of press-forging after hot-pressing. Studies at AMMRC have demonstrated that transparent Y_2O_3 with excellent optical and mechanical properties can be fabricated by conventional hot-pressing techniques. Success in this method requires careful control of heating rates, the temperature range of pressure application, and the selection of tooling materials.

A variety of polycrystalline fluorides and II-VI compounds are being industrially produced by the hot-pressing of powders for a number of optical and infrared window applications. The "Intran" materials produced by Kodak Corporation are examples of those materials.

High-intensity infrared laser window development under Air Force sponsorship for CO_2 lasers operating at 10.6μm has led to important applications of press-forging in the processing of alkali halides (KCl, NaCl, and KBr), II-VI compounds (CdTe, ZnSe, ZnTe, and CdSe), and alkaline-earth halides (CaF_2 and SrF_2). The press-forging of KCl single crystals at 300 °C produces low-angle (5-10μm spacing) grain boundaries and increases the yield strength from 300 to 3000 psi. Doping the KCl with 500 ppm of strontium further increases the yield strength to 5000 psi, which is sufficiently high to withstand a one atmosphere pressure drop superimposed on thermal stresses. The Harshaw Chemical Company is now supplying 12-in. alkali halide windows by these techniques. Although these windows were developed under Air Force sponsorship, Army applications requiring windows in the high infrared will benefit from this technology which, again, derives from the ARO research.

Propulsion and Power Generation. Under ARPA sponsorship an iterative design and materials development program conducted by the Ford Motor Company and the Westinghouse Electric Corporation aimed at demonstrating the technical feasibility of operating a small vehicular gas turbine (Ford) and a large stationary gas turbine (Westinghouse), each utilizing uncooled ceramic components, at temperatures above 2500 °F. The AMMRC had technical monitoring responsibilities under the program. The use of ceramics in combustors, stators, rotors, ducting, and other components led to higher efficiency, lower weight, lower cost, higher fuel economy, and lower exhaust emissions. Silicon carbide and silicon nitride were the prime ceramics being evaluated because of their favorable resistance to oxidation, erosion, thermal shock, and creep. Ceramics of the SIALON-type (silicon aluminum oxynitrides) were also evaluated because of their favorable thermal shock resistance and superior oxidation resistance compared with silicon nitride.

Slip casting or injection molding followed by reactive-sintering had been the leading processing method for the production of complex shapes of Si_3N_4. However, the thermal stress and creep resistance of these components were limited by incomplete densification (porosity), silicon grain growth, and the segregation of impurities, such as calcium, during the reactive-sintering process. Consequently, hot-pressed Si_3N_4 demonstrated superior performance compared with reactive-sintered bodies for such components as rotor hubs and stator vane assemblies which require high creep resistance. For example, a hot-pressed Si_3N_4 rotor hub did not fail at 82,000 rpm in rotor spin tests conducted by Ford Motor Company, a speed which is well above the 64,200-rpm maximum design speed for the small vehicular gas turbine. Hot-pressed SiC and Si_3N_4 stator vanes fabricated by the Norton Company were extensively inspected and subjected to long-term evaluation tests at 2500 $^{\circ}$F.

Commercial availability of these ceramics was limited, however, and process development at AMMRC further optimized the high-temperature strength and creep properties of hot-pressed silicon carbide and silicon nitride ceramics and to demonstrate the cost-effectiveness of fabricating larger and more complex shapes by hot-pressing techniques. These studies explored the use of additives that will increase the refractoriness of the grain boundary phase in addition to promoting complete densification, thereby increasing strength and creep resistance at 2500-2700 $^{\circ}$F.

Lightweight Opaque Armor. Lightweight opaque ceramic armor was in use for personnel ballistic protection on the ground and in aircraft under conditions where weight limitations are of paramount importance. After several years of development, it had been demonstrated that boron carbide (B_4C) and silicon boride (SiB_6), which have been hot-pressed to minimize porosity, have excellent resistance to caliber 38 AP projectiles. Although boron carbide exhibits a slightly higher ballistic performance, silicon boride has the lower density (2.43 g/cm^3 compared with 2.54) and can be hot-pressed at lower temperatures (1550-1600 °C compared with 2000-2100 °C).

The mechanical energy imparted during hot-pressing substantially reduces the temperature at which densification of ceramic bodies takes place. However, combined thermomechanical and thermochemical processing methods have been under active development to further reduce densification temperatures and thereby to reduce die wear, energy costs, and overall fabrication costs. These methods include reactive-sintering, in which the heat liberated during a chemical reaction contributes to the consolidation process, and the introduction of a sintering aid, a liquid-phase solute or impurity, to enhance densification. As an additional benefit, lower hot-pressing temperatures facilitate the attainment of smaller grain sizes which, in turn, promotes improved strength and ballistic performance.

During 1970-1971 AMMRC developed a reactive-sintering/hot-pressing procedure for the fabrication of silicon boride armor material which had a ballistic performance close to that of boron carbide armor, and which could be hot-pressed at a temperature 400 °C lower than could boron carbide. These methods have since been extended to aluminum boride.

In research conducted at AMMRC, the use of Na_2SiO_3 and $Mg(NO_3)_2$ additives to boron carbide had been found to reduce the hot-pressing temperature for boron carbide. Other agents, such as aluminum, silicon, nickel, titanium, yttrium, and some hydrides, which will catalyze or accelerate the sintering process in boron carbide, silicon boride, and aluminum boride, were explored at AMMRC to further improve material integrity at lower cost. These techniques also applied to the fabrication of high-density silicon bodies containing various alloy additives (B, Al, B_4C, SiC), lightweight lithium-based ceramic compounds (density 2.0 to 2.3 g/cm^3), and beryllium oxide compounds with ultrafine grain size (1-5μm) (see Chapter VI-I-6, page VI-173).

Other Applications

Gradient Armor. Armor plates consisting of a hard ceramic facing plate (TiB, TiB_2, and HfB_2), a deformable backup plate (Ti), and a graded interface between the two plates have been developed under a joint program between AMMRC and Manlabs, Inc. These plates are produced by pressure-sintering methods.

Missile Nose Tips and Radomes. Boron nitride and silicon nitride, densified by reaction-bonding and/or hot-pressing, are being evaluated for the next generation of radomes and radar antenna windows (ABM). In the case of silicon nitride, it has been confirmed at AMMRC that this ceramic possesses a low dielectric constant (at 10^{10} cycles/sec) and low dielectric loss in addition to having superior strength, thermal shock resistance, and erosion resistance.

Ceramic Bearings. Hot-pressed Si_3N_4 rollers, developed for helicopter rotors, exhibit eight times the fatigue life of bearing steels, and may be able to survive under conditions of lubricant starvation. Ceramic gas bearings are also being developed for high-temperature use in gas turbine engines.

Battery Spacers. Hot-pressed lithium aluminate, which was developed with transparent armor in mind, has been found to be an excellent insulator and container for liquid lithium in Li/Se batteries. Therefore, it has an excellent potential for power-generation and storage systems.

Cutting Tools and Dies. These represent an expanding application of ceramics, and the industrial capability for manufacturing these parts has grown out of pressure-sintering and press-forging technology. The use of these tools and dies has greatly increased production rates, and has resulted in significant cost reductions in defense-related manufacturing industries.

Summary of Results (see Item 1, page V-24)

Basic research conducted over approximately ten years under ARO sponsorship by Professor Spriggs, his associates, and students, established important relationships between processing variables and the microstructure and mechanical properties of polycrystalline ceramics densified by a variety of thermochemical and thermomechanical methods. Out of this understanding has emerged several novel processing techniques, a growing ceramics industry in the manufacture of high-strength, fully dense, polycrystalline ceramics, and an expanding list of Army applications of these ceramic materials.

Hot-pressed, press-forged, and reaction-hot-pressed oxides, carbides, borides, and nitrides have Army applications in propulsion and power generation, electromagnetic window and optical materials, and protection and survivability materials which serve a number of mission areas. One of the more dramatic recent applications of hot-pressed ceramics (silicon carbide and silicon nitride) is in the development of uncooled ceramic gas turbine engines and power generators. The successful development of these systems will have a dramatic impact on the thermodynamic and mechanical efficiency of transportation systems, on the net efficiency of electric power generation stations, on the versatility and economy of hydrocarbon fuel combustion, and on the reduction of thermal and NO_x pollution.

2. Graphite Intercalation Compounds for High Electrical Conductivity

Research performed by F.L. Vogel at the University of Pennsylvania and sponsored by a grant from the U.S. Army Research Office on the processing of special graphite materials (called intercalation compounds) (Ref. 9) has yielded highly stable and reproducible products which have a higher electrical conductivity than that of copper. The excellent electrical properties of these materials depend upon the deposition of certain chemical species at atomic positions between sheets of the highly layered graphite structure. Other important properties of these materials include a 75% weight saving over copper for the same size specimen, and substantial changes in the amount of infrared light which is reflected from the material's surface. The ARO project was part of a larger effort at the University, funded also by AFML, AMMRC, ARPA, ERDA, MERADCOM, NSF, and ONR. The potential Army applications include protection from damage by lightning strikes on advanced polymer matrix composites, infrared camouflage, and lightweight electrical wires for equipment requiring greater mobility.

3. Systems Analysis of Brittle Fracture

In this research (G.T. Hahn, Battelle-Columbus) into the mechanisms that lead to the failure of materials by fracture, Dr. Hahn studied crack propagation in order to relate (1) the speed at which the crack advances, (2) the energy dissipated by the cracking, and (3) the path of the crack after it becomes unstable, with the microstructural and mechanical properties of materials. Crack propagation is important technologically because it influences the amount and type of damage that accompanies failure. For example, a low-energy, fast-moving crack is likely to produce the complete fragmentation that is desirable in certain projectiles. In contrast, a high-energy, slow-moving crack keeps damage to a minimum in structures, such as gun tubes, wherein structural integrity is indispensable.

From his studies, Dr. Hahn has been able to develop a mathematical expression for the speed of propagation of a shear crack. Although the formulation is approximate and still tentative, it has already been exploited in an analysis of the speed of fracture in pressurized gas transmission lines, and in steel plate (ship steel). This work has led to a new experimental method for studying the rate of propagation of cracks, and a new way for evaluating the dynamic toughness of metals. These methods of testing and analysis may prove exceedingly useful in the prediction of the fragmentation characteristics of materials considered for use as projectiles. This application is being investigated in discussions at Picatinny Arsenal with Army personnel engaged in fragmentation studies.

4. Kinetics of Halogen Gas/Solid Reaction

Studies by Daniel E. Rosner, Yale University, of high-temperature heterogenous fluorine surface reaction kinetics have led to experimental methods to investigate rates of recombination of halogen atoms on solid surfaces and gasification of surfaces exposed to transonic flow and high temperature. These methods, in turn, form the basis for experimental techniques to design, evaluate, and select materials suitable for uncooled nozzles for high-power hydrogen fluoride chemical lasers, for container materials for these atomic gases, and in the diagnosis of performance of gasdynamic laser systems employing the H_2 + F reaction. The laser application was the motivation for a DARPA Materials Research Council Summer Conference on Fluorine Resistant Materials, and the investigators' DARPA project on fluorine reactions on ceramic materials.

5. Deformation Behavior of Solidifying Metal Alloys

Investigation by M.C. Flemings, Massachusetts Institute of Technology, of the roles of fluid velocity, viscosity, and amount of second phase in liquid-solid slurries in determination of final structure and homogeneity of cast solids indicated the following.

Through control of velocity of stirring, viscosity, and other variables in the "mushy" region, the investigators were able to:

- pour or die-cast metallic alloys at much lower than conventional temperatures (longer die life, improved handling),
- obtain much lower shrinkage on complete solidification (and provide ability to cast very thin, reproducible sections),
- greatly reduce gas content of final casting (gas escapes during stirring), and
- homogenize cast structures (eliminating the need, in many cases, for subsequent working and heat-treatment).

Present and future extensions of work on alloys include the following tasks:

1. Improved thermomechanical processing techniques.
2. Hot-tearing in cast alloys.
3. Strengthening (and weakening) mechanisms.
4. Processing of novel composite materials.
5. Strength and deformation of sea-ice and snow, wet sand.

6. Alloy Chemistry of the Transition Metals

Professor Paul A. Beck, University of Illinois; years of support--1953-71.

Impact: revelation of how to avoid the range of composition and temperature leading to embrittlement of steels and nickel-based superalloys during service at high temperature.

The scientific quality of Professor Beck's work was highly regarded throughout the period during which ARO sponsored him. In 1959, his accomplishment in the discovery of new phases in several alloy systems was cited. In 1961, attention was given to his identification of more new phases, and to his measurements of the low-temperature heat capacity of his phases, a key to the electronic structure of alloys.

In 1962, Professor Beck organized and chaired a Symposium on "Electronic Structure and Alloy Chemistry of the Transition Elements," then edited the book that derived from it, a book which is a classic reference in the field. Year-by-year, he was increasingly able to systematize his data on the constitution and the thermodynamics of the alloys of those transition elements. Then, in 1970, recognition was made to an outstanding practical development that was built upon Beck's fundamental research on such metals as iron, chromium, cobalt, manganese, nickel, and tungsten, which had provided knowledge of the structure, composition range, and physical properties of a host of phases of variable composition.

Beck's results contributed mightily to the development of an industrially important application known in engineering circles as "PHACOMP." This is a computerized procedure for calculating the limited range of composition of the high-temperature alloys used in turbine blades in which precipitation of an embrittling constituent in the grain boundaries will be minimized during the prolonged heating of those parts in service. These alloys contain several of the transition metals--particularly, nickel, chromium, cobalt, molybdenum, tungsten, and iron. Guidelines established by Beck allow design engineers to select alloy compositions that promise freedom from the precipitation of embrittling intermetallic phases. The superior alloys that have resulted from this development have significantly improved the safety, efficiency, and durability of the jet engines used in military and civilian aircraft. Everyone who flies in such aircraft benefits, as do those who pay for the engines.

7. A Case Study of Basic Research on Rare-Earth Compounds to Army Applications as Permanent Magnets and Hydrogen Storage Materials (1974)

Motivation

There was great need for permanent magnets which had higher energy products, had higher operating temperatures, would not demagnetize in service, weighed less, and cost less than commercially available expensive platinum cobalt magnets.

Army Interest

Such magnets were required for important Army applications, such as radar amplifiers in missile guidance systems, motors, generators, electronic sensors, electronic warfare communications systems, and electronic fuzes.

Background

Early research by Professors Wallace and Craig, University of Pittsburgh, led to the discovery of samarium cobalt compounds that are the most powerful permanent magnets known today. Subsequent research by the Air Force Materials Laboratory, Dayton, the General Electric Research Laboratories, the Raytheon Company, and Wallace and Craig (sponsored during 1965 to 1974 by ARO) have contributed to the development of samarium cobalt and related magnets to the point that they are now being used in a number of Army systems and being considered for use in many others. Several Army laboratories, including ECOM, HDL, MERDC, MICOM, and PA had active in-house programs and contracts, ranging from 6.1 to 6.4, which were concerned with research and development of devices or systems using such magnets.

Advantages

The usefulness of a particular material as a permanent magnet depends on the amount of magnetization that can be induced in the material before it becomes saturated and on the amount of magnetic field needed to demagnetize it. Samarium cobalt and related alloys have the highest magnetic energy product of any material yet discovered, i.e., they are perhaps the strongest permanent magnets known.

This high magnetic energy product results in many useful engineering properties. The designer can use less-exacting and less-expensive circuits, because magnetic overloads will not harm the magnets. The manufacturing tolerances can be less precise for generators, motors, and other devices without reduction in performance. This results in greatly reduced manufacturing costs and improved reliability.

These magnets have a magnetic energy product which is four to five times that of the commonly employed AlNiCo permanent magnets, and scientists predict that the energy product of samarium cobalt alloys may double once the alloys and circuit designs have been optimized. Such magnets allow a large reduction in size and weight for comparable performance in many devices or a large increase in energy output for a comparable size and weight. An additional reduction in size and weight of devices results because the bulky iron magnetic circuits required by conventional magnets are not necessary for these magnets.

Samarium cobalt materials cost approximately $60/lb compared to over $2000/lb for platinum cobalt, which is the material having the highest magnetic energy product other than samarium cobalt. (The magnetic energy product of samarium cobalt is two to three times that of platinum cobalt.) A breakthrough at the General Electric Research Laboratories resulted in a new process to produce the samarium cobalt powders. The projected cost for these powders was $8 to $12 per pound which was comparable to AlNiCo materials produced in 1974.

In summary, samarium cobalt permanent magnets will become available in large quantities at costs that compete with AlNiCo and that are less than 1% of the cost of platinum cobalt. The magnetic energy product of samarium cobalt is more than double that of platinum cobalt and more than four times that of AlNiCo. Devices and machines using samarium cobalt will be lighter, less bulky, more reliable, and have a greater cost-effectiveness. In many cases, new devices and machines will be made available which were impossible with conventional magnetic materials.

Specific Army Applications

SAM-D Missile System. The magnetic components and associated electron gun for the traveling wave tube for the downlink amplifier in the SAM-D missile system have been designed, built, tested, and accepted. The last item required to complete the development of the tube was the cathode structure. In particular, the fast cathode warm-up was tested, and lifetime tests were run on the tube. The final engineering development model was scheduled to be system-tested in mid-1974 and the tubes scheduled for delivery in 1975. The cost to develop the traveling wave tubes exceeded one million dollars, and each tube cost approximately $2-3000. The periodic permanent magnets for these tubes were made from samarium cobalt. The tube has a gain of 73 dB compared to a gain of 45-50 dB from a tube of comparable size that uses conventional AlNiCo magnets. Approximate calculations indicate that a tube made with AlNiCo to produce a gain of 73 dB would be four times the diameter, 6-9 inches longer, and the magnet weight would double or triple. The size and weight requirements of the guidance system could not be satisfied at all if the samarium cobalt magnets were not available. The gyroscope in the guidance system in the missile also contains samarium cobalt magnets rather than the conventional AlNiCo because of the reduced size and weight. The developer of the traveling wave tube and the gyroscope magnets is the Raytheon Company.

High-Speed Permanent Magnet Generator

A high-speed permanent magnet generator utilizing samarium cobalt magnets was developed by the Westinghouse Electric Corporation under contract from MERDC. The machine is rated for

100 kW at 60,000 rpm, 2000 Hz, and is intended to be driven directly from the high-speed output shaft of a gas turbine. The generator is to be part of an alternator/convertor system wherein power will be generated at 2 kHz and then converted to 60 or 400 Hz, as required, to power Army end-item systems in the field, e.g., radar, missile guidance, and computer systems. The 100-kW system would weigh approximately 350 lb and would replace a comparable generator of conventional design that weighs approximately 850 lb. The magnetic components have passed the required tests, but failure of certain structural members of the system to withstand the rotational stresses imposed by higher speed operation slowed the pace of development. This is the only known DoD project involving development of a high-speed permanent magnet generator of 100-kW capacity.

Advanced Electronic Fuze Systems

Many advanced fuzing systems for projectiles (e.g., bomb, rocket, artillery, and mortar) require a small economical power source that is capable of remaining inert for long storage times and then reliably providing full power within a few thousandths of a second after launch. Military Standard 1316-A requires that such projectiles have two independent safety and arming systems. HDL, ECOM, and P′ have demonstrated that these requirements can be met with electromechanical devices that convert the mechanical energy of setback during launch or the ram-air from flight to electrical energy to power the fuzing system.

The design of the fluidic alternator was scheduled for type classification in FY 75 for use in the XM-734 multioption fuze for the 60mm mortar. This resulted in the production of many million fuzes through 1980. These plans included adaptation to the 81mm mortar which would double the production. The older generators used AlNiCo magnets, but scientists at HDL and ECOM plan to replace them with the more powerful samarium cobalt alloys to increase the reliability and decrease the cost.

The General Electric Company has developed a setback generator using samarium cobalt under contract from Picatinny Arsenal for use in powering electronic fuzes for a wide range of artillery ammunitions. The device generates a high voltage pulse in 1/1000 sec during the inertial setback when the round is launched. This energy is used to power the fuze in flight. The General Electric designs with samarium cobalt have produced four times the electrical energy productivity of the presently available generators which use AlNiCo magnets. Three prototype devices have been delivered to Picatinny for firing tests. In December 1973 a final design was adopted and 20 generators were delivered for further firing tests. These setback generators were ultimately meant to be used as part of a standard universal fuzing package for use in a range of artillery projectiles from hand-held recoilless rifles to 105mm tank guns.

Microwave Filters and Power Limiters

The Electronic Warfare Laboratory at Ft. Monmouth has indicated a need for an electronically tunable magnet for microwave filters and power limiters for surveillance and communications in the K- and K_a-bands. Designs, which use electromagnets to provide the magnetic fields for tuning the filter to the desired frequency, weigh over 30 lb and require 200 W of electrical power to operate. The feasibility of new designs by ECOM scientists using samarium cobalt permanent magnets had been demonstrated. The device could be optimized to occupy 5% of the space of the older device, weigh less than 5 lb, and operate on about 0.5 W.

Permanent Magnet Alternator

Samarium cobalt permanent magnets were scheduled for use in the alternator for the T700 power plant for the UTTAS (Utility Tactical Transport Aircraft System) and the AAH (Advanced Attack Helicopter) Army aircraft.

New Devices as of 1974

Scientists at ECOM have proposed many new devices, based on samarium cobalt permanent magnet technology, in response to current Army needs. Examples include a blast-proof loudspeaker for battlefield applications, scaled-up fluidic generators for powering identification lights for aircraft, inexpensive power sources for emergency radios for downed aircraft, and frictionless bearings. Many of these devices await development. In most cases, only modest funding would be required to develop the designs to the 6.3 stage.

Summary of Results (see Item 7, page V-38)

In summary, basic research at the University of Pittsburgh, sponsored over a continuous period of about nine years by ARO, has been coupled with military and industrial research and development resulting in a new permanent magnet material, samariam cobalt, which was used previously in several Army applications. The success of the presently adopted systems, e.g., electronic fuzes, alternator for the T700 power plant for the UTTAS and the AAH Army aircraft, and the traveling wave tube for SAM-D, will pave the way for an expanding list of permanent magnet samarium cobalt generators, alternators, motors, radar amplification tubes, etc. The engineering advantages of these magnets will allow improvement of existing hardware, increase cost-effectiveness, and permit design of devices which would not have been possible with previously available magnetic materials in 1974.

Another fortuitous result from the research at the University of Pittsburgh is that the lanthanide-iron compounds and their hydrides show promise for fuel cell applications. If the data generated at ECOM substantiate such potential, these compounds will improve the Army's fuel cell technology.

8. Additional ARO Basic Research Accomplishments

A summary of additional Basic Research accomplishments of ARO is given in Table 2.

TABLE 2. ARO BASIC RESEARCH ACCOMPLISHMENTS

Investigator	Accomplishment of Research	How Results Were (or are being) Applied
A.S. Tetelman, UCLA "Development of Small Scale Fracture Testing Techniques"	Developed a small-scale fracture toughness testing technique for high strength materials	System is being used by many DoD installations, industrial organizations, and others for rapid, reliable, routine screening and quality control at cost reductions of 50-80% (e.g., steels for gun tubes and ship plate)
M.C. Flemings, MIT "Behavior of Semi-Molten Metallic Alloys"	Established effects of processing variables in the semi-molten slate upon quality of cast materials	Development of high-quality, uniform cast alloys, with substantial extension of life of casting equipment (scale-up by FA, AMMRC, and ARPA)
P.A. Beck, University of Illinois "Alloy Chemistry of the Transition Metals"	Demonstrated how to avoid embrittling range of temperature in steels and nickel-based superalloys	Superior classes of alloys were designed, based upon the "PHACOMP" method of phase analysis, extending operating ranges of jet engines
W.E. Wallace, R.S. Craig, University of Pittsburgh "Intermetallic Compounds Containing Lanthanides"	Development of rare-earth cobalt-based compounds, most powerful permanent magnets known (sponsorship also by USAF)	Traveling wave guide tubes for radar detection and guidance (e.g., SAM-D); power sources for munition fuzes, tunable microwave filters and power limiters (electronic warfare and counter-intelligence)
R.R. Stromberg, National Bureau of Standards "Adsorption of Polymers on Metals"	Development of a precise method (ellipsometry) for measurement of thickness of organic films on metals	Control tests for adequacy of corrosion-inhibiting films and coatings on weapons, vehicles, machinery, etc.
R.M. Spriggs, Avco Corp; Lehigh University "Strengthening of Ceramics by Thermomechanical Processing"	Attainment of marked increase in strength of structural ceramics, and development of transparency	AGILE systems, transparent armor, infrared windows in heat-seeking rockets, high-temperature windows (e.g., for lasers), microwave apparatus
D.E. Rosner, AeroChem Laboratories; Yale University, "Kinetics of Interaction of Solids with Atomic Oxidizers"	Improvement of incandescent lamps for high intensity and long life ("self-cleaning")	Floodlighting for security of installations; (possible: surveillance, acquisition of targets)

VI. SELECTED CASE HISTORY INFORMATION

A. THE C4 MISSILE SYSTEM

1. Systems History of Fleet Ballistic Missiles (FBMs)*

a. Genesis of the FBM. A February 1955 report of the National Security Council, while stressing the urgency of the intercontinental ballistic missile (ICBM) program, also recommended development of a sea-launched intermediate range ballistic missile (IRBM). Initially, the deployment concepts focused on surface ships with secondary consideration to submarines. In early November 1955 the Secretary of Defense directed the Army and the Navy to proceed jointly with the development of a sea-based 1500-nmi liquid-fueled JUPITER system and subsequently the Navy Special Projects Office was created (the forerunner of the present Strategic Systems Project Office) on 17 November 1955.

The Special Projects Office soon concluded that there would be severe operational problems in launching a cryogenic liquid-fueled missile from a surface ship. In addition to safety questions, the fueling process was long and complicated and the missile would have a slow acceleration at takeoff that could give rise to launch difficulties in heavy sea state. The Navy reasoned that a solid propulsion system would be more rugged and reliable and, in March 1956, was authorized to proceed on a solid-fueled backup program; this still, however, had a surface

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*This section is mainly excerpted from the very thorough review article by R.A. Fuhrman given as the 1978 AIAA von Karman Lecture (Ref. 10). That article is also a guide as to what systems information is presently unclassified.

launch as the primary consideration. A strong reason for surface-launch deployment was the state of nuclear weapons technology in 1956. A 3000-lb payload was necessary which resulted in a large missile (gross weight of about 80 tons). A reasonable submarine could only carry about four of these.

A conceptual breakthrough occurred in the summer of 1956 during a National Academy of Sciences study (Project NOBSKA) sponsored by the Office of Naval Research. Dr. Edward Teller predicted dramatic reductions in warhead weight for an acceptable yield, which was quickly calculated to result in a two-stage solid-fueled missile in the 15-ton class. After more Atomic Energy Commission studies resulted in certifying Dr. Teller's prediction, the decision was made in November 1956 to scrap the entire JUPITER idea and proceed to design an entirely new missile which was called the POLARIS. In June 1957 the development was officially approved, but funding for the surface ship application was deferred and never restored.

At about this same time, the FBM submarine characteristics (with 16 missiles per submarine) were approved. As an interim step, several SKIPJACK class SSNs were designated for redesign as the GEORGE WASHINGTON (SSBN 598) class for POLARIS. Design proceeded with the ETHAN ALLEN (SSBN 608) class which were the first submarines designed from the keel up to carry POLARIS missiles. A later, more advanced design became known as the LAFAYETTE (616) class. Table 3 shows some data for these submarine classes and Fig. 7 shows a POLARIS submarine schematic.

b. POLARIS A1. In January 1957, a Special Task Group was formulated to define the system operating envelopes and come up with preliminary definitions of the new system that would take advantage of the projected state of the art. In three months the Task Group had fixed on the missile dimensions and weight. These parameters were never changed during hardware development and are as shown in Fig. 8.

TABLE 3. POLARIS FBM SUBMARINE CHARACTERISTICS (Ref. 10)

	598 CLASS (5 SUBMARINES)	608 CLASS (5 SUBMARINES)	616 CLASS (31 SUBMARINES)
LENGTH	380 FEET	410 FEET	425 FEET
BEAM	33 FEET	33 FEET	33 FEET
SUBMERGED DISPLACEMENT	6,700 TONS	7,900 TONS	8,250 TONS
PROPULSION	STEAM TURBINES POWERED BY WATER COOLED NUCLEAR REACTOR		
CREW	10 OFFICERS 100 ENLISTED	12 OFFICERS 100 ENLISTED	14 OFFICERS 126 ENLISTED
MISSILES	16 POLARIS A1 OR A3	16 POLARIS A1, A2 OR A3	16 POLARIS A2, A3, C3 OR C4
LAUNCH SYSTEM	AIR EJECT	AIR EJECT	GAS STEAM GENERATOR
NAVIGATION	SINS	SINS	SINS

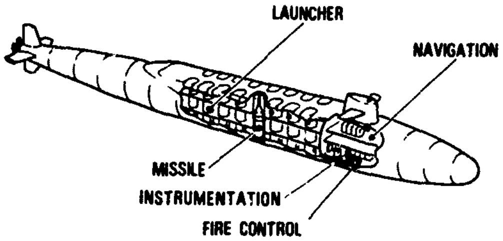
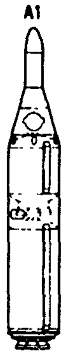


FIGURE 7. POLARIS Submarine (Ref. 10)



- AN INTERIM CAPABILITY
- 1200 NAUTICAL MILES
- 54-INCH DIAMETER
- 28.5-FOOT LENGTH
- 28,500-POUND WEIGHT
- DEPLOYED 15 NOVEMBER 1960

FIGURE 8. The POLARIS A1 (Ref. 10)

Lockheed had been conducting Air Force sponsored subscale reentry vehicle flight tests for some time using the AFX-17 three-stage test rocket. The third stage ignited during reentry and drove the reentry vehicle into the atmosphere at high Mach numbers. The Navy benefited from this activity. It renamed the X-17 as the FTV-3 and continued extensive flight-test programs using a one-third scale POLARIS reentry body.

Early tests at the Naval Ordnance Test Station (China Lake) in 1955 on solid propellant technology demonstrated a significant increase in specific impulse obtained by the addition of finely divided aluminum powder to the propellant.* This result was confirmed in 1956 by the Atlantic Research Corporation and later (mid-1957) reconfirmed by large-scale tests at Aerojet, the POLARIS boost propulsion developer. This assured that the booster contributions to the performance goals could be achieved. The solid rocket program for POLARIS lasted until November 1960 and included static and flight testing of 339 motors. The most frequent problem encountered in the first series of tests was the loss of the carbon throat and exit cone liners from the nozzles. Eventually, molybdenum throat inserts backed by a graphite heat sink and molybdenum liners were used and worked satisfactorily.

During full-scale static testing of the first-stage motor, a serious problem was encountered in the failure of the thrust vector control device. Called the jetevator, this device (as first designed) was simply a solid ring with a spherical inside

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*Author's note: NOTS did not, of course, invent this idea. As early as 1895 the French had demonstrated that adding aluminum or magnesium to gunpowder greatly increased the flame temperature. Dr. Sidney Golden did a Ph.D. thesis (Harvard) on the subject in about 1944 and Dr. Henry Shuey of Rohm and Haas did seminal research at Redstone Arsenal in 1955. In that same year, Thiokol did considerable metal additive work under the BOMARC program.

surface. It was mounted so that it could be rotated past the rim of the nozzle and into the exhaust stream. Originally, it had a blow-back seal to prevent exhaust gases from passing between the jetevator and the nozzle and impinging against the aft end of the motor. The jetevators were made of a molybdenum alloy that is brittle below 400 °F and the jetevators tended to crack early in a firing either from ignition shock or from the first immersion into the exhaust stream. Also, the jetevator bearing tended to overheat. Finally, the blow-back seal caused sticking due to the accumulation of aluminum oxide from the propellant combustion products. A redesign was developed that alleviated the thermal stresses and structural loads, and the blow-back seal was eliminated, but this series of problems delayed the start of the flight program by about two months.

Propellants in both stages were an aluminized polyurethane formulation, although the ratio of aluminum powder to ammonium perchlorate oxidizer differed. The delivered specific impulse was about 230 sec with densities of about 0.061 lb per cubic inch.

A great deal of effort went into the development of underwater launch techniques. The first test of a full-scale dummy vehicle was successfully launched during March 1958 from a submerged platform. Continuing testing was carried out until the final stage, the first underwater launch of a live missile (with a cut-grain first stage) from the USS GEORGE WASHINGTON on 14 April 1960.

Flight testing of [illegible]ypes began at Cape Canaveral on 24 September 1958 with the launching of what was designated the AX series of flights. The first five flights were partial or complete failures for a variety of reasons including excessive base heating. After some quick fixes, the AX-6 flight test was finally successful on 20 April 1959. Another 11 flights were made in the AX series up to October 1959. In September 1959, the next series of flight tests began, designated A1X. Forty flights were made. New and redesigned hardware were continually introduced up to A1X-14. The remaining test vehicles were substantially the same as the production design. On 20 July 1960, the first functional POLARIS was launched from the USS GEORGE WASHINGTON for a successful full-range demonstration. A second missile was also successfully launched three hours later. The POLARIS missile was actually deployed on 15 November 1960.

A unique event on 6 May 1962 is worth noting. A POLARIS A1 missile was launched from the USS ETHAN ALLEN while submerged in the Pacific and its nuclear warhead was detonated on target at the end of its flight. This is the only time any strategic missile from the U.S. arsenal has been fired and its nuclear payload detonated.

c. POLARIS A2. In April 1958, the program to achieve a 1500-nmi range was accelerated and the missile was designated as POLARIS A2. It is schematically shown in Fig. 9. The highest development priority was maximizing the efficiency of the second stage. A savings of inert weight in the second stage would result in a range increment over eight times that of the same reduction in the first stage. Also, the effects of increased specific impulse were more significant in the second stage. The first stage would use the same propellant as the first-stage POLARIS A1 and would be increased in length by 30 inches using the space originally set aside for the launcher's bouyancy compensation tanks.

The major advances in the second stage were the development of a new cast-in-case double-base propellant (nitrocellulose/nitroglycerin), modified by adding aluminum and ammonium perchlorate, and the use of glass-epoxy (filament wound) instead of steel for the motor casing. The composite has over three times the strength-to-weight ratio of steel and thus gave a significant weight savings. In addition, the second-stage engine jetevators were replaced by rotatable nozzles which gave thrust vector control with a smaller loss in axial thrust.

Development flight testing began in November 1960 and the first successful submerged launch came from the USS ETHAN ALLEN on 23 October 1961 off the Florida coast. The A2 was deployed in the 608-Class SSBN submarines starting in June 1962.

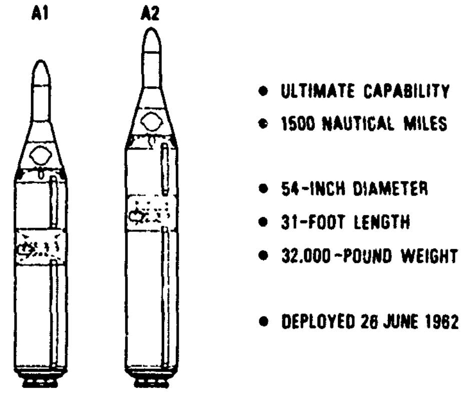


FIGURE 9. The POLARIS A2 (Ref. 10)

d. <u>POLARIS A3</u>. In order to counter the expanding Soviet antisubmarine warfare capability, greater sea room and operating area appeared necessary. A range goal of 2500 nmi was fixed upon together with increased penetrability and accuracy of the payloads. Multiple reentry bodies were adopted and advantage was to be taken of the technology advances in propellants, electronics, materials, and TVC concepts developed throughout the earlier POLARIS programs as well as the comparable Air Force efforts. Designated the POLARIS A3 (shown schematically in Fig. 10), the missile began development in October 1960.

The need to use newer technologies was evident in that the missile had to have a 60 percent greater range but no increase in its overall size. Unlike the case of the A2 versus the A1, there simply was no more available room in the submarine to allow the motors to be lengthened. As a result, the A3 was about an 85 percent new missile.

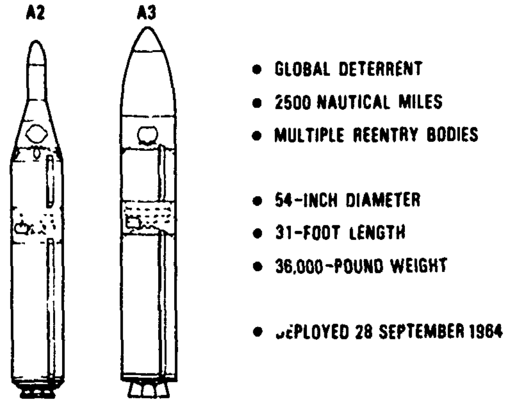


FIGURE 10. The POLARIS A3 (Ref. 10)

The TVC was based on injection of freon into the nozzles. This concept was successfully flight-demonstrated on 29 September 1961 and chosen as the baseline second-stage TVC system two months later. The major advantages of the system were its low inert weight, relative insensitivity to flame temperature and a low constraint imposed on the nozzle design. The first-stage TVC system was changed to rotatable nozzles and the rather cumbersome jetevator concept was completely dropped. The first-stage chamber casing was changed to a high strength glass/resin composite from the original steel. The propellants were changed to formulations with higher specific impulse and density. Because the first-stage motor was designed to operate at considerably higher temperatures and pressures, its nozzle throat material was changed to silver-impregnated tungsten from the molybdenum which was used in the A1 and A2.

The inertial guidance instrumentation and on-board computer hardware were miniaturized and simplified so that they occupied less than half the weight and volume used in the A1 and A2 while increasing component accuracy.

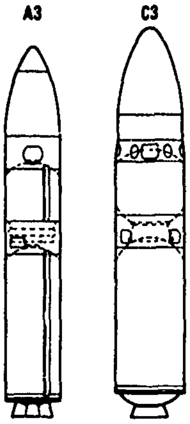
The first A3 flight test was conducted at Cape Canaveral on 7 August 1962 and the production prototype flight test was the 18th of this series. The first launch from a submerged submarine took place on 26 October 1963 and the A3 became operational on 28 September 1964. On 25 December 1964, the USS DANIEL BOONE (SSBN 629) began the first Pacific Ocean operational patrol.

e. POSEIDON C3. By reducing the overly conservative shock mitigation requirement imposed on the POLARIS series of missiles, it was found possible to obtain missile diameters as large as 74 inches (as compared to 54 inches for the POLARIS A series) and still use the same size launch tubes. At the time, increased range did not appear warranted and the new opportunities, opened up by the increased missile diameter, focused on increased payload flexibility and improved defense penetration. In November 1962, the CNO expressed an interest in a hard-target capability (the first time for an FBM system) and in various penetration aids. This hard-target mission requirement was later deleted and the penetration aids were never deployed.

Coincidentally, the Air Force generated a requirement during 1962 for a high ballistic coefficient reentry vehicle that became known as the Mark 12 and development was authorized in late 1963. However, the DDR&E directed that this be a joint Navy-Air Force development where the Air Force application would be the MINUTEMAN III and the Navy application the POLARIS A3 and/or the POLARIS B3 (as the POSEIDON C3 was known in those days). In March 1964, the General Electric Company Reentry Systems Division was funded to develop the Mark 12.

From Navy studies, the B3 payload concepts evolved from multiple Mark 12 reentry bodies to the larger Mark 17 and then finally to a larger number of smaller bodies (simply known as the Mark 3). On 13 January 1966, the DDR&E approved the Mark 3 as the baseline Navy payload and, later, requirements for compatibility with the Mark 12 and Mark 17 were dropped. The new missile (known since January 1965 as the POSEIDON C3) was the first Navy use of the multiple independently targetable reentry vehicle (MIRV) concept, which allows one delivery missile to attack several separate targets.

In addition to increased diameter, the POSEIDON C3 was three feet longer than the A3 and about 30,000 pounds heavier (see Fig. 11). It clearly needed a redesigned submarine and 31 of the Navy's 41 FBM submarines were scheduled for shipyard modification. The first to be converted was the USS JAMES MADISON (SSBN 627) which made the initial POSEIDON deployment on operational patrol in 1971.



- GLOBAL DETERRENT
- MIRV
- RANGE-PAYLOAD OPTIONS

- 74-INCH DIAMETER
- 34-FOOT LENGTH
- 64,000-POUND WEIGHT

- DEPLOYED MARCH 1971

FIGURE 11. The POSEIDON C3 (Ref. 10)

Largely because of the MIRV concept, the C3 has an equipment section which contains the payload (reentry bodies and guidance system) and a post-boost control system (PBCS) and which acts as sort of a third stage. The post-boost control system has a solid propellant gas generator and an associated steering capability. After the equipment section separates from the second stage, the PBCS allows the guidance system to maneuver the equipment section and to eject reentry vehicles into ballistic trajectories to individual aim points.

The C3 propulsion developer was the joint venture of Thiokol and Hercules. Unlike any previous Navy missiles (which all had four nozzles on each stage), the C3 motors each had a single movable nozzle which was activated by a gas generator (which also supplied roll control). The propellant for the first stage was a composite and that for the second stage a double-base. In general, the C3 propulsion system represented a conservative approach.

After a 20-vehicle flight-test program, the POSEIDON C3 system was first deployed during March 1971.

f. <u>TRIDENT C4</u>. By about 1970-1971, the threat pendulum had swung toward antisubmarine warfare. For several reasons, in addition to survivability, it was desirable to have an ICBM-range capability. On 14 September 1971, approval was given for the Undersea Long-Range Missile System (ULMS). Besides much longer range and other improvements as compared to POSEIDON, an ULMS requirement was that the new missile system had to be compatible with the POSEIDON submarines in their existing configuration. Early in 1972, the ULMS-1 was redesignated TRIDENT I(C4), a schematic of which is shown in Fig. 12. Full-scale engineering development was authorized in October 1973. The first successful C4X-1 flight test took place on 18 January 1977.

The range goal was approximately twice that of the C3 with a comparable payload. This had to be accomplished with no increases in dimensions so considerable innovation and use of new technology were necessary. The most obvious step was to introduce a third-stage booster that was mounted in the center of the equipment section. In addition, propellants were picked with increased energy per unit volume; higher internal pressures were achievable by use of higher strength Kevlar composites for the motor cases, and the use of pyrolytic graphite nozzle throat sections with a combination of low recession rates and light weight.

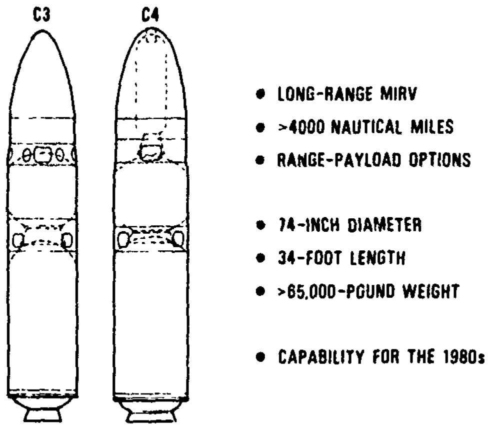


FIGURE 12. The TRIDENT I(C4) (Ref. 10)

No thrust termination system was included. Instead, a concept called generalized energy management system (GEMS) was used whereby all stages are burned to burnout, shaping the trajectory to use all the energy. The PBCS operates at higher temperatures than that of the C3, which required more refractory materials of construction. To improve the launch-phase aerodynamic performance, an extendible aerospike was included to reduce (by about 50 percent) the high drag of the blunter nose fairing required by the third stage.

Inert weights were reduced by fabricating certain structures from graphite-epoxy composites which give about a 40 percent weight saving compared to similar aluminum structures.

Some of the most remarkable advancements were made in the missile electronics particularly in the flight control package. The microelectronic technologies (new at that time) of beam lead, dielectric isolation, and low-power Schottky TTL were utilized in these electronic devices.

The TRIDENT submarine is designed to carry 24 (instead of of 16) C4 missiles and allows for future missile systems growth in both length and diameter. The first TRIDENT submarine, the USS OHIO (SSBN 726) is currently under construction.

g. Comparison Summary. A schematic comparison of the five versions of submarine-launched ballistic missiles is shown in Fig. 13. A similar comparison of the three submarine platforms and a typical World War II submarine is given in Fig. 14.

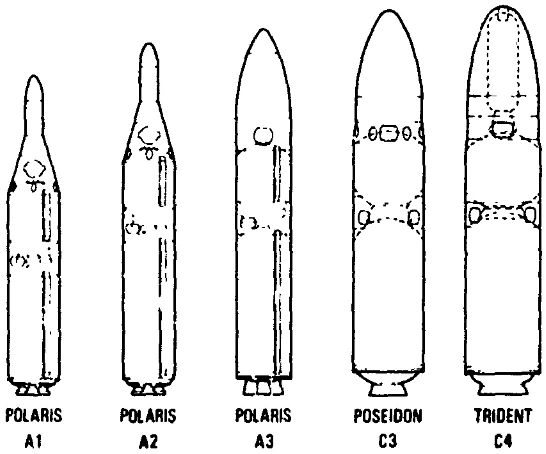


FIGURE 13. Fleet Ballistic Missiles (Ref. 10)

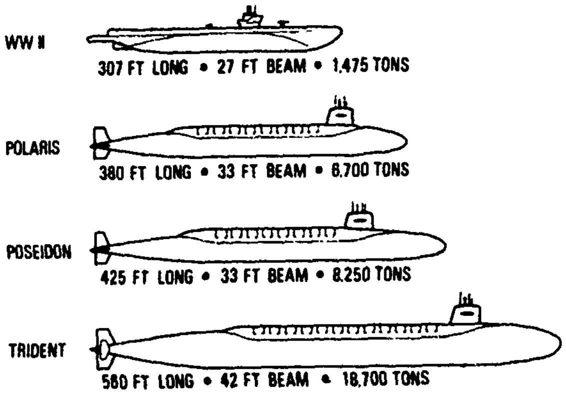


FIGURE 14. Submarine Comparison (Ref. 10)

2. Critical C4 Materials and Structures Technologies

a. Introduction. The ULMS (Undersea Long-Range Missile System) program, later designated as TRIDENT C4, had sufficient time to permit a reasonably thorough examination and evaluation of new technology concepts that might be used on the new missile. Whereas prior systems were highly schedule-oriented in order to meet urgent operational requirements, there was no such pressure for TRIDENT. POLARIS and POSEIDON were operational in 41 submarines and there was no immediate danger of them becoming a less-effective deterrent.

Actually, technology studies at LMSC, funded by SSPO, were started about 18 months before the ULMS program received DoD approval (in September 1971). This appears to be the first time that any of the FBM programs had the luxury of a 6.3A type of effort for any length of time. It evolved in the following manner.

Extensive studies of the next-generation strategic ballistic weapon system were conducted by IDA during 1967 under the STRAT-X program. In this study, the major emphasis was on alternate basing concepts and a conservative state of the art was assumed for the missiles that were synthesized. The specified payload was carried to the specified range simply by increasing the size of the missile to that required to accomplish the mission. Naturally, if the missiles were to be carried inside the pressure hull, this assumption often resulted in some pretty large submarines and thus STRAT-X also investigated concepts such as externally stored missiles. After completion of STRAT-X, the Navy continued weapon and missile system sizing studies well into 1969. It became apparent that the original missile technology assumptions needed updating and a more optimistic projection into the near future.

Thus, in about March 1970, SSPO sponsored LMSC in a first phase of a missile technology study which was designated the

Missile System Technology Evaluation (MSTE). An in-house LMSC effort, the purpose of MSTE was "to identify and evaluate the new and expanding technology that offered potential benefits to sea-based strategic missile systems both constrained and unconstrained by present SSBN configuration" (Ref. 11). An overview of the entire study indicates that most of the interesting performance improvement items were in the area of boost propulsion. By and large MSTE was a paper study and financially very modest.

Shortly after the completion of MSTE (near the end of CY 1970), the second phase was started, designated the Missile System Advanced Technology (MSAT) program. This was more hardware-oriented and was to investigate the more important technology areas identified in MSTE primarily by competitive subcontracts. It was originally planned that each selected technology would be pursued from two to four years leading eventually to combined technology demonstration tests. However, after about a year, MSAT was superseded by the approval of the Engineering Development Program (EDP) for ULMS, which actually got started about January 1972. The more leisurely technology program was not to be.

The total dollars appropriated for MSTE and MSAT together were about $1.0-2.0 million (mostly during the one year of MSAT) but an unknown amount of IR&D effort was put in by all companies involved. As a point of trivia, the authors were not able to ascertain just when the name ULMS was officially changed to TRIDENT; there are people today who still refer to the missile as ULMS. The first 18 months of the EDP were devoted to predesign technology and, in effect, were a greatly intensified and accelerated continuation of MSAT. The total funding trend for the TRIDENT missile EDP is as shown below (from FYDP Historical Summary, 23 May 1978, Update, OSD):

Fiscal Year	1972	1973	1974	1975	1976	1977
PE6.4362 ($ million)	65	348	526	640	725	568

The major changes in going from the C3 to the C4 are the following:

- Addition of a third stage
- Addition of the aerospike
- Advanced Boost Propulsion System
- Advanced Post-Boost Control System
- Stellar inertial guidance update system
- Advanced electronics
- Advanced payload (Mark 4 or 500)
- Advanced composites forward structure
- Generalized Energy Management System (GEMS)

In addition, an extendible exit cone (EEC) concept was seriously considered for the nozzles but dropped (approximately at the end of CY 1973) largely due to uncertainties in reliability and cost. Present thinking is that the EEC will be used on both future TRIDENT designs and on the MX. Of the above major changes, the ones which involve advanced materials and structures technologies to the greatest degree are the boost and the post-boost propulsion systems, the payload, and the forward structures. These will now be further discussed.

b. Boost Propulsion Systems. The approximate timing sequence of the efforts described in the Introduction (pertaining to propulsion) is indicated in Fig. 15. Table 4 compares the parameters and materials used in the A3, the C3, and the C4 propulsion systems. For the sake of completeness, it should be noted that the A1 had molybdenum as the throat material for both stages and the A2 substituted pyrolytic graphite (PG) for molybdenum on the second stage.

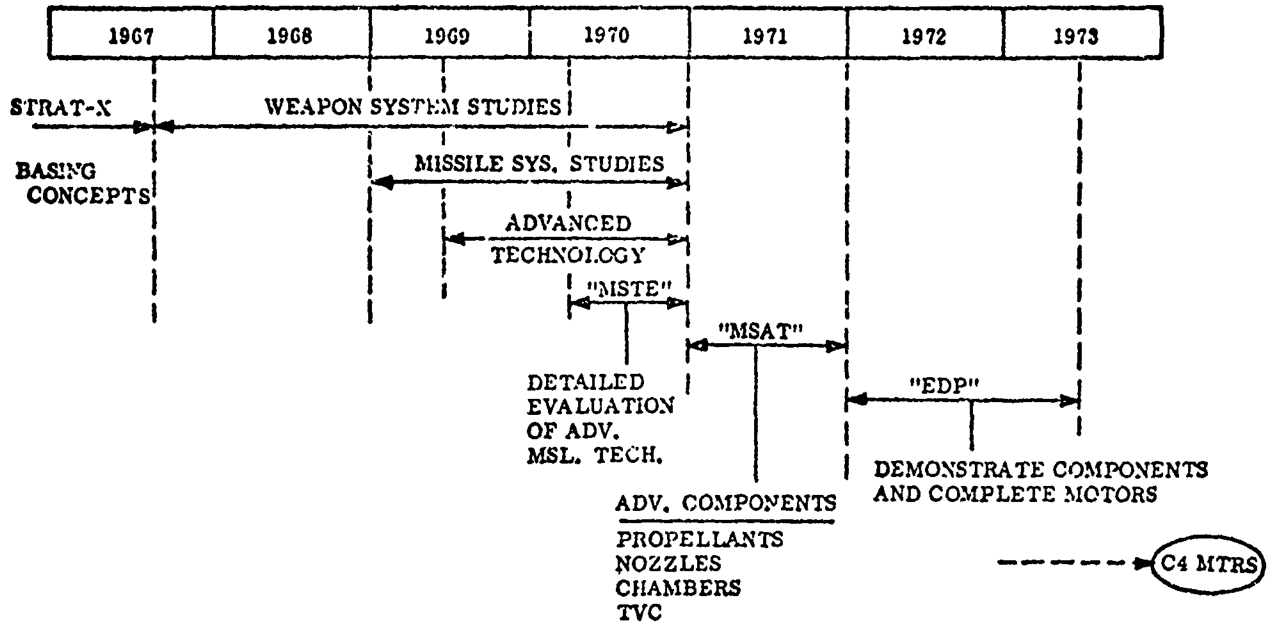


FIGURE 15. TRIDENT I (C4) EVOLUTION (Ref. 11)

TABLE 4. PROPULSION SYSTEM PARAMETERS OF A3, C3, AND C4
(Courtesy of C.A. Zimmerman, Lockheed Missiles and Space Company)

	A3P FS	A3P SS	C3P FS	C3P SS	C4P FS	C4P SS	C4P TS
CHAMBER							
MATERIAL (FIBER)	S-901	S-901	S-901	S-901	KEVLAR 49	KEVLAR 49	KEVLAR 49
COMPOSITE STRESS LEVEL (PSI)	94,000	115,000	110,000	120,000	115,000	139,000	112,000
MAXIMUM OPERATING PRESSURE (PSI)	940	400	920	410	1570	1285	1218
SKIRT/SKIRT LENGTH (IN)	155	55	157	59	157	69	97
DIAMETER (IN)	54	54	74	74	74	74	30
NOZZLES							
NUMBER	4	4	1	1	1	1	1
THROAT MATL	Ag/W	ATJ	GRAPHITE PHENOLIC	GRAPHITE PHENOLIC	P.G	P.G.	P G.
TVC	ROTATABLE	FLUID INJ	FLEX JOINT (STEEL REINFORCEMENTS)	FLEX JOINT (STEEL REINFORCEMENTS)	FLEX JOINT (GLASS EPOXY REINFORCEMENTS)	← SAME	← SAME
TYPE	GRAPH CLOTH/ CARBON PHENOLIC	GRAPH CLOTH/ GLASS PHENOLIC	GRAPHITE, CARBON ASBESTOS PHENOLIC	GRAPHITE, ASBESTOS PHENOLIC	CARBON/CARBON, GRAPHITE/CARBON PHENOLIC GRAPHITE EPOXY	← SAME	← SAME
MOTOR							
NOZZLE WT (EACH)	365	43	765	425	657	368	109
CHAMB/INSUL WT	1547	410	2,156	720	2088	913	175
PROP WEIGHT	20,777	8844	38,800	15,880	39,056	17,646	3748
TOTAL WEIGHT	23,895	9492	41,860	17,115	42,018	19,034	4070

The rationale for the use of PG on the A2, going to ATJ graphite on the A3 (which appears to be a step backward in technology), was difficult for either the authors or LMSC personnel to reconstruct. Most likely, the complicated assembly of the PG plate design was causing reliability problems that overbalanced the better performance of the PG in resisting thermal cracking and erosion. Clearly the PG was not essential to the A2 second stage since the A3 second stage had similar operating temperatures and pressures and the ATJ obviously worked. The reader will recall that in the A3, boost performance advances arose mainly from redesigning the first stage where it did become necessary to change the nozzle throat from molybdenum to silver-infiltrated tungsten because of increased temperature and pressure. On the POLARIS series, each stage had four nozzles which naturally were much smaller than the single nozzle design of the POSEIDON and TRIDENT. Therefore, it may have been possible, within economic bounds, to selectively pick only the very best ATJ graphite billets for actual use.

In the pre-TRIDENT MSAT effort, the high-energy propellant subcontractors to LMSC were JV (the Joint Venture between Thiokol and Hercules) and UTC (United Technology Center, now United Technologies Chemical Systems Division). They both chose ethylene propylene diene monomer (EPDM) as the most promising casing insulation candidate material. Both were also heavily oriented toward pyrolytic graphite for the throat either in the form of washers or of coatings (presumably over conventional graphite among other substrates--this point was never clarified). Atlantic Research Corporation (ARC) was funded during MSAT (and part of the EDP) to continue the "pyrostrand" work started earlier. Pyrostrand is a carbon filament-reinforced pyrolytic graphite composite. Scale-up to large sizes was one of the problems (in the context of the C4 schedule) that caused this to be eventually dropped.

The goal for the nozzle section erosion rate was 1.0 mil per second, although it was recognized that was probably unachievable with state-of-art materials. However, PG coatings came pretty close in small-scale (about 1.0 to 4.0 inch diameter) firings--with an average rate between 1.0 and 1.5 mils/second. Part integrity could not be achieved partially due to thermal expansion compatibility with the substrate. To improve this problem, carbides of silicon, hafnium or zirconium were codeposited with the PG; silicon carbide proved to be the best from a part integrity standpoint. However, then the erosion rates went up by a factor of three. Eventually the coating concept was dropped but, if a little more time could have been spent, it is by no means clear that it wouldn't have worked. Scale-up might actually have made the process easier.

In small-scale firings, PG washers exhibited acceptable erosion rates (1.0 to 4.0 mils/second) and maintained structural integrity. Based on this, all three competing subcontractors (Aerojet had become the third) chose this system for full-scale firings of all three stages. For the throat approach and backup sections, several carbonaceous materials were evaluated in small-scale tests; the two best were a two-dimensional carbon-carbon composite and G-90 graphite. The latter was a commercial graphite (sold by Carborundum) of large grain size but reimpregnated to achieve the relatively high density of 1.90 g/cc (which is where the appellation "90" came from). The JV and UTC chose the C/C composite for both the front and back ends while Aerojet elected to use G-90 for the front and C/C for the back.* Since JV eventually became the motor subcontractor, G-90 was never used; to the best of the author's

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*There is some confusion, in the available documentation, as to just when JV chose the C/C for the front end. One statement says the decision was made at the finish of small-scale testing. Another says that the first full-scale third-stage test failed, induced by the G-90 approach ring. By this account,
(continued)

knowledge, G-90 has never been significantly used on any weapons system and is no longer being sold commercially.

All three subcontractors evaluated the use of 2-D C/C composites for the exit cone in small-scale tests. As compared to the conventional ablative liner/insulator/ambient-temperature structure configuration, the C/C composite offered the potential of simplicity, lower weight and lower erosion rate (i.e., better nozzle shape retention). Although small-scale firing data looked encouraging, only UTC opted to use the C/C composite for full-scale testing. It should be noted that only one third-stage UTC firing (out of seven) showed any failure of the C/C exit cone. That was traced to a manufacturing lay-up deficiency, which merely pointed up a need for improving inspection and quality control techniques. It appears that the 2-D C/C composite has a high potential for future use. In the meantime, JV and Aerojet took the more conservative approach of using the conventional ablative liner/insulator/structural shell configuration, which eventually became the C4 production design. An improvement over the C3 was the use of a graphite-epoxy composite for the structural shell (instead of aluminum).

Figures 16 and 17 show schematics of the JV nozzle design after the first 18 months of the Engineering Development Program and the final flight design. A major problem with the early design was that of throat retention in static firings. Prior to the initial flight designs, three first-stage and three second-stage static firing failures occurred due to throat problems. Basically, excessive thermal loads combined with, and resulting in, deflection of components in the throat

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the G-90 was then replaced by C/C in all stages and no further problems in the subsequent 12 full-scale firings (7 third, 4 second, and 1 first stage) were experienced in the throat region. In any case, JV wound up using C/C for the front and back sections in all stages.

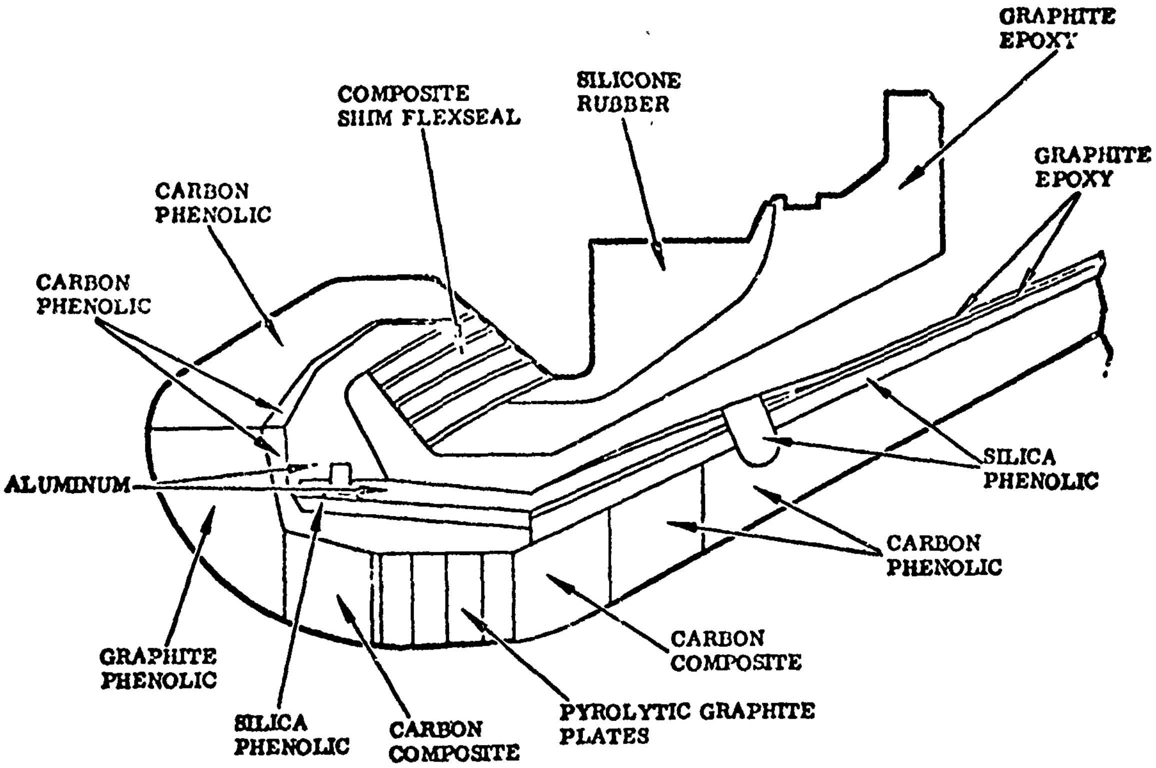


FIGURE 16. Early C4 Nozzle Design Schematic (Ref. 11)

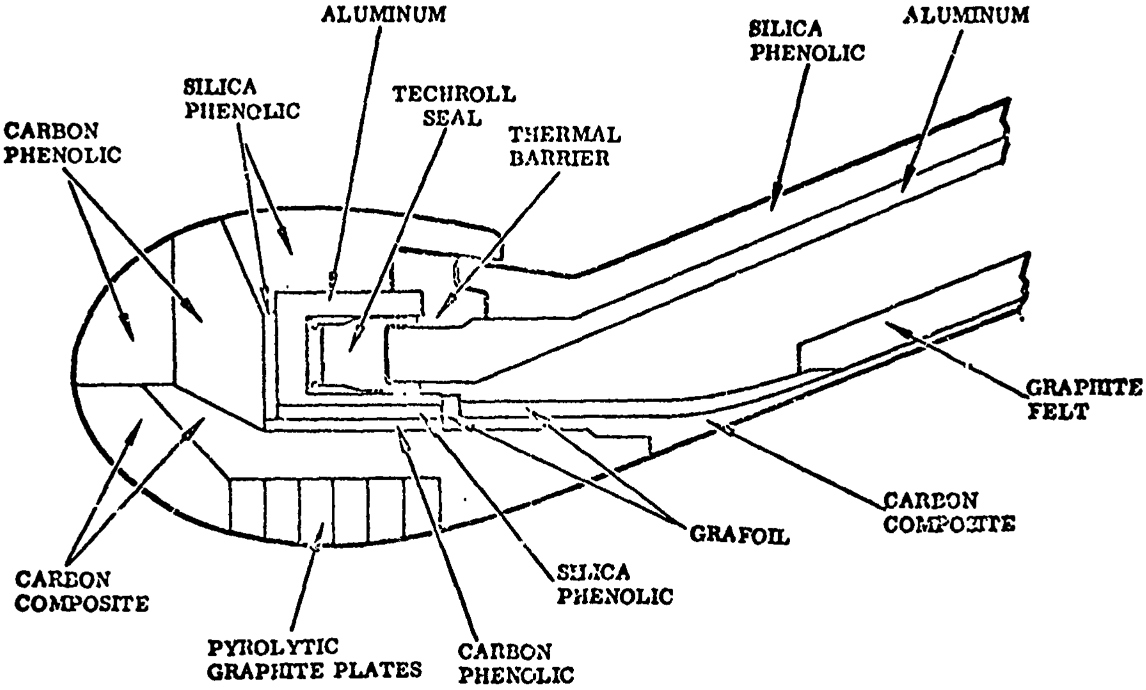


FIGURE 17. Final C4 Nozzle Design Schematic (Ref. 11)

area caused the throats to be ejected (or unacceptably degraded). Reducing the number of PG washers and replacing materials where needed for better mechanical and thermal expansion properties eventually resulted in the satisfactory flight design shown in Fig. 17.

Nozzle throat erosion rates in all full-scale motor firings were generally somewhat higher (3 to 7 mils/sec depending on stage and nozzle design) than anticipated based on small-scale results. These rates, however, were not bad enough to be seriously detrimental to motor performance. Except for the experimental mixed pyrolytic graphite/carbide coatings mentioned earlier, no lightweight material was as good as the PG from the erosion standpoint; certainly the availability of good reliable PG plates of large size was a major factor in allowing the C4 to come close to its performance goals in the scheduled time. The throat ejection problems mentioned above were not because of any difficulties with the PG material; rather, they were the result of the complicated nightmarish design required to hold the throat together. During the period when throat ejection problems were the most severe, a backup program was initiated based upon three-dimensional C/C components. Such a materials systems had undergone extensive reentry vehicle nose-tip testing and is discussed more fully in Section VI-D of the present paper. 3-D C/C composites would permit integration of the throat and entrance and/or exit sections into one or two pieces and avoid the existing complexity of design. However, before a high degree of confidence in such materials could be established, the problems faced by the baseline PG plate nozzles were resolved and 3-D program was deemphasized. Although a great deal more technology R&D is needed (primarily in large shape processing, quality control, nondestructive testing, and improving erosion resistance), it is generally believed that future large solid systems (including MX) will utilize 3-D C/C composites.

The flex seal concept of thrust vector control (alternate layers of reinforcement and elastomer as shown in Figs. 16 and 17) was used on the C3. The major improvement in going to the C4 was the replacement of the steel reinforcements by glass cloth and epoxy resin with carbon phenolic joint thermal protectors. With some minor design changes, this resulted in lower weight and reduced torque as compared to the C3 joint.

For the motor chambers, the major structural change was in the casing materials. Table 5 shows the three composites considered for the C4 compared with the S-901 fiberglass used on the C3. The key parameters are the specific strength and specific modulus which should be as high as possible. X-2285 is a newer fiberglass, Type A carbon is a carbon fiber/resin and PRD-49 is the proprietary duPont fiber later known as Kevlar 49. Eventually Kevlar 49 was chosen for all three stages. The epoxy resin system, designated HBRF-55A, was used for the first and third stages; a special more flexible resin, HBRF-241, is used on the second stage. The adoption of Kevlar was a very important step in that it allowed much higher chamber pressures with little or no weight increases (this also meant that a low nozzle throat erosion rate became even more critical).

The nondestructive testing efforts during the EDP were (1) basic development of advanced techniques for inspectibility of reduction of time and cost of inspection and (2) determining wherever existing NDT techniques were inadequate to verify conformance to specification requirements. In addition, some effort was devoted to NDT technique engineering of the more basic NDT applications.

The following programs were carried on during the Engineering Development Program (EDP).

High-Energy Radiography - High-energy radiography (betatron and linac) has historically been used for the inspection of POLARIS and POSEIDON motors. The C4 propellant is more dense

TABLE 5. C4 BOOST PROPULSION CHAMBER MATERIALS PROPERTIES (Ref. 11)

	S-901 (C3)	X-2285	TYPE A CARBON	PRD-49-III
FIBER DENSITY, LB/IN.^{3}	0.090	0.088	0.065	0.053
ROVING TENSILE STRENGTH				
ABSOLUTE, X10^3 PSI	570	590	434	534
SPECIFIC*, X10^6 IN.	6.33	6.70	6.68	10.08
ROVING TENSILE MODULUS				
ABSOLUTE, X10^6 PSI	12.2	13.4	32	20
SPECIFIC*, X10^6 IN.	135.6	152.3	492	377.4
TYPICAL COMPOSITE DENSITY**, LB/IN.^{3}	0.074	0.072	0.057	0.050
ATTAINED FIBER STRENGTH IN COMPOSITE				
ABSOLUTE, X10^3 PSI	450	500	310	350-400
SPECIFIC*, X10^6 IN.	6.08	6.94	5.44	7.00-7.55
INTERLAMINAR SHEAR, PSI	11,200	10,700	8,000	6,600 - 9,200

*SPECIFIC STRENGTH OR MODULUS $= \frac{\text{ABSOLUTE STRENGTH OR MODULUS}}{\text{DENSITY}}$

**AT 65% FIBER VOLUME

and will exhibit different attenuation characteristics; therefore, controlled experiments were performed to demonstrate radiographic resolution and sensitivity.

Thermography - Thermography (infrared) techniques are being investigated on a laboratory basis. Results of this work may enable the utilization of the technology on full-scale hardware.

Acoustic Emission - Acoustic emission is a promising technique for chambers that may enable one to predict the ultimate burst pressure for the chamber of its next pressurization. This information may be derived from the rate and amplitude of emission and the triangulation of the stress wave.

X-ray (Real-Time) Imaging - X-ray imaging with high-energy X-rays (1 to 25 MeV) has not been practical to date; however, with hew low-light-level TV cameras, phosphors, and image amplifier tubes, it is felt that real-time viewing of large motors can be achieved.

Foil Phosphor - Foil phosphor technical results indicate that an improvement in conventional film radiography may be possible using other metals or metal/phosphor intensifying screens. While sensitivity increases of up to two to three times that of lead screens have been demonstrated using the metal/phosphor screens, image quality under these conditions has not yet been investigated.

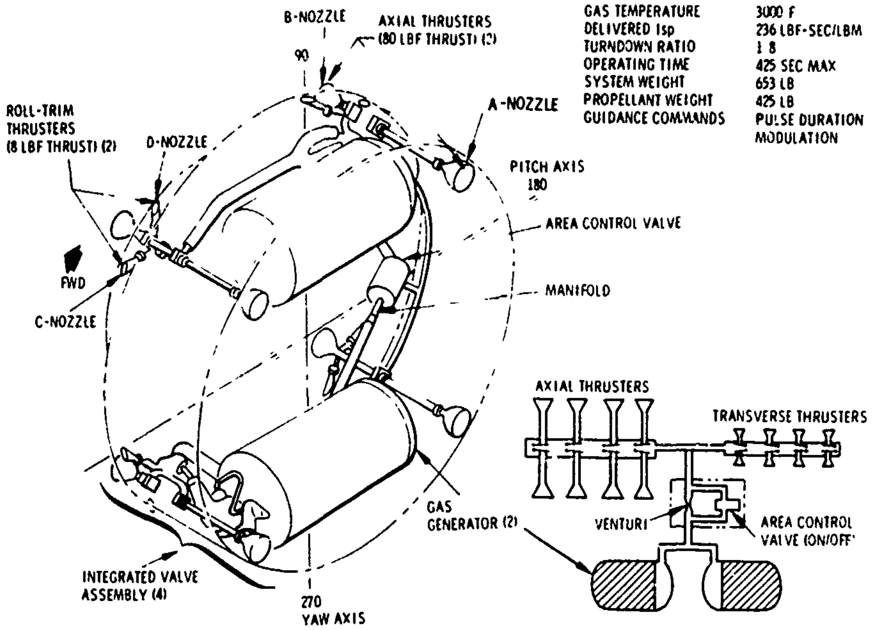
Acoustic Holography - Acoustic holography results in an inspection technique which produces a visual image of the internal condition of the motor being examined. This technique uses ultrasound to detect and visually display the presence of any anomalous condition in the material. The visual image presented conceivably shows the position and orientation of all flaws. The detection of cracks, voids, nonbounds, and other anomalous conditions may be rapidly and reliably determined.

<u>*In-Situ* Sensors</u> - *In-situ* sensors are being evaluated for possible placement within the grains of C4 motors. These can be activated remotely by an FM antenna and measure the propellant elastic properties and weakening of the insulation-propellant bond.

<u>Image Enhancement (Radiographic Film)</u> - Image enhancement techniques are being developed which allow the operator to perform successfully in real-time the enhancement of 14x17" radiographic film. New methods for signal and image enhancement allow the operator to perform each processing step by turning a knob on a console, and the cumulative results are displayed instantaneously on a screen in front of him.

c. <u>Post-Boost Control System (PBCS)</u>. Attitude, direction, and velocity of the C4 post-boost vehicle (equipment section) are controlled by a gas reaction PBCS that is similar in concept to that of the C3, using a dual-pressure-level solid-propellant gas generator system. However, there is little or no similarity between the PBCS hardware of the C3 and C4. This is because the C4 system has a higher energy gas generator and the PBCS must operate at 3000 ^{o}F for about seven minutes. In the C3, the maximum temperature is about 2200 ^{o}F which permits the use of cobalt-base superalloys. For C4 conditions refractory metals must be used throughout. A schematic of the PBCS is shown in Fig. 18.

Table 6 shows the composition and exhaust product of the propellant. Table 7 shows the composition of the refractory metal alloys used for the PBCS. It should be noted that the columbium and tantalum alloys require a coating for protection against the oxidizing gaseous species. The coating used was hafnium disilicide ($HfSi_2$) that required special application techniques. It was never quite clear why the Cb and Ta could not be directly siliconized, but in any event the $HfSi_2$ coating worked. The molybdenum alloy (TZM) did not require a protective coating for corrosion resistance but was carburized to prevent galling at the joints.



GAS TEMPERATURE	3000 F
DELIVERED Isp	236 LBF-SEC/LBM
TURNDOWN RATIO	1 8
OPERATING TIME	425 SEC MAX
SYSTEM WEIGHT	653 LB
PROPELLANT WEIGHT	425 LB
GUIDANCE COMMANDS	PULSE DURATION MODULATION

FIGURE 18. Post-Boost Control System (Ref. 12)

TABLE 6. C4 POST-BOOST CONTROL SYSTEM GAS GENERATOR PROPELLANT--ARCADENE 311B (WS 16300)

COMPOSITION	WEIGHT PERCENT
HMX, Class 1	55.12
HMX, Class 5	29.68
Carbon Black	0.05
HTPB/IPDI	15.15
(A0702 Antioxidant	1.00)
	100%

THEORETICAL PARAMETERS
(P = 500 psia, ε = 25:1)

Flame Temperature, °F	3063
Specific Impulse, lb-sec/lb	259.4
Characteristic Velocity, ft/sec	4567.4
Density, lb/in.^{3}	0.059

EXHAUST PRODUCTS
(P = 500 psia, ε = 25:1)

PRODUCT	CHAMBER	EXHAUST
CH_4	0.0005	0.0344
H_2	1.8766	1.6577
N_2	1.1499	1.1505
CO_2	0.0186	0.4247
H_2O	0.0693	0.2219
CO	2.2096	1.2449
C(s)	0.0000	0.5259 (6.4% by weight)

TABLE 7. C4 PBCS ALLOYS OF CONSTRUCTION

	Cb 103	Ta 10W	TZM
Composition	10% Hafnium 1% Titanium	10% Tungsten	5% Titanium .1% Zirconium
Density, lb/in^3	.320	.608	.370
Melting temperature, °F	4260	5495	4730
Strength at 3000 °F (2% yield in 10 min), psi	2500	8500	9000
Resistance to 3000 °F gas	Needs Coating	Needs Coating	No Coating Required

Figure 19 shows a schematic of one of the four integrated valve/nozzle assemblies and Fig. 20, showing one of the outlet components from the gas generator, is meant to illustrate the difficulties of the forming and joining processes. The difficulties were extreme and pushed refractory metal technology very hard. For reasons not relevant here, the use of liquid gas generators might have alleviated these conditions but this was generally unacceptable to the Navy. Therefore, the existence of an advanced state of refractory metal technology was essential for the successful performance of the PBCS, which in turn was one of the major advances necessary for the successful performance of the C4 missile.

d. Reentry Vehicle (RV): Mark 4. The discussion of the reentry vehicles will be quite brief in order to keep the present paper unclassified. While a great deal of innovative design went into the Mark 4, only a few advances in materials were critical. Part of the reason for this (as compared, for example, to the Air Force RVs) is that the C4 does not have as rigorous accuracy requirements.

By way of general background, the tabulation below shows the external materials used in the RVs for the various FBM systems. When a beryllium (Be) heatshield was used, there is

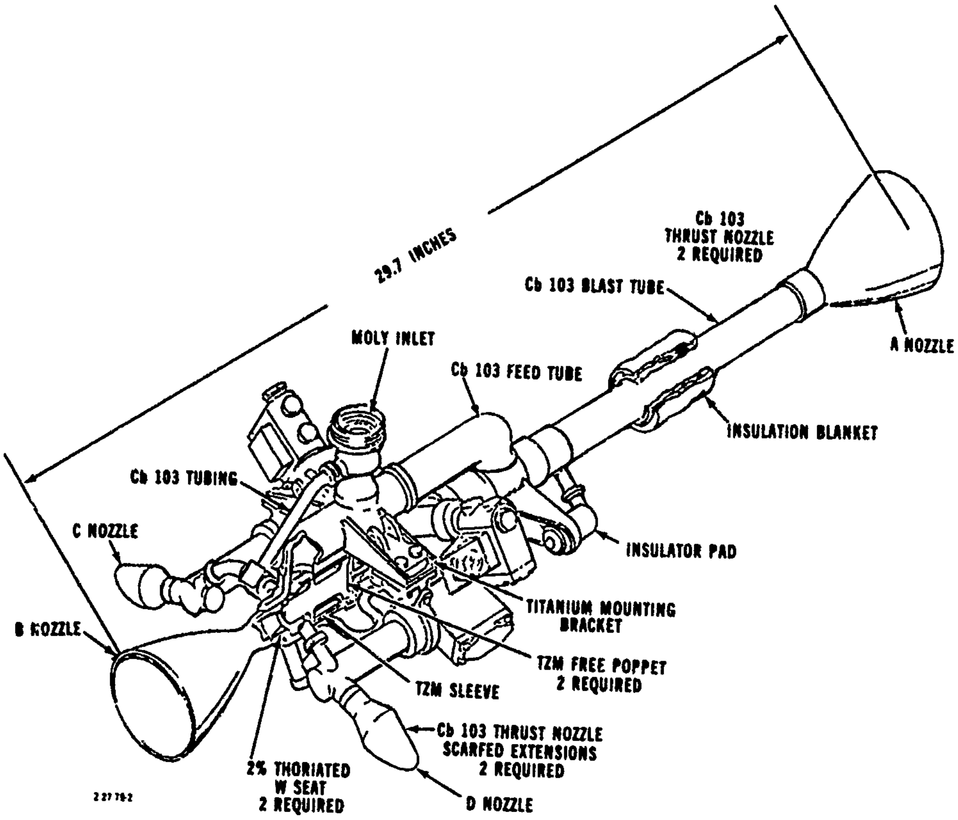


FIGURE 19. C4 Integrated Valve Free Poppet with Flapper Pilot (Courtesy of C.A. Zimmerman, Lockheed Missile & Space Company)

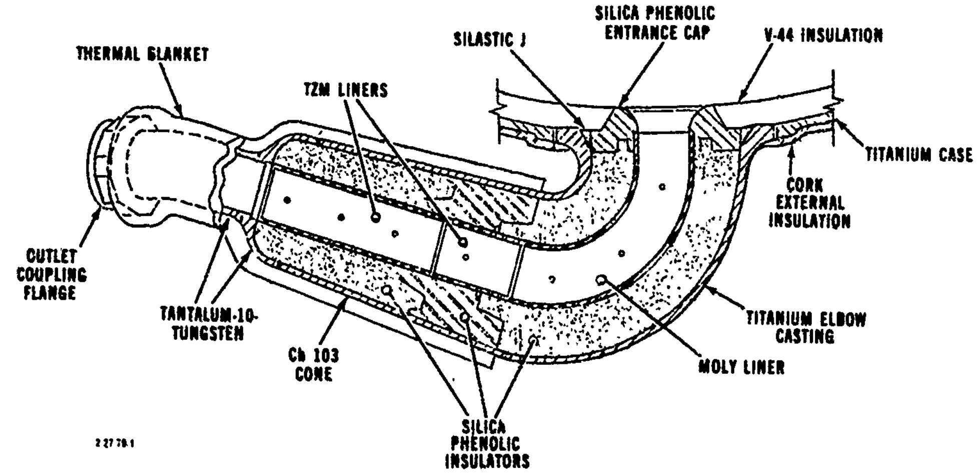


FIGURE 20. C4 PBCS Gas Generator Outlet Components (Courtesy of C.A. Zimmerman, Lockheed Missile & Space Company)

of course no substructure. The vehicles with charring ablators all had an aluminum substructure. The Mk 500 will not be discussed.

	Mk 1	Mk 2	Mk 3	Mk 4	Mk 500
Nose tip	Be	Be	ATJS	CMT	C/C
Heatshield	Be	Nylon/ Phenolic	Be	Carbon/ Phenolic	Carbon/ Phenolic
Antenna Window	Stub	BeO	Silica	AS3DX	Stub

A significant materials change in the Mk 4 (as compared to the Mk 3) is the nose tip. The CMT graphite is a fine-grain material produced in a dedicated Controlled Line Facility (CLF) owned by the Navy and run by Union Carbide Corporation. It was developed to have better and more uniform stuctural, thermal, and mechanical properties than the ATJS used on the Mk 3. The ATJS flight-test performance has not been entirely satisfactory and the CMT will also be retrofitted on the Mk 3. It should be realized that CMT is not so much a "new" kind of graphite as it is the product of a process line design with unusually rigorous quality control.

The tape-wrapped carbon phenolic heatshield (bonded to a thin-wall aluminum substrate for the shell) is similar to the heatshields used by the Air Force, although the carbon filler particles have been eliminated. AD3DX is the commercial designation for a silica body reinforced with a three-dimensional weave of fused silica fibers (sometimes incorrectly called quartz fibers). This is also similar to one of the window materials currently used by the Air Force.

e. Missile Structure. The structure includes those portions of the missile that connect the stages (interstage and adapter sections); provide support and mounting provisions for the guidance, electronics, and other elements that make up the third-stage section (equipment section); and a jettisonable nose fairing and cap to provide the desired hydrodynamic and aerodynamic shape. The interstage section, which joins the first- and second-stage motors, is similar in construction to that of C3, with an aluminum alloy shell and interchangeable field joint rings welded at each end of the section. The nose fairing, as on C3, is laminated Sitka spruce and aluminum with aluminum end rings. The nose-cap structure is a phenolic fiberglass shell with an aft ring machined from an aluminum rolled-ring forging. Locks in the ring properly position and retain the nose cap on the fairing structure. Mounting provisions for an aerodynamic spike are contained in the cap. The basic structural concept of the adapter section is a 5000-series-type aluminum material semimonocoque shell with field joint rings welded to each end of a cylinder.

The equipment section (Figs. 21 and 22) is a graphite-epoxy cone design with center mounting and separation provisions for the third-stage motor. Early studies had suggested that significant range gains could be obtained by the use of graphite-epoxy instead of metal in the equipment section structure. Initially, composites made from unidirectional tape were created, but subelement testing showed that fabrication costs and product and mechanical property reproducibility required improvement. These problems were attributable to the use of 3-inch-wide prepreg tape, which, though excellent for many aircraft and spacecraft applications, was deficient for the squat conical C4 cone structure.

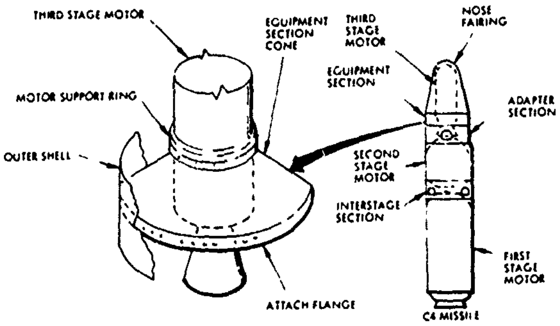


FIGURE 21. C4 Equipment Section Cone (Ref. 13)

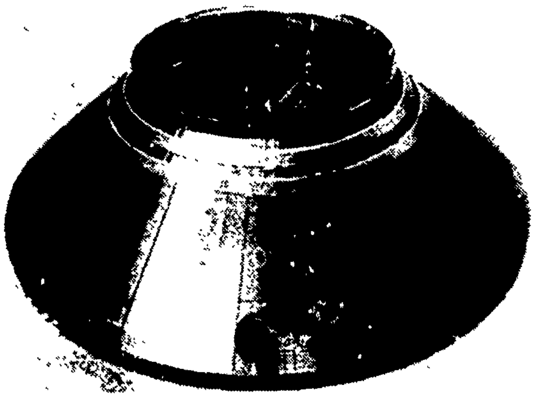


FIGURE 22. Equipment Section Cone (Ref. 13)

In June 1973, 42-inch-wide woven graphite cloth (broadgoods) impregnated with epoxy resins had reached the point where it could be introduced into the development program. Initial manufacturing studies confirmed its excellence, resulting in a 55 percent reduction in lay-up time relative to tape and fabrication cost reductions of about 35 percent. Additionally, improved mechanical property reproducibility, product control, and fabrication of structures not feasible with tape were demonstrated.

The cone consists of graphite-epoxy (cloth) facesheets on glass-reinforced phenolic honeycomb core, with integral graphite-epoxy (tape/cloth) end rings. Two aluminum Z-rings are provided on the aft side of the cone for mounting equipment and cabling. The outer skin is an aluminum shell with bayonet-type field joints at each end. Nose fairing and adapter sections are separated by a mild detonating fuze (MDF) supported by aluminum backup rings.

The payload is supported by an aluminum inboard support ring and outboard titanium tubes located by a graphite-epoxy (tape) honeycomb interface panel.

The third-stage motor is attached to the equipment section by an aluminum ring. Third-stage separation is accomplished by zipcord (MDF inside a stainless-steel tube).

The inner eject cylinder, which aids in guiding the third-stage motor during separation, is made of graphite-epoxy with graphite-epoxy stiffening rings.

Design and analysis required the development of computer programs and techniques particularly suited to the characteristics of composite materials. The application of these tools resulted in a balanced design that satisfied the structural requirements and avoided unnecessary additional inert weight.

In addition, a 40 percent weight saving in electronic housing has been realized by the development of plastic in place of aluminum housings for several components. Protection against RFI/EMI is achieved by plasma spraying all surfaces with aluminum.

An interesting C4 innovation is the use of investment-cast aluminum for about 28 separate small parts (shelving, etc.) of complex design. Even though investment casting is inherently expensive, it turns out (for these parts) to be cheaper than conventional forging and machining as used on the C3. A special alloy, called Al-201, was employed that has the following composition in weight percent:

	Minimum	Maximum
Cu	4.0	5.0
Mn	0.2	0.4
Ag	0.4	1.0
Mg	0.18	0.35
Ti	0.15	0.35

To keep the composition uniform, 99.99 pure aluminum is used as a starting material. Al-201 has the highest strength of any Al casting alloy and good ductility; the yield strength at 2 percent elongation is 50,000 psi but the tensile elongation is normally about 10 percent at rupture. This alloy was the invention of a private company (Electronic Specialties in California, which is no longer in business) and its production was licensed to Consolidated Aluminum Company (St. Louis, Missouri). It is now in the Mil Handbook and has a Mil-A-21180 high-strength casting specification. Apparently AFML brought the alloy into acceptance by sponsoring trial casting at Cercast (Montreal) and Hemet (Los Angeles). This was a case where there was a lack of user incentive to take a risk on a new material; LMSC waited until it was in the Mil Handbook before specifying use.

Some comments should be made regarding the use of the graphite-epoxy composite for the equipment section cone. This was considered, almost dropped, and then seriously reconsidered during the MSTE and MSAT technology programs immediately prior to the initial Engineering Development Program start. There was an initial reluctance for two reasons: one was the usual reluctance to pioneer--no primary missile structure had been built using an organic matrix composite; the other was mentioned previously--at that time (circa 1971) graphite-epoxy prepreg was commercially available only in the form of unidirectional tape. As is frequently the case, there was an individual who acted as an evangelist; he was the prime pusher for the LMSC efforts to get vendors to produce preimpregnated cloth (which was critical) and to obtain enough property data for good computerized structural designs. Prior to this effort, the designers wanted to increase the safety factor from 1.25 (used for metals) to 1.50 to compensate for lack of knowledge. Naturally this would have made the composite unattractive.

LMSC points out that the graphite cloth/epoxy material was essentially developed by a combination of LMSC technology "seed contract money" and financing put in by various vendors. Thus, while the MSAT funding was very small by Engineering Development Program standards, it was highly leveraged. In a direct sense, LMSC did not use any results of the AFML Advanced Development Program on Advanced Composites. In an indirect sense, the vendors would probably not have the requisite level of technology if it had not been for the Air Force ADP.

The graphite-epoxy materials used in C4 are all supplied by Fiberite, Inc. (Winona, Minnesota). The fabric is woven from 3000 filament T-300 tow, supplied by Union Carbide Corporation, which uses a polyacrylonitrile (PAN) precursor. Further details of properties, processing, and testing are given in Ref. 13. In passing, LMSC has little interest in the use of graphite-epoxy for the reentry vehicle substructure, which is being considered for the MX.

As a generality, LMSC points out that many new and promising materials were not used because (1) there is little incentive for taking risks, (2) high reliability is a prime requirement, (3) qualification of a new material is expensive and time-consuming, and (4) system managers want predictable costs.

f. Classification and Contacts. All missile performance (or other critical) data have been omitted, or described in generalized terms, to keep the present document unclassified. It appears at the moment that such classified data will not be especially pertinent to the objectives of this study. If it should turn out otherwise, a supplemental classified report will be issued during Phase II.

In addition to the perusal of written background material, many personal discussions were held, largely with personnel of the Navy Strategic Systems Project Office (SSPO) and the Lockheed Missile and Space Company (LMSC). The authors wish to express their appreciation for the excellent cooperation of both groups. In addition to these people, some of whom are named below, R.A. Ellis (of United Technologies Corporation's Chemical Systems Division) and M.A. Steinberg (of the Lockheed Corporation) gave particularly valuable input concerning the materials technology of the C4 as well as other systems.

Arranged in no particular order, the Washington area SSPO people with whom the authors held technical discussions were:

Jerry Needham (missile structures)
Sanford Weinger (rocket motors)
Fletcher Vandersmith (rocket motors)
John Kincaid (SSPO consultant)
Mark Messerole (reentry vehicles)

In addition, contacts were made with the following SSPO personnel located in Sunnyvale:

Phillip Barlow
Lt. Cdr. R.W. Benzin
Eo Kawahara
Carl Webb

At LMSC, the authors wish to single out Chester A. Zimmerman (propulsion) and Sol E. Singer (Chief Scientist) of the Missile Systems Division (MSD) for their yeoman service in arranging an extensive meeting at Sunnyvale starting 8 August 1978. Both formal and informal discussions were held with many other MSD people, some (but by no means all) of whom are listed below:

Goldin Parker (flight technology)
Herman Bettencourt (reentry vehicles)
Frank Crossley (structures)
James Huizinga (propulsion)
R.E. Hull (propulsion)
Robert Krapp (PBCS)
Kris Stigen (structures)
Clark Canffy (design)
Ray Ernst (propulsion)
Al Johnson (reentry vehicles)

Aside from merely being a matter of record, the above names serve as private communication references for many areas of the text of this Section A of Chapter VI where formal references are not readily available.

B. ADVANCED COMPOSITES AND FIBERS

1. Introduction

One obvious force that drove the development of advanced composites and fibers was the ever-increasing Service demands for materials to survive harsher environments created by higher performance equipment. Accompanying these requirements have been the pressures for lower material weight and lower material cost. Single materials (steel, aluminum, etc.) have their limitations in satisfying the need for higher performance so that the ancient concept of combining dissimilar materials to obtain superior properties adds a new dimension to the list of candidate materials. So it is understandable that the DoD and its Commands and Laboratories have been sponsoring programs for many years to advance the state of the art in composite materials to meet Service needs.

Reference 14 gives an excellent review of fiber and composite development and excerpts are included throughout this section. Composite materials are combinations of two or more distinct solid materials that are bonded to each other in order to combine the properties of the component parts to obtain composite properties which may be new or unique. They have long been used to fabricate useful artifacts and products. Wood and bone are natural composite materials that have been used since time immemorial. The credit for developing the first engineering composite probably belongs to the early Egyptians who found that a product of superior properties was obtained by gluing together a number of thin strips of wood. The armorers of Japan also understood the advantages to be derived from composite materials. The outstanding performance of the Samurai blade was a result of the craftsmanship of these sword makers and their ability to combine steel and iron to form a blade that has an extremely hard and keen edge while retaining a flexible body. At present, a myriad of combinations of metals, ceramics, and nonmetallic materials, all of which can properly be called composites, are in engineering use.

Albert G.H. Dietz described the evolution of fibrous composites in Ref. 15 and describes their origin.

> "Fibrous composite materials as we know them today got their start in life during one of those recurring confluences or ripening technical developments and urgent needs that

> have a way of sparking new ideas. During the last war, when metal supplies were extremely tight, a considerable technical search was on for viable substitutes. In the early 1940's, two new materials--neither of them useful of themselves at that time--came to fruition. One of these was glass in filament form. The other was the first of a series of what we now call thermosetting plastics--long-chain polymers in liquid form which, by heating, could be cured or polymerized into transparent solids."
>
> "Then along about 1942, somebody in the Army Engineering Corps got the bright idea that these two materials could be put in harness, so to speak, much the same way that steel rods were used to reinforce concrete so as to carry heavier loads than either one separately--for the same weight--could carry alone. Some glass fibers were woven into cloth; the cloth was then draped over a plaster of Paris mold of boxlike shape; liquid resin with a catalyst mixed into it was soaked into the fabric by brushing and the excess rolled out. After several successive layers of resin-soaked fabric had been built up, the laminate was allowed to cure. And in an hour or so, a rigid structure was pried off the mold--the first modern fibrous composite; strong and rigid, yet light and reasonably durable."
>
> "Immediately, attention turned to refining this process--which we still term "hand lay-up"--and to seeing whether it could be applied to a number of items of military interest. Before the war was over, glass fiber reinforced plastics, as they came to be known, found use as substitutes for metal in such diverse objects as ammunition cases, fenders for military vehicles, radomes, and even experimental armor for infantry."

The following examples of DoD Laboratory and industry work on reinforced plastics in the 1940s and 1950s demonstrate the DoD involvement in the early composite technology development. This information was obtained from Refs. 16 and 17.

Prior to 1949, epoxide resins were used primarily as casting and encapsulation materials. Examination of the resin system, however, lead to the belief that minor modifications would result in a laminating resin that would be superior to those available at the time. A coordinated effort was undertaken by the Navy (Naval Ordnance Laboratory now Naval Surface Weapons Center) and E.I. duPont and Company to modify and evaluate the epoxy system. As a result of these studies, a family of epoxide laminating resins was produced which has been one of the most successful systems ever developed.

In 1953, at the Naval Ordnance Laboratory, glass fiber reinforced plastics composite materials were found to have strength properties which make then usable in underwater ordnance structural applications.* Data were collected on the properties of glass laminated materials in small tube form and as half-scale models of Mine Case Mark 57. Analysis of the data indicated that these materials could assume a practical role in the construction of underwater ordnance primary structures.

The development of plastics materials for primary structural applications in ordnance is particularly significant since these unique materials are light in weight, nonmagnetic, and noncondutive. In addition, when exposed to salt water they do not corrode or erode away. They can be formed into a variety of complex shapes without great difficulty and are derived from basic materials of a noncritical nature. Availability of a body of data on performance characteristics results in construction and testing of full-scale Mark 57 Mine Cases. Based on these tests, a glass-reinforced composite construction was selected and put into production as the warhead and flotation chamber material for the Mark 57 Mine Case.

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*Of course, glass-reinforced plastics were used in surface small craft as early as 1946.

In the first stage of development of the POLARIS Fleet Ballistic Missile (1955), the feasibility of using a lightweight, high-strength glass-fiber-reinforced motor case was investigated. Aerojet-General, Goodyear Aircraft, Naval Ordnance Laboratory, and the Naval Research Laboratory were involved in this investigation. Based on the results of these studies, additional work was carried out to develop reliable economical materials and procedures for filament winding motor cases. Mechanical and environmental tests were performed on small-scale and full-size filament wound motor cases, and fabrication materials and techniques were optimized.

Development of a reliable filament wound composite was highly significant in that a motor casing material was made available which combined lightweight with high-strength properties. When applied to the second stage of POLARIS A2, it was found that for an equal overall weight, the range could be significantly increased by the additional propellant that could be used. Subsequent application of the filament wound motor cases to both the first and second stages of POLARIS A3 resulted in achievement of its design range, a goal which would not have been met if steel motor casings had been used.

2. High Performance Composites

Approximately two decades ago, in order to meet the increasing requirements of advanced military systems, a number of low density fibers were produced that were significantly stiffer than glass fibers with comparable, or superior, strength properties. In the ensuing years, the category, high performance fibers, has grown to include such diverse materials as boron, carbon, aramid (an organic compound), silicon carbide, and alumina.

Concurrently, as the availability of these fibers increased, and their price fell by two orders of magnitude, it became possible to consider using these materials in a variety of nonaerospace applications, a transition similar to the one experienced by fiberglass.

The principal physical properties of the high performance fibers that are currently commercially available in the U.S. are presented in Table 8. The properties of E-glass fiber most commonly used in reinforced plastics and of higher strength S-glass fibers developed for the aerospace market are also included in Table 8 for purposes of comparison. The principal physical properties of high performance fibers presently under development and that may be commercially available in the future are presented in Table 9.

Incorporating one or more of these high performance filaments in a suitable matrix produces a class of composite materials that exhibit physical and structural properties not unattainable with conventional engineering materials. The matrix can be a thermosetting resin, such as an epoxy, polyester, or polyimide; a thermoplastic resin, such as nylon or polysulfone; a metal, such as aluminum or titanium; or a ceramic such as glass.

The mechanical properties of fibrous composites depend principally on the type, volumetric concentration, and relative orientation of fibers in the matrix. In synopsis, fibrous composites are strongest and stiffest in the direction of the fibers where the mechanical properties of the fibers predominate and are relatively weak in the direction normal to the fiber where the matrix properties predominate. This is a very different situation than exists for homogeneous materials of construction, such as aluminum and steel, which exhibit isotropic mechanical properties. Because of the directional nature of the structural properties of composites, the design of components made from composites is more difficult than for components made from homogeneous materials, and a better understanding of the stresses the component will experience if service is required. These nonisotropic characteristics, however, can also

TABLE 8. PHYSICAL PROPERTIES OF COMMERCIALLY AVAILABLE HIGH STRENGTH FILAMENTS (Ref. 14)

					Graphite Filaments		
Material	E-Glass Fiber	S-Glass Fiber	Aramid Fiber	Boron/Tungsten Fiber	High Strength Fiber	High Modulus Fiber	Very High Modulus Fiber
Product	(Roving)	(Roving)	Kevlar 49	5.6 mil Diam.	Thornel 300 (WYP 15-1/0)	Magnamite HMS	Celion GY-70
Supplier			Dupont	Avco/CTI	Union Carbide	Hercules	Celanese
Density lbs/in^3	0.092	0.090	0.052	0.090	0.062	0.067	0.071
g/cm^3	2.54	2.48	1.44	2.48	1.72	1.86	1.96
Tensile Strength 10^3 psi	372	550	400	500	360	340	270
MPa	2500	3700	2800	3400	2600	2300	1900
Tensile Modulus 10^6 psi	10.5	12.4	18.0	58.0	32.5	50	75
GPa	73	86	124	406	225	350	520
Ultimate Elongation, %	4.8	5.4	2.5	0.8	1.1	0.58	0.38
Specific Strength, in	4.0×10^6	6.0×10^6	7.9×10^6	5.6×10^6	6.1×10^6	5.0×10^6	3.8×10^6
cm	9.8×10^6	1.5×10^7	1.9×10^7	1.4×10^7	1.5×10^7	1.2×10^9	9.7×10^6
Specific Modulus, in	1.1×10^8	1.4×10^8	3.5×10^8	6.4×10^8	5.2×10^8	7.5×10^8	1.1×10^9
cm	2.9×10^8	3.5×10^8	8.6×10^8	1.6×10^9	1.3×10^8	1.9×10^9	2.7×10^9
Filament Diameter, mils	0.20-0.55	0.35-0.40	0.47	5.6	0.3	0.3	0.33
cm	0.0005-0.014	0.0009	0.0012	0.014	0.0007	0.00075	0.00084
Thermal Conductivity BTU-ft/hr (ft^2)(°F)	0.56				12	70	
W/m °K	0.97				20.8	121	
Electrical Resistivity Ω mil ft					9000	4500	3900
μΩcm					1500	750	650
Current Price, $/lb	0.49	2.00	8-10 are up to 27 for fine denier fiber	200	32	70	110-250

TABLE 9. PHYSICAL PROPERTIES OF DEVELOPMENTAL HIGH STRENGTH FIBERS (Ref. 14)

Material	Pitchbase Graphite	Boron/Carbon	Silicon Carbide/Carbon	Alumina	Boron Nitride
Product	Thornel P-VSB-32			FP	
Manufacturer	Union Carbide	AVCO	AVCO	DuPont	Carborundum
Density lbs/in^3	0.073	0.082	0.113	0.143	0.065-0.069
g/cm^3	2.02	2.26	3.11	3.95	1.8-1.9
Tensile Strength 10^3psi	200	475	450	200	120 (ave.)
MPa	1380	3300	3100		830
Tensile Modulus 10^6psi	50	53	62	55	30 (ave.)
Gpa	345	363	425	377	210
Ultimate Elongation %	0.4			0.4	
Specific Strength in.	2.8×10^6	5.8×10^6	4.0×10^6	1.4×10^6	1.8×10^6 (ave.)
cm	6.8×10^6	1.5×10^7	1.0×10^7	3.6×10^6	4.7×10^6
Specific Modulus in.	6.8×10^8	6.4×10^8	5.5×10^8	3.5×10^8	4.6×10^8 (ave.)
cm	1.7×10^9	1.6×10^9	1.4×10^9	8.9×10^8	1.2×10^9
Filament diameter mils	0.44	5.6	5.6	1.0	0.24
cm	0.0011	0.014	0.014	0.002	0.0006
Thermal Conductivity BTU ft/hr(ft^2)($^\circ$F)	48			0.074	1.7
W/m$^\circ$K	83			0.13	3.0
Electrical Resistivity, ohm-cm					10^{14}
Current Price $/lb	20	250	450	200	

be an asset to a designer who can tailor a composite by selectively positioning the reinforcing fibers to meet specific requirements.

The major attribute of advanced structural composites is their combination of high strength and high stiffness with low density. Structures made of composites are usually much lighter than structures of comparable strength and/or stiffness made of standard materials of construction such as aluminum or steel.

Figure 23 compares the longitudinal tensile strength and tensile modulus of the composites listed in Table 10 with those of aluminum and steel. It is to be noted that the composite materials exhibit higher strength than steel and aluminum. In addition, the carbon and boron composites are all much stiffer than aluminum, with stiffness comparable to that of steel, and the very high modulus carbon-epoxy has nearly twice the modulus of steel. The modulus of the aramid-epoxy composite is comparable to that of aluminum, whereas the modulus of fiberglass-epoxy is lower than that of aluminum.

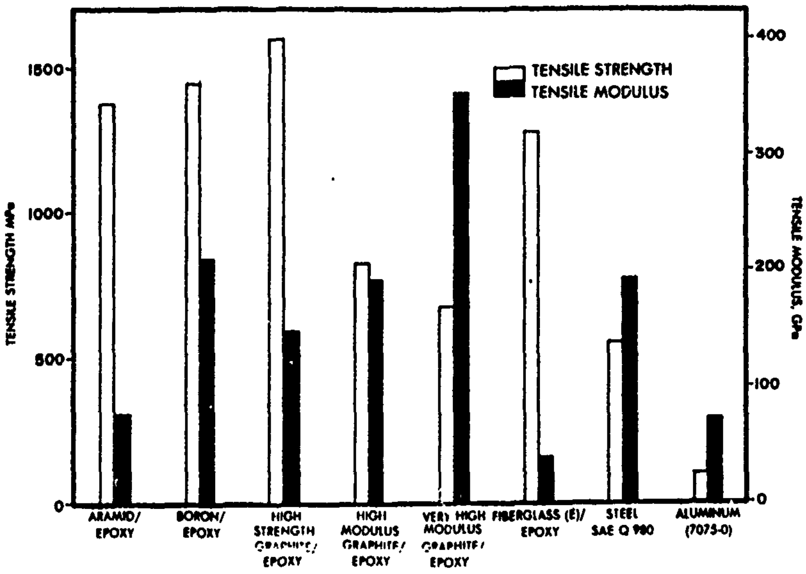

FIGURE 23. Tensile Properties of Materials of Interest (Ref. 14)

TABLE 10. ROOM TEMPERATURE PROPERTIES OF COMMERCIAL UNIDIRECTIONAL FIBROUS EPOXY COMPOSITES (Ref. 14)

Fiber Manufacturer			**DuPont**	**AVCO or CTI**	**UCC**	**Hercules**	**Celanese**
Fiber Type	**E-Glass**	**S-Glass**	**Aramid (Kevlar-49)**	**Boron/W 4mil**	**Thornel 300 Graphite**	**HMS Graphite**	**GY 70 Graphite**
Fiber Volume %	60	60	60	50	60	62	60
Prepreg Mfg	3M	Fiberite	DuPont	3M	Fiberite	Hercules	Narmco
Prepreg Type	1009-26	HY-E 9034B	PR 286	SP292	HY-E 1076C	HMS 3501/6	5208
Density lbs/in^3	0.072	.0.075	0.050	.068	0.058	0.059	0.062
gr/cm^3	2.00	2.08	1.38	1.88	1.61	1.63	1.72
Longitudinal (0^o) Properties							
Tensile Strength 10^{3}psi	185	235	200	186-232	233	120	98
MPa	1280	1620	1380	1280-1600	1610	830	680
Tensile Modulus 10^{6}psi	5.7	8.5	11	29.6-32.0	21.5	28	51
GPa	39	59	75	204-220	148	193	352
Flexural Strength 10^{3}psf	200	270	90	245	298	140	115
MPa	1380	1860	620	1690	2060	966	794
Flexural Modulus 10^{6}psi	7.0	8.5	10	28	21.5	24.5	40.0
GPa	48	48	69	193	148	169	276
Compressive Strength 10^{3}psi	90		40	443-460		55	
MPa	620		280	3060-3180		380	
Compressive Modulus 10^{6}psi			10.5	35.0-35.5		15.5	
GPa			72	242-545		107	
Transverse (90%) Properties							
Tensile Strength 10^{3}psi			4.3	9.8-17.8		4.5	
MPa			30	68-123			
Tensile Modulus 10^{6}psi			0.8	3.16-3.92		1.1	
GPa			6	21.8-27			
Interlaminar Shear 10^{3}psi		.14	8.7	17	17	8.0	8.5
MPa		97	60	11.7	117	55	59

The inherent advantage of using advanced composite structures is demonstrated more graphically in Fig. 24 which compares the specific strength, i.e., strength-to-density ratio, and specific modulus, stiffness-to-density ratio, of the advanced composites with those of traditional materials. Consideration of specific properties allows a comparison to be made on a unit weight basis. On a specific basis, all the composites are all much stronger than steel or aluminum; however, only the advanced composites are stiffer on a specific basis than the metals, while the specific stiffness of fiberglass-epoxy composites is slightly lower than that of steel or aluminum. Advanced composites differ from the more traditional fiberglass composites in that they can exhibit both a significantly higher specific modulus as well as a significantly higher specific strength than those of the traditional metals.

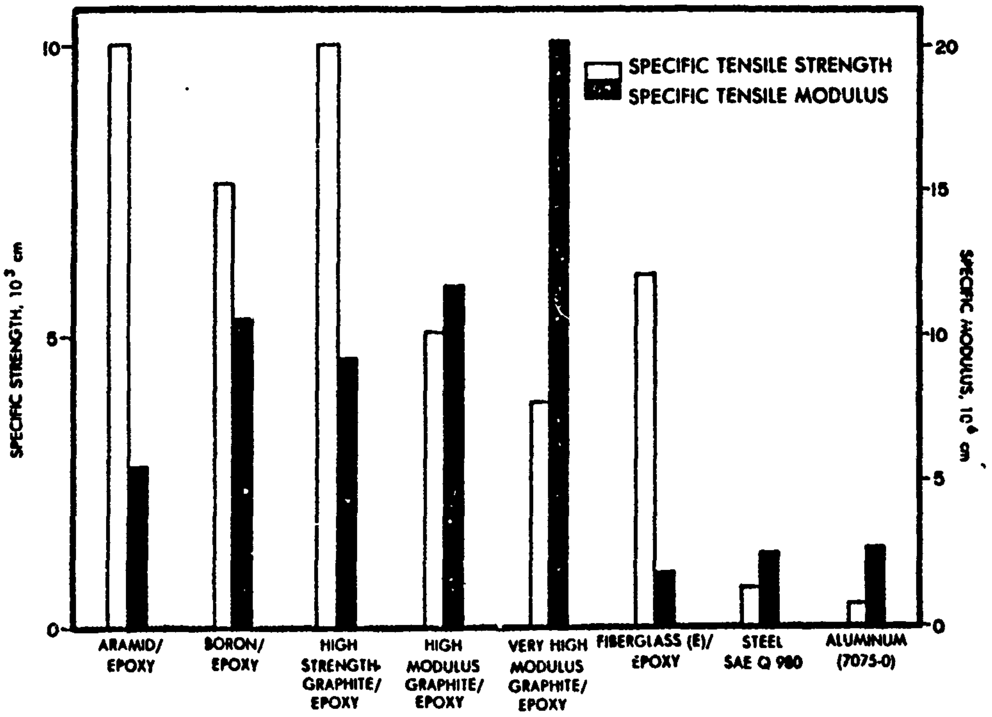

FIGURE 24. Specific Tensile Properties of Materials of Interest (Ref. 14)

The excellent fatigue properties of advanced composites are another of their major attributes. Figure 25 illustrates the fatigue resistance of uniaxial advanced composites compared to those of other materials. The three advanced composite materials presented in Fig. 23 have a residual strength of about 70% of the static value after 10 million cycles, which is significantly higher than the strength retention of either aluminum, steel, or fiberglass-epoxy composites. The significantly better fatigue characteristics of the advanced composites than those of fiberglass composites are striking. Since many useful structures (such as machinery and transportation equipment) are often designed for fatigue rather than for strength, the excellent fatigue characteristics of advanced composites, as compared to other materials, further enhance the weight reduction that can be achieved by the use of these composites in highly stressed fatigue critical structural parts.

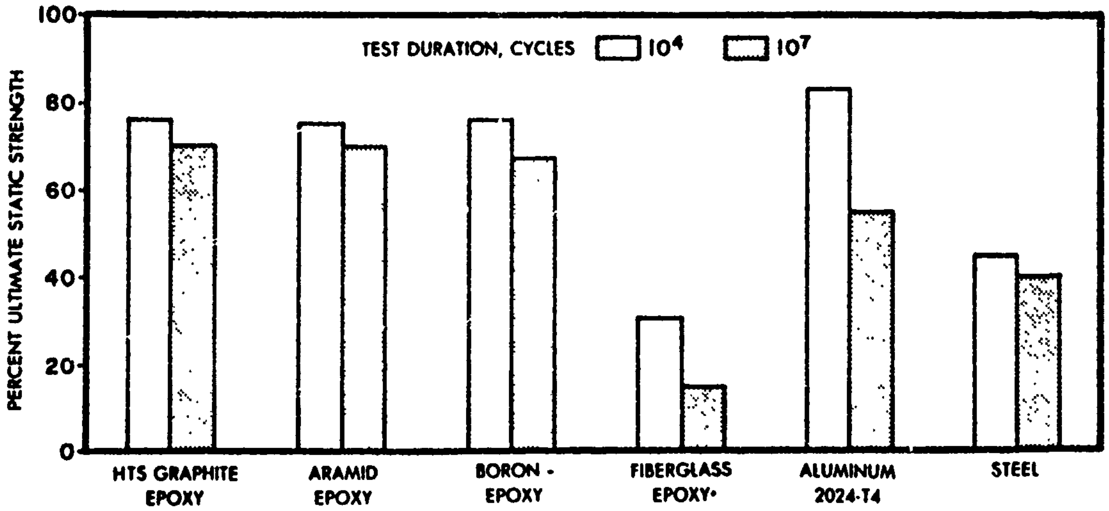

FIGURE 25. Fatigue Resistance of Materials of Interest (Ref. 14)

Still another general attribute of high performance composite is their tendency to dampen vibrations more rapidly than materials that are not as stiff.

It should not be inferred from the above that all advanced composites have similar or equivalent properties. Each of the high performance fibers has unique properties that differentiates it from other fibers as outlined in Table 11. In addition to the general high strength, high modulus characteristics observed above, a specific composite will have other properties that will make it a better choice for a specific application than other composites. In fact, the material properties desired for a given component may best be achieved with hybrid composites that are mixtures of different filaments in a common matrix. Hybridization greatly expands the range of properties that can be achieved with composites. The concept of hybridization also blurs the distinction between the advanced composites, as previously defined herein, and the more generally established fiber reinforced materials such as glass fiber reinforced plastics. Depending on the ratio of carbon fiber to glass fiber in the composite, carbon/glass-epoxy hybrids will exhibit a significant range in properties. Furthermore, if the stiffer carbon fiber is selectively placed in the structure, the overall properties (such as stiffness and fatigue) of the structure may be enhanced disproportionately to the amount of carbon fiber added.

Most of the environmental properties of advanced composites materials are a function of the matrix material rather than the high performance fiber. In this aspect, resin matrix composites have the general characteristics of reinforced plastics, metal matrix composites will behave like metals, and so forth.

TABLE 11. COMPARISON OF MAJOR ATTRIBUTES OF CURRENTLY AVAILABLE HIGH PERFORMANCE FIBERS (Ref. 14)

Fiber	Major Positive Attributes	Major Limitations
Aramid	**Highest specific strength** **High impact strength** **Low cost**	**Poor compressive strength** **Difficult fiber to cut** **Limited to resin matrix components**
Boron	**High compressive strength**	**High cost fiber** **Low bending radius** **Not suitable for complex shapes**
Silicon Carbide (continuous fiber)	**High compressive strength** **Inert towards molten metals**	**Low bending radius** **Relatively high cost**
Graphite/Carbon (PAN)	**Lowest coefficient of thermal expansion** **Electrical conductor**	**Brittle fiber relatively poor impact strength** **Electrical conductor**
Graphite/Carbon (Pitch)	**Potentially lowest cost fiber**	**Low tensile strength** **High coefficient of variation**
Alumina FP	**Inert towards molten metals** **Electrical Insulator** **Potential low cost**	**High density resulting in low specific properties**

3. Graphite Fibers

Roger Bacon of Union Carbide did much of the original work on graphite filament development. His description of some of the early work (Ref. 18) is given below.

> "Public disclosures of practical processes for converting cellulose textiles into useful carbon fibers have been confined largely to the patent literature. Edison is credited with the first invention of this kind: an incandescent-lamp filament made by

carbonizing a filament of natural or regenerated cellulosic material. After the advent of tungsten lamp filaments in the early 1900s, filamentary carbon was not used significantly by industry until the middle 1950s, when Abbott developed a process for converting rayon into a fibrous carbon material for such uses as insulation, filtration, and adsorption. This material, produced by the Carbon Wool Corporation, was carbonized at temperatures of up to 1000 °C; it possessed tensile strengths as high as 40,000 psi. In 1959 the Union Carbide Corporation began commercial production of "graphite" cloth as well as other fibrous forms made by a slow batch process consisting of baking the fibers in an inert atmosphere to approximately 900 °C, followed by graphitizing to temperatures usually higher than 2500 °C. Following Union Carbide into this field were such companies as HITCO, the 3M Company, the Great Lakes Carbon Corporation, and the Carborundum Company. In addition to graphite fibers, lower temperature carbon fibers (usually heat-treated to 1200 to 1800 °C) were also produced, primarily in cloth and felt forms. The single-filament tensile strengths of these fibers ranged from 50,000 to over 100,000 psi, while their Young's moduli were only 4×10^6 to 7×10^6 psi. The most important application was for the reinforcement of phenolic resins to form ablative materials for rocket and missile components."

"In the first years of this new carbon-fiber industry, several companies experimented with chemical pretreatments of the rayon raw material. These treatments proved capable of increasing carbon yields and speeding up processing rates, especially during the early "heat-treatment" stage. The first commercially produced carbon textile made according to this type of process was 3M Company's Pluton, introduced in 1961. An advantage of the chemical pretreatments is the ability to use rapid continuous-processing methods. The first continuously processed fibers were Union Carbide's carbon and graphite yarns, introduced to the market in 1963.

> Similar types of yarn were subsequently produced by HITCO and the Carborundum Company."
>
> "The availability of strong and uniform continuously processed carbon yarns permitted, for the first time, very high degrees of stretch to be applied to these yarns during graphitization to temperatures in excess of 2500 °C. These experiments led to the commercial production of high-strength and high-modulus graphite fibers (Thornel 25) late in 1965 by the Union Carbide Corporation. That same year an Air Force Materials Laboratory contract was awarded to Union Carbide to further develop this stress-graphitization process and to discover new processes for producing high-performance carbon fibers. Higher modulus yarns were subsequently produced commercially, the most recent (1970) being Thornel 75, a yarn with a modulus of 75 x 10^6 psi. The only other major producer of high modulus carbon yarns from a rayon precursor is HITCO, which entered the market in 1967 with yarns produced by similar methods. At about the same time as the UCC development of graphite fiber from rayon precursors, the Royal Aircraft Establishment developed a graphite fiber with polyacylonitrile (PAN) as the precursor."

A simple outline of graphite process is shown in Fig. 26. As shown in the outline, graphite is formed by passing precursor fibers through a series of controlled ovens. For ease of handling, the fibers are arranged in bundles (tows) which consist of from 1,000 to 160,000 individual fibers approximately 12μm in diameter. The time-temperature history of the fiber establishes the grade of fiber produced: the higher the temperature reached, the higher the degree of graphitization and resulting modulus. However, in general, the strength of the fiber decreases as the modulus increases. It is possible to obtain many combinations of strength and modulus with graphite fibers. In this aspect, graphite fibers are unlike other high performance fibers that have fixed properties. The fibers, after leaving the oven, often are coated with polyvinyl alcohol

(PVA) or a resin of compatible size to protect the fiber from handling damge. The tows are then wound into spools or woven into cloth or chapped into a mat for further use.

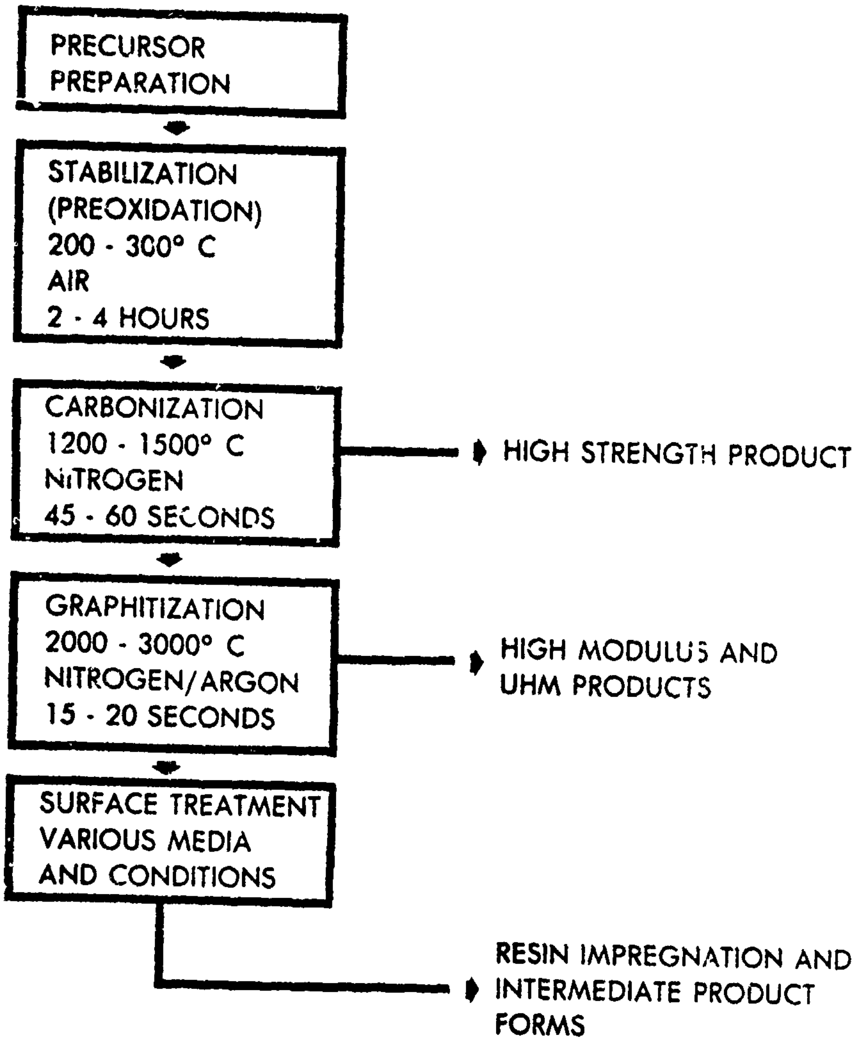

FIGURE 26. Graphite Fiber Process Outline (Ref. 14)

There is significant weight loss during graphitization. From stochiometry considerations, a 45% graphite yield would be expected per unit weight of a PAN precursor. In practice, a lower yield is attained with approximately three pounds of precursor required per pound of graphite produced. A higher yield is obtained with a pitch precursor since pitch has a higher initial carbon content than does PAN.

Graphite filaments are based on petroleum or coal-derived chemicals. PAN precursors used to make graphite are variants of the acrylic and modacrylic fibers commonly used in the textile industry (e.g., Orlon, Dynel). In 1972, 349 million pounds of acrylic and modacrylic fibers were consumed in the United States. It should be noted that no significant quantities of acrylic filament are currently produced in the U.S. Therefore, PAN graphite consumed in the U.S. at the present is based to a larger extent on precursors made in other countries, primarily in England and Japan.

Pitch base graphite is based on petroleum or coal tar pitch. Pitch is an inexpensive by-product of the petrochemical industry similar to tar and is readily available. A fair amount of preparation is necessary to obtain a grade suitable for fiber extrusion.

There are five major U.S. distributors of graphite-carbon fibers at the present time. The types of products sold by these firms are shown in Table 12. These fibers are PAN-derived except for Union Carbide Corporation's Pitch fiber (Thornel P). Union Carbide and Celanese do not manufacture all the graphite fibers sold under their trademark. Union Carbide's Thornal 300 (PAN), and Celanese's Celion fibers are imported from Japan. Union Carbide now only manufactures its Thornel P fiber, but has development facilities for other fibers. These facilities are located in Ohio and South Carolina. The capacity of the pitch plant is rated in "... pounds per hour" or possibly 100,000 lb/yr (~50 metric tons/yr). Celanese manufactures its high modulus BY70 graphite fiber in a pilot plant associated with the Corporate Research and Development Laboratories in Summit, N.J. This plant has a current capacity of 1000 lb/month (0.5 metric ton/month) that could be rapidly expanded if required to 30,000 lb/yr (14 metric tons/yr). Hercules, Inc. manufactures its graphite products in Magna, Utah. Plant capacity has been reported as approximately 100 tons/yr. Stackpole's plant capacity has been reported to be 30 tons/yr, and Great Lakes Carbon's capacity is reported to be 20 tons/yr. Avco Corporation's Specialty Materials Division has recently announced that it is building a graphite filaments manufacturing plant that is expected to be in operation by 1979.

TABLE 12. PRINCIPAL U.S. SUPPLIERS OF GRAPHITE FIBERS (Ref. 14)

Fiber Type	High Strength	High Modulus	Ultra High Modulus
Normal Fiber Properties			
Tensile strength Ksi (MPa)	~400 (2800)	320 (2200)	250 (1700)
Tensile Modules Msi (GPa)	30 (220)	50 (350)	70 (500)
Specific Gravity	1.76	1.86	1.96
Distributor			
Union Carbide	x	x (pitch)	
Hercules	x	x	
Celanese	x		x
Great Lakes Carbon Corp.	x	x	
Stackpole	x	x	
Others (Hitco, Avco, imports)			

Consumption of graphite fibers in the U.S. has increased from experimental quantities in the late 1960s to approximately 300,000 lb (140 metric tons) in 1977. A manufacturer's perspective of the historical growth of the graphite fiber market is presented in Fig. 27. Industry estimates of 1977 consumption range from 350,000 lb (115 metric tons) to 400,000 lb (180 metric tons). Over 80% of this material was used as continuous filaments, with the balance being chopped fiber (10%), mat (5%), and cloth (5%). At present, the market for graphite fibers is fairly evenly divided between aerospace and aircraft applications and others. Sporting goods represent the major commercial use of graphite fibers. The estimated market distribution for graphite fibers is:

Aerospace and Aircraft	40% - 50%
Sporting Goods	35 - 45
Other Commercial and Developmental Use	Balance

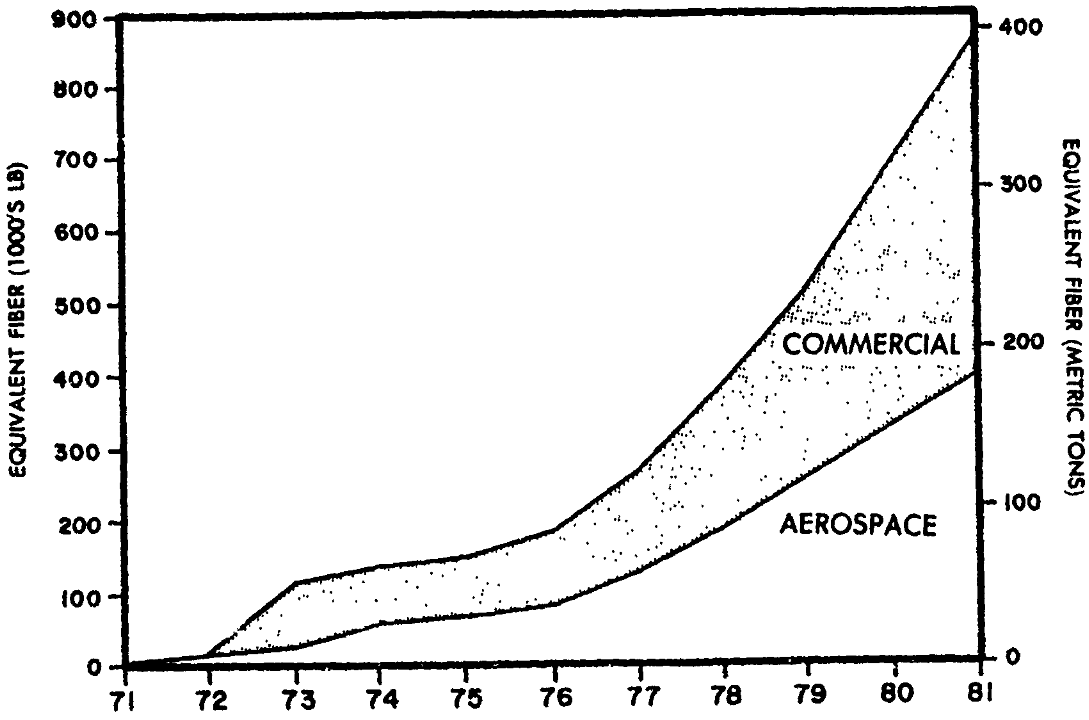

FIGURE 27. Total Graphite Fiber Market (Ref. 14)

The current price of PAN-base graphite fiber ranges from $17/lb to $250/lb. The price of high strength graphite ranges from $17/lb for 160,000 filament tow to $105/lb for 1000 filament tow, with 3000 and 6000 filament tow material selling at $35/lb and $32/lb, respectively. This price schedule reflects increasing manufacturing costs associated with lower filament tow material. The lower the tow count, the more expensive the raw material from more than $3.00/lb for low tow count precursor to less than $1.00/lb for high tow count precursor.

Very high modulus graphite (Celanese GY70) currently sells for $110/lb to $150/lb depending on the quantity purchased. The single end grade of this material sells for $250/lb. Union Carbide's pitch base material currently sells for $20/lb in fiber form, and $7.50/lb to $8.50/lb in mat form.

High modulus and very high modulus PAN-based graphite fiber would be more expensive than the high strength fiber because of higher processing costs.

Pitch-base graphite fiber could potentially be made available at a lower price than PAN-based graphite. Because of the lower cost of the precursor material: less than $1 per pound of graphite for pitch base, as compared to $2 to $10 per pound of graphite for PAN-base material. A price of $5/lb for pitch-base graphite has been projected if a sufficient (unspecified) volume develops.

4. Kevlar 49$^{(R)}$ (Aramid) Fiber

Kevlar$^{(R)}$ is the registered trademark for one member of a family of aromatic polyamide fibers introduced by E.I. duPont de Nemours & Co., Inc. in 1972. Aramid is the generic name assigned by the Federal Trade Commission to this class of high strength, high modulus organic fibers. Three types of Kevlar fibers are available: Kevlar 49 designed for the reinforcement of plastics, Kevlar 29 designed for ropes, cables and protective clothing, and Kevlar designed for the reinforcement of rubber, especially tires, belts, and hoses. Only Kevlar 49 is considered to fall within the scope of the present study.

p-Amino benzoyl chloride is polymerized in the presence of N, N dimethly-triamino phosphate. The resulting polymer, Poly-p Benzamide, is then spun at a temperature above 500 °F (260 °C) to form Kevlar 49 fibers. Manufacturing details were not available. It is presumed that the manufacturing equipment and process are similar in many respects to the manufacture of other more common polyamide fibers, such as nylon 6, except for the choice of raw materials and operating temperature.

duPont manufactures its Kevlar products in Richmond, Virginia. The current plant capacity for all types of Kevlar fibers is 10 million lb/yr (4500 metric tons/yr). Facilities, which will increase capacity by 50%, are under construction, and will be on-stream in 1979.

Distribution of product output is currently as follows:

Kevlar (tire cords)	80%
Kevlar 29 (cordage)	12-15%
Kevlar 49 (reinforcements)	5-8%

An equivalent current annual capacity for Kevlar 49 is thus approximately 225 to 350 metric tons.

Depending on future market needs, total annual capacity for Kevlar products could be expanded from 40 million pounds to 70 million pounds (18,000 to 33,000 tons) in the next decade. The next round of plant expansion could occur as early as 1982. Product distribution would not be expected to change significantly.

The current price of Kevlar fibers range from $4.50/lb for chopped fiber to $27/lb for fine 200 denier filament. Most grades of Kevlar 49 used for reinforcement range from $8/lb to

$10/lb. The price structure is considered stable and no further economies of scale are foreseen with increasing production levels.

Consumption of Kevlar 49 fibers in the U.S. has increased from experimental quantities in 1972 to a minimum of approximately 500,000 lb (225 metric tons) in 1977. At present, the market for Kevlar 49 is distributed as follows:

Boats and Marine Applications	40%
Aerospace and Aircraft	40%
Miscellaneous	20%

5. Boron Fiber

In 1959, C.P. Talley of Texaco Experiments, Inc. reported for the first time the high strength and stiffness of boron formed by chemical vapor deposition. The potential of such a material for advanced aircraft applications was recognized by the Air Force Materials Laboratory which funded a large-scale research and development effort to advance the filament from the laboratory stage to production level. Boron was the first available continuous reinforcement for lightweight high performance composites.

Boron fibers are made in continuous form by reducing boron trichloride with hydrogen and depositing the elemental boron formed on an electrically heated, continuously moving tungsten substrate (0.0005-in.-diameter wire). The reaction occurs in a glass deposition tube which is fitted with gas inlet and outlet pools, two mercury-filled electrodes, a variable DC power supply connected to the two electrodes, a tungsten substrate let-off system, and a boron filament take-up unit. A stoichiometric mixture of boron trichloride and hydrogen is introduced at the top of the reactor. These react at the surface of tungsten wire that is heated to a maximum temperature of 1300 °C to form a mantle of deposited boron according to the following reaction:

$$B\,Cl_3 + 3/2\,H_2 \rightarrow B + 3\,H\,Cl$$

A typical reactor produces about 90 lb (40 kg) of boron per year.

The principal raw materials for the manufacture of boron fibers are tungsten wire, boron trichloride, and hydrogen gas. The fine diameter tungsten wire on which boron is deposited is an imported product and is quite expensive, viz., $2.50/1000 meters, which is equivalent to about $500/lb.

There are two producers of boron fibers in the U.S. at the present time. Avco Corporation has a 35,000 lb/yr (16 metric tons/yr) plant in Lowell, Massachusetts. Composite Technology, Inc. (CTI) has a 10,000 lb/yr (5 metric tons/yr) plant in Broad Brook, Connecticut. Both manufacturers offer 4, 5, 6, and 8.0 mil boron fiber with a tungsten core. CTI also offers for sale a 2.2 mil boron-tungsten fiber as well as a Borsic(R) tungsten fiber in 2 sizes, 5.7 and 8.2 mil in diameter. Borsic is a boron fiber with a silicon carbide outer shell (see Section 6 below on silicon carbide fibers). A small fraction less than 10% of Avco's current production capacity is boron on a carbon substrate.

The current demand for boron fibers is approximately 32,000 lb/yr (15 metric tons/yr) with the following market distribution:

Military Aircraft	81%
Sporting Goods	13%
Miscellaneous Other Applications (Aerospace and Commercial)	6%

This market is shared by Avco and CTI in an 80:20 ratio.

The prices of the types of boron fiber that are currently available are summarized in Table 13. The price of boron on tungsten fiber decreases significantly with increasing fiber diameter. This is due principally to the significant decrease in the weight fraction of tungsten in the fiber with increasing diameter. There are variations in price between fiber certified for aerospace applications and commercial grade fiber, which is similar to aerospace grade except that the manufacturing control and testing requirements are less stringent.

TABLE 13. CURRENT PRICE OF BORON FIBERS (Ref. 14)

Fiber Type	Diameter mil.	Sales Price $/lb
Boron/tungsten	2.0	500
	4.0	240
	5.6	180-220
	8.0	170-200
Borsic/tungsten	5.7	350
	8.1	350

As yet there is no standard price established for boron on carbon fiber which is quoted on an individual order basis. The price is claimed to be competitive with boron on tungsten fiber.

6. Silicon Carbide Fibers

Silicon carbide is available in continuous filaments and as whiskers. Silicon carbide (SiC) filaments were originally developed by General Technology Corporation in 1966 under contract to the Air Force Materials Laboratory. SiC filaments are made by chemical vapor deposition in the same manner as boron. The original impetus for developing SiC filaments was to obtain a fiber better suited than boron for incorporation in metal matrix composites. Boron rapidly loses its strength at temperatures above 1000 °F, and is not suitable for high temperature applications. Furthermore, boron reacts with molten metals, such as aluminum, which makes the fabrication of metal matrix composites by liquid infiltration or standard casting techniques not feasible. Silicon carbide filaments retain their mechanical properties at temperatures well over 1000 °F and can withstand exposure to molten aluminum. A variant product is Borsic$^{(R)}$ which is made by depositing an outer layer of silicon carbide over a boron filament.

Silicon carbide (SiC) filaments are made in continuous form by decomposition of methyl chlorosilanes on a heated substrate in the presence of hydrogen, in the same manner and equipment as are used to make boron fibers. SiC filaments have been made on both tungsten and carbide substrates.

The raw material requirements are the same as those for boron, except that methyl chlorosilanes are used instead of boron trichloride. Methyl chlorosilanes are common chemicals currently used to make silicones and organic silanes. The current price of methyl dichlorosilane is less than $1/lb.

Avco Corporation in Lowell, Massachusetts, is the only U.S. producer of silicon carbide fibers. Avco can currently produce 150 lb/yr (70 kg/yr) of either 4.0 mil or 5.6 mil fiber. It is currently expanding its facilities to be able to produce 500 lb/yr (220 kg/yr). A similar material is being made in West Germany by the Berghof Research Institute, which is represented in the U.S. by Fiber Materials, Inc. of Biddeford, Maine. A different type of silicon carbide filament is also manufactured in Japan by Nippon Carbon on a developmental basis.

If the market for silicon carbide develops, a facility similar to Avco's existing boron plant would be able to produce 75,000 lb/yr (35 metric tons/yr) of silicon carbide.

The pyrolysis of rice hulls, which naturally contain silicon and carbon, has been found to produce particulate silicon carbide. About 10% to 15% of the pyrolysis product consists of SiC whiskers, approximately 1μm to 3μm in diameter, that have length-to-diameter ratios in excess of ten. These whiskers should be suitable as discontinuous reinforcements for either metal matrix or resin matrix composites. Work is currently under way to increase the whisker yield. Whisker concentrations in excess of 30% have been reported.

Experimental quantities of SiC whiskers, mixed with particles obtained by the pyrolysis, are currently available from the Silag Division of Exxon Enterprises, Inc., at a price of $250/lb. If large volume demand developed, it is envisioned that the material could be made available at a significantly lower price, of the order of $10/lb at a production volume of a million pounds per year.

7. Alumina Fibers

E.I. duPont de Nemours and Co. Inc. is currently developing a high modulus inorganic fiber designed as Fiber FP. This material is a continuous filament polycrystalline alpha alumina yarn. This fiber is being considered principally for high temperature metal matrix applications.

According to the patent literature that has been issued to date, FFP alumina fibers are presumed to be prepared by extruding an aqueous gel of $Al_2(OH)_5Cl$ through spinnerets, drying the resulting fibers to remove most of the water, and then heat-treating the filaments under a programmed time-temperature cycle to form a continuous refractory oxide yarn. Maximum processing temperature is 1300 °C. The $Al_2(OH)_5Cl$ gel is prepared by reacting finely divided (less than 3μm diameter) Al_2O_3 particles suspended in water with hydrochloric acid, adjusting the concentration of alumina by distillation, and adding small amounts of magnesium chloride to adjust its viscosity. Since hydrochloric acid is formed during the firing of the unsintered fiber, vapors from the furnaces must be sent to a caustic scrubber before being vented.

The principal raw materials for the manufacture of alumina FP fibers appear to be alumina powder, water, hydrochloric acid, and magnesium chloride, all of which are commonly available materials.

At the present time, alumina FP fibers are only made in pilot quantities at a prototype facility at the duPont Experimental Station in Wilmington, Delaware. With some modification, this facility could produce up to 30,000 lb/yr (13 metric tons/yr). At present, two types of fiber are available: pure alumina, and alumina coated with silica for resin and nonmetal applications.

There is no established market for alumina FP at the moment. The current price of standard alumina FP fiber yarn is $200/lb.

8. Boron Nitride Fibers

Current development activities by the Carborundum Company in Niagara Falls, New York, may result in a boron nitride fiber that could be commercially important in a few years. Boron nitride fiber is prepared in two steps. Boron oxide precursor fibers are first prepared by conventional melt spinning techniques used in the manufacture of standard glass fibers. The boron oxide fiber is converted to boron nitride (BN) by reaction with ammonia at elevated temperature (>1500 °C). The reaction is slow and is limited by the diffusion of ammonia into the solid fiber. Since the two raw materials, ammonia and boron oxide, used to make the BN fibers are commodity chemicals and since the process makes use of existing fiberglass drawing technology, the projected costs for large-scale boron nitride manufacture could be quite low; higher than for fiberglass, but lower than for any of the other high performance fibers currently being used. Representative BN fiber properties that are currently achieved were presented in Table 9.

The development is still at an exploratory level, and boron nitride fibers are currently being made on an experimental basis in gram quantities. The properties of the fibers have been found to vary significantly from sample to sample, a single fiber may have significantly higher or lower properties than the average presented in Table 9. Individual fibers have been prepared that have a tensile strength of 340 ksi (2350 MPa) and a modulus of 41×10^6 psi (280 GPa), values comparable to those of a high strength graphite filament. Boron nitride fibers also have about the same density as graphite fibers. The filaments differ in that boron nitride does not conduct electricity and does not react with molten metals. The current program goals are to make high strength and modulus BN fibers on a reproducible basis.

Key properties of the principal resin systems that have been used to make advanced composites are summarized in Table 14.

9. Organic Matrix Composites

Epoxy resins have been the predominant matrix material for advanced composites. Two classes of epoxy resins have been used: high temperature resins which retain their mechanical properties at 350 °F (170 °C) and the general-purpose resins that can be used at temperatures of up to 180 °F (82 °C). The high temperature grades are used principally in aerospace applications, whereas the general-purpose resins are used principally in commercial applications which find principal use at ambient temperature. The general-purpose resins are significantly less expensive than both in terms of lower cure temperature, 200 °F (90 °C) versus 400 °F (300 °C), and faster cycle time (1- to 2-hr cure cycle versus 4- to 6-hr cure cycle).

10. Carbon Matrix Composites

Carbon matrix composites are a specialized class of composites made by pyrolysis of graphite reinforced thermoset resin

TABLE 14. RESINS USED AS MATRICES IN ADVANCED COMPOSITES (Ref. 14)

Resin		Epoxy	Phenolic	Polyester (Rigid)	Poly-imide	Nylon 6-6	Poly-sulfone	Polyaryl Sulfone	Polyether Sulfone	Polyphe-nylene Sulfide	Poly-Amide/Imide
Property	ASTM Test Method	THERMOSETTING RESINS				THERMOPLASTIC RESINS					
Specific Gravity	D 792	1.1-1.4	1.25-1.30	1.10-1.46	1.37	1.14	1.24	1.36	1.37	1.34	1.40
RT Mechanical Properties											
Flexural Strength Ksi	D 790	13-21	12-15	8.5-23	19	15.0	15.4	17.2	18.7	20	30.7
Flexural Modulus Msi	D 790				0.6	0.4	0.4	0.4	0.4	0.6	0.6
Tensile Strength Ksi	D 638	4-13	7-8	6-13	12	12	10	13	12	10	27
Tensile Modulus Msi	D 638	0.4-0.6	0.7-1.0	0.3-0.6	0.6	0.5	0.4	0.4	0.4	0.5	0.7
Distortion Temperature @ 264 psi, °F	D 648	115-550	240-260	140-400		150	345	525	397	275	525
Resistance to Heat Continuous Expousre, °F		250-500	250	250	>600	180	300	500	300	400-500	>650
Flammability	D 635	slow	very slow	Burns to self extin-guishing	Self extin-guishing	Self extin-guishing	Self extin-guishing	Self extin-guishing	Self extin-guishing	Non-burning	Self extin-guishing
Representative Cure/ Molding Conditions											
Temperature		250-450	270-360	270-350	400-700	520-620	250-325	720-750	610-710	550-675	600-625
Pressure psi			2000-4000	300-1200	2000	variable	100-3000	1000-2000	1000-1500	1000-1500	20,000 (inj. mold)
Price $/lb		0.70 to 10	0.47	0.36 (gen. purpose)	75-100 (cur.) 25(proj.)	1.16	2.95-4.75	20-40	6.50-11.00	2.05	9.75
Suppliers		7	31	19	2	13 (incl. other nylons)	Union Carbide Corp.	Carbor-undum Corp., Plastics Division	ICI America	Phillips Petro-leum Co.	Amoco Chemicals Corp.

matrix composites. These carbon-carbon composites can be considered to be a structural form of graphite. They possess the desirable refractory properties of graphite plus higher strength, stiffness, toughness, and resistance to thermally induced stresses. Carbon-carbon composites are mainly used in specialized applications where bulk polycrystalline graphite cannot meet the performance requirements. Most carbon-carbon applications have come about from needs of the aerospace industry for materials of unique thermal stability, such as rocket engines and ablative reentry systems. The use of carbon-carbon composites in aircraft brakes represents the most significant commercial application of this material. Carbon-carbon technology is treated more fully in Section D of this chapter.

11. Metal Matrix Composites

Metal matrix composites, as the name implies, are a class of composites in which the matrix is a metal. Interest in metal matrix composites developed as a result of the desire of the Department of Defense to obtain efficient (high specific strength and specific modulus) materials for high temperature applications where the utility of organic matrix composites is limited. In view of the inherently higher strength of metals than that of the resins used as organic matrices, metal matrix composites would have superior sheer and transverse properties. Metal matrix composites would also be good thermal and electrical conductors, be efficient sound and vibration absorbers, and have better tolerance for shocks and ballistic impacts. In some applications, metal matrix composites would also be expected to have better environmental resistance than resin matrix composites.

Metal matrix composites have been the subject of investigation for over a decade. The progress of the technology has been much slower than that of resin matrix technology. At the present time, most of the metal matrix composite activities are all of an experimental nature. There are a number of reasons for this slow progress:

1. Metal matrix composite technology has not received the same support as resin matrix composite technology.

2. Translation of fiber properties to a metal matrix composite has not always been successful. The specific interaction between the fibers and the metal matrix, either in the solid or liquid state, is critical. There can be reactions between the fiber and the metal that result in the formation of interfacial compounds which can act as stress concentrators and result in a weakened composite. Conversely, the metal may not wet the fibers so that poor bonding of the fiber and the matrix results. Since metal matrix systems have to be fabricated at elevated temperatures, the mismatch in coefficients of thermal expansion of the reinforcing fiber and metal matrix may result in significant internal stresses, which if not properly released, would weaken the composite.

3. Nonstandard fabrication methods are required to make metal matrix composites because of problems associated with the interaction (or lack thereof) of the fiber and the matrix. A major reason for the rapid development of advanced organic matrix composites was the existence of an established fiberglass fabrication technology that was applicable to the new generation of composites. This is not the case for metal matrix composites. This technology has required the development of specialized fabrication techniques concurrently with materials development efforts.

4. Metal matrix composites are expensive specialty materials that currently cost from many hundreds to a few thousand dollars a pound. At the moment, it is economically difficult to justify using metal matrix composites instead of metals, except for experimental or exotic purposes. There has been a chicken/egg situation in that markets and applications for metal

matrix composites would not develop unless the costs of these materials decrease significantly; at the same time, without definition of sizable markets for these materials, there has been hesitancy to invest major amounts of money in manufacturing development programs and automated fabrication equipment that could result in lower costs. The situation is changing in view of the new emphasis being placed on fabrication technology by DoD, in order to support the development of items of military hardware that have been identified as benefiting from the use of metal matrix components.

Metal matrix composites have been produced in a wide variety of models, ranging from low melting alloys of metals, such as lead and tin, to superalloys that can operate at elevated temperatures. The majority of metal composite work has been concerned with the low density light metal alloys, aluminum and magnesium, with major emphasis being placed on using high performance reinforcement as a means of increasing the service temperature of these metals to at least 600 $^{\circ}$F (315 $^{\circ}$C).

12. Applications of Advanced Composites

As with any product or material, advanced composites have been used or considered for use only in those applications where it appears economically desirable to do so. Advanced composite systems have been readily adopted in systems where their use has proven to be "cost competitive," that is, the total costs of manufacturing a given system are less if advanced composites are used instead of other materials of construction. These are applications wherein opting for the use of advanced composite materials instead of an alternate material lowers design, tooling, and labor costs by a sufficient margin to more than offset any differences in materials and other costs.

Advanced composites are also considered for use in applications where they appear to be "cost-effective." In these applications, the manufacturing costs of the product or system are higher than those of alternate materials, but the use of composites results in a more valuable system for which a market exists. The additional value could take the form of meeting improved performance characteristics or of lowering system operating costs (fuel, maintenance, etc.). The imperative for using advanced composites is not as high in "cost-effective" applications as they are in "cost-competitive" applications.

A major driving force behind the substitution of advanced composite materials for metals in structural systems is that an advanced composite structure will weigh significantly less than a functionally equivalent metal structure. This is of major importance in any dynamic system.

Substitution of an advanced composite for a metal in a structural component can result in weight reduction for the structure that is significantly larger than the difference in the weight of the metal component and the advanced component. Since the composite component is now lighter, any structures needed to support it do not have to be as massive and, in turn, other structural elements that are a function of the weight of the total system may be made lighter. Depending on the specifics of the system, reducing the weight of a component by one pound may reduce system weight by two or three pounds if extensive weight propagation occurs. In all the above applications, some premium is associated with weight savings that impacts on the economic decision of whether or not to use an advanced composite.

In these applications, the extent to which advanced composites are used in a given system is as much a function of the value associated with the weight saved as with the relative costs of using advanced composites or metals in the system. The value of weight savings depends on the application. It is much easier to justify use of advanced composites in applications that place a high premium for weight savings than in those applications where the premium is negligible. Some of the published values of weight premium for different applications are given in Table 15.

TABLE 15. VALUE OF WEIGHT PREMIUM FOR DIFFERENT APPLICATIONS (Ref. 14)

Application	Premium Value of Weight Saving $/lb
Space Vehicles	1000
Hypersonic Vehicles	250
Supersonic Vehicles	200
Subsonic Aircraft	125
Aircraft	50-200
	50-100
Trucks, Bulk Liquid Materials	2
Bridge, Simple	0.06

13. Military Aircraft Structures

Three branches of the U.S. military service have supported the development and use of advanced composites in military aircraft since the early sixties. Since 1960, these programs have progressed through the various phases of materials development, design concepts, manufacturing prototype evaluation, component production, and service experience. Use of advanced composites has advanced from replacement of metal in a few selected secondary structures to current designs in which advanced composites represent 20% of the structural weight, including primary structures critical to the air-worthiness of the plane. Projected usage of composites is expected to increase to 30 to 65% of the total structural weight for high performance fighter aircraft by 1990, concurrently with a 20% reduction in costs.

The major components for which limited production experience has been obtained prior to 1975 are given in Table 16. Other more recent Air Force fabrication programs include the YF-17 fuselage, the B-1 horizontal stabilizer, F-15 speed brakes, F-111 horizontal stabilizer, and ADP (Advanced Development Program) torque box. Air Force advanced composites service experience as of 1975 is summarized in Table 17. The first production advanced composite to reach flight status was the Grumman/ U.S. Navy F-14 horizontal stabilizer which has been in service since 1970. The structure consists of boron-epoxy skins adhesively bonded to a full-depth aluminum honeycomb core, and bonded to a stepped metal pivot fitting. Between 1970 and 1977, 250 F-14s have been built which have required over 1000 boron-epoxy skins. Additional Navy advanced composite service experience with graphite-epoxy components is summarized in Table 18.

TABLE 16. ADVANCED COMPOSITES USE IN MILITARY AIRCRAFT LIMITED PRODUCTION EXPERIENCE (Ref. 14)

Component	Composite	Preproduction Program Duration
F-4 Rudder	Boron-Epoxy	1968-1969
C-5 Leading Edge Slat	Boron-Epoxy	1969-1970
F-14 Horizontal Stabilizer	Boron-Epoxy	1970-1975 (currently in production)
F-111 Wing Doubler	Boron-Epoxy	1971
F-15 Empennage	Boron-Epoxy	1971-1975 (currently in production)

TABLE 17. AIR FORCE ADVANCED COMPOSITES SERVICE EXPERIENCE (Ref. 14)

Component	Material	No. in Service	Approx. Cum. Flight Hours (up to 1975)
F-111 Wing Tracking Edge Panel	Boron-Epoxy	21	32,050
C-5A Leading Edge Slat	Boron-Epoxy	11	23,450
F-4 Rudder	Boron-Epoxy	45	51,000
C-141 Gear Pod Door	Boron-Epoxy	1	8,800
F-111 Underwing Fairing	Graphite-Epoxy	286	44,700
F-15 Empennage	Boron-Epoxy	33	5,000

TABLE 18. EVALUATION OF GRAPHITE-COMPOSITE COMPONENTS IN NAVAL AIRCRAFT (Ref. 14)

Component	No. of Aircraft	No. of Components	Start of Flight Evaluation
BOM 34E wing	8	8	Aug 1973
S-3 Spoiler	14	28	May 1975
F-4J Access Door	4	8	Dec 1975
F-14 Landing Gear Door	9	18	May 1976
F-14 Overwing Fairing	5	10	May 1976
Total	40	72	

A number of important composites applications were included in the B-1 bomber program which was cancelled by presidential order. These included the horizontal stabilizer, vertical stabilizer, weapons bay doors (6 each), avionics access doors (6 each), dorsal longeron, the No. 3 left-side slat, and the No. 3 left-side flap. The total weight of these components, 5581 lb (2536 kg), was 1899 lb (863 kg) less than the all metal baseline system. The life-cycle costs for the composite components ranged from 0.5% to 47.5% less than for the metal equivalents. The 26-ft (7.8m) long horizontal stabilizer alone would have required 1840 lb (840 kg) of advanced composites.

Advanced composites applications in U.S. military aircraft that are currently in production are summarized in Table 19. As mentioned above, the F-14 is the oldest of these production aircraft.

TABLE 19. ADVANCED COMPOSITES APPLICATIONS IN MILITARY AIRCRAFT CURRENTLY IN PRODUCTION (Ref. 14)

			Advanced Composites Utilization	
Plane	Material	Component	Weight per Aircraft, lb (kg)	Weight Percent of Aircraft Structural Weight
F-14	Boron-Epoxy	Horizontal Stabilizer	186 (85)	0.8
F-15	Boron-Epoxy	Empennage	215 (98)	1.6
	Graphite-Epoxy	Speedbrake Fins		
F-16	Graphite-Epoxy	Empennage Fin Covers	180 (82)	2.5
F-18*	Graphite-Epoxy	Upper & lower wing skins, horizontal surfaces, speed brake panel, fuselage ventral section, leading edge extension, landing gear doors	1100 (500)	9.5

*Preproduction.

Use of composites in the USAF/McDonnell Douglas F-15 "Eagle" entered production status in 1975. By January 1978, 200 boron-epoxy empennages and 400 graphite-epoxy speed brakes had been built. The speed brake was incorporated as a running change. Production is of the order of 100 planes/yr.

The USAF/General Dynamics F-16, which went into production in 1977, uses approximately 200 lb (90 kg) of graphite-epoxy per aircraft. Production of this aircraft is about 150 planes/yr and may increase threefold by the early 1980s.

The U.S. Navy/McDonnell Douglas F-18 Hornet multimission strike fighter differs from previous aircraft design in two ways: (1) the airframe will consist of about 10% of graphite-epoxy composites whereas prior aircraft contained less than 2% composites and (2) advanced composites for the first time will be used in a fighter aircraft as primary structural materials in the upper and lower wing skins. Fourteen preproduction aircraft were made for testing and flight evaluation in 1978.

McDonnell Douglas is also developing the AV 8-B Advanced Harrier aircraft for the U.S. Navy. Under a prototype program, two aircraft will be built which will use about 1000 lb (450 kg) of advanced composites each. This represents about 20% of the structural weight of the aircraft. Two prominent changes from prior design practice are the use of composites in the forward fuselage and the use of composites in the wing substructures. The spars and ribs, as well as the wing skins, will be made from graphite-epoxy composites. About 70% of the wing weight (15% of the aircraft structural weight) will be made of graphite-epoxy composite, resulting in a projected wing weight saving of 20%.

NASA will begin flying the Rockwell built HiMAT (Highly Maneuverable Aircraft Technology) (approximately 1978) aircraft that will employ technology that could be incorporated into fighter aircraft of the 1990s. Under a joint NASA/USAF contract, Rockwell is building two Highly Maneuverable Aircraft Technology remotely piloted research vehicles. The HiMAT is currently designed with about 25% of the structural weight of 3400 lb (1545 kg), or 850 lb (386 kg) is graphite-epoxy composites.

Extensive use of advanced composites will be required for the Navy's proposed Type A vertical and short takeoff and landing (VSTOL) aircraft. In this aircraft, a premium is placed on airframe weight reduction because of propulsion weight considerations. For example, in the V-530 design proposed by the Vought Corp. (Division of LTV, Inc.) more than 50% of the airplane empty weight is graphite-epoxy composite.

14. Helicopters

The introduction of advanced composites in helicopter design has been slower than in high performance aircraft described in the Section 13. With the exception of the rotor blades, there is relatively little use currently being made of advanced composites in helicopter structures. The first applications of advanced composites in Army helicopters were made on the Sikorsky/U.S. Army CH-54 in 1972. These were the reinforcement of the aluminum stringers on the tail cone with unidirectional boron-epoxy strips, and a boron-epoxy tail skid. The Sikorsky/U.S. Army UH-60A Blackhawk, and the comparable U.S. Navy SH-60B Lamps, the current state-of-the-art military helicopters utilize about 150 lb (70 kg) of aramid-epoxy composites in the secondary structural elements, and only a small amount of graphite in the rotor structures.

Composites have been used extensively for helicopter rotor blades. Initially, fiberglass composites replaced metal in the blade structure because composites were easier to fabricate, had better resistance, and improved structural damage tolerance. Most blades are now hybrid systems, consisting of fiberglass selectively reinforced with aramid or graphite fibers, such as the Blackhawk and CH-46 main rotor blade which may go into production. A unique application of composites to rotors is the Sikorsky/U.S. Army UH-60 bearingless tail rotor in which a unidirectional graphite-epoxy span extends from the tip of one blade through the hub to the tip of the other blade. This approach has resulted in a simpler, lighter, and more reliable design than would have been possible with metal. A number of experimental blades have been made with boron-epoxy and with graphite-epoxy composites on an experimental basis.

In addition, an Advanced Structures Technology Demonstrator (ASTD) program has been formulated to develop a baseline design that makes maximum use of advanced composites and provides a test bed for structural testing and service evaluation. The ASTD aircraft is intended to overcome the limitations of replacement components and show the synergistic effect of advanced composites on the structural weight, design gross weight, and power requirements. Components that would be made from advanced composites would include the main rotor hub and blade assembly, an all-welded metal composite reinforced gear box, molded composite air frame shell structure, hybrid sandwich floor construction, tail boom assembly, etc.

The gear box would be an interesting application of metal matrix composites. Boeing-Vertol is fabricating an experimental gear box housing (12 in. or 30 cm in diameter) made of alumina reinforced magnesium composite. It is expected that there will be a significant reduction in the noise and vibration transmitted into the aircraft, which place severe limits on the endurance of the flight crews. Similar noise transmission problems exist in many industrial environments and if this program is successful, it would have a potential utility that is much broader than helicopter design.

15. Jet Engine Components

Use of advanced composites in engine structures theoretically offers a major potential for weight reduction, cost reduction, and increased composites include fan blades, stator blades, stator vanes, compressor blades, and frame sections. Extensive use of composites could result in weight and cost reductions of the order of 30 to 45% as shown in Table 20. While organic matrix composites can be used in the cooler regions of a turbine, extensive use of advanced composites would require metal matrix systems. Because of their higher impact resistance and better thernal stability, boron-aluminum composite compressor blades have been used on an experimental F-100 engine (Pratt & Whitney), which was flight-tested successfully for several hundred hours. Borsic-aluminum composite fan blades have been developed by Pratt & Whitney for the JT 8D, a commercial engine that powers the Boeing 727 and the McDonnell Douglas DC-9. Borsic-aluminum composite compressor blades have been developed for use in the compressor section of the TF-30, a military engine for the Grumman F-14. Pratt & Whitney has also developed a graphite-epoxy composite first-stage fan blade for the JT 9D, a commercial engine now in use on the DC-10; and is experimenting with the use of graphite-epoxy composite for fabrication of JT 9D fan exit cases currently made of titanium.

TABLE 20. POTENTIAL IMPACT OF ADVANCED COMPOSITE STRUCTURES IN MILITARY ENGINES (Ref. 14)

		Estimated Savings	
Engine	Pounds of Composite	Weight (lb)	Component Cost (%)
TF 39	186	37	44
TF 34	68	32	31
CF 6	280	37	39
F 101	41	46	38

16. Missile Applications

Rockets and missiles were among the earliest applications of advanced composites, with notable use being made in critical structures, reentry vehicle thermal protection systems, and rocket components. The state of the art in this area is believed to be represented by the TRIDENT (C4) ICBM which used graphite composites and aramid composites to improve range and performance.

Composites will also be used extensively on the new MX missile. Applications would include motor cases (aramid-epoxy), upper-stage adapter structure (graphite-epoxy, possibly graphite-aluminum) and reentry vehicle substructure (graphite-epoxy).

The metal wings currently on the TOMAHAWK cruise missile made by General Dynamics/Convair will be replaced by aramid-polysulfone wings. This results in a light low-cost system of low radar cross section. In future systems, alumina fibers may be added to the composite to improve compressive strength. Production of this system is expected to reach 150 to 200 units per month in the early 1980s (Ref. 14).

17. Reentry Vehicles

Considerable R&D effort has gone into the development of thermal protection systems for reentry vehicles (RVs). Nose tips and heatshields have been a continuing challenge to material developers and missile designers because of the harsh environments experienced upon reentering the atmosphere at high velocities.

The loads imposed on a typical reentry vehicle configuration are shown in Fig. 28 and consist of:

1. Axial compression and bending induced by launch acceleration.
2. Axial and lateral compression and shear induced by aerodynamic pressure.
3. Bending resulting from angle-of-attack flight conditions.
4. Strains resulting from thermal gradients through the heatshield and compression followed by tension as a result of shock loading due to short-time energy deposition at the surface. In addition, the structural heatshield must be capable of supporting internal components.

In the early days, the metal heat-sink approach was used to survive the relatively modest environments of the early ballistic missiles. But as the demands for higher performance were imposed, the material developers and missile designers moved to ablative-type materials and the graphites. The early nose tips were either made of beryllium or fabricated by molding and curing laminated cones of silica phenolic, quartz phenolic, carbon phenolic or a combination of these materials. These materials eventually gave way to graphite and carbon-carbon nose tips which could better withstand the environment.

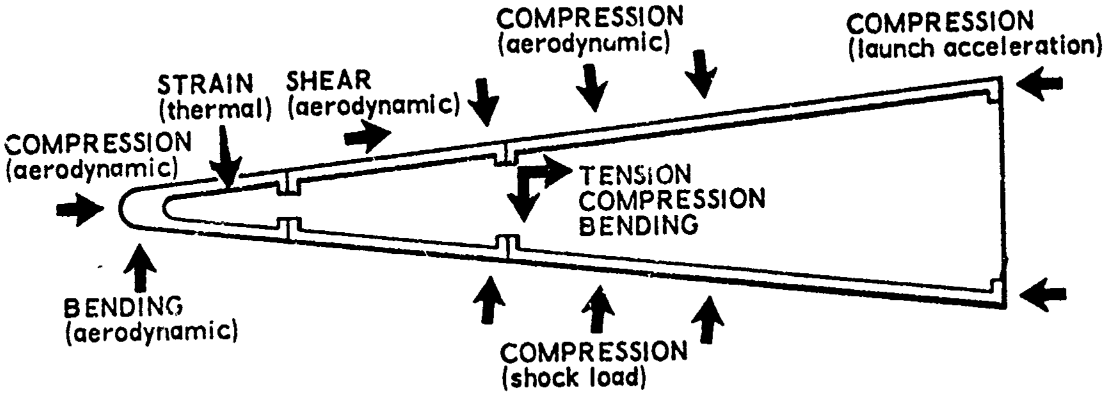

FIGURE 28. Reentry Vehicle Loads (Courtesy of S.L. Channon, Aerospace Corporation)

The early heatshields were either made from beryllium or tape wrapped or laminated fabrics of silica, carbon, and quartz fibers impregnated with phenolic resin. Many fabrication methods have been developed to deal with the many difficulties which arise because of the reentry environment. Carbon phenolic heatshield over an aluminum substructure is the current typical heatshield design.

The composite technology has been advanced to a great degree by the reentry work in the area of fabrication techniques, high temperature exposure, stress problem solutions, etc. The reentry effort also was a driving force behind the improvement in the quality of fibers used in composites and their optimum orientation.

18. Space Systems

Utilization of advanced composites in space structures has increased rapidly in a short period of time. One of the first applications was a cooler cover of graphite-epoxy composite weighing less than 1 lb (0.5 kg) on the 1500-lb (680 kg) Air Force Synchronized Meteorological Satellite (SMS) launched in 1974. EXPLORER 49 had light support booms made of boron-epoxy. PIONEER 10 had an antenna feed strut, platform strut, and magnetometer boom made of boron-epoxy that had a combined weight of about 3 lb (1.4 kg). In comparison, over 58% of the components on Ford Aerospace's INTELSTAT V, which will be launched in 1979, will be made of advanced composite materials, principally graphite-epoxy composite.

The largest single-space system use of advanced composites has been the space shuttle orbiter. The orbiter utilizes about 5000 lb (2300 kg) of advanced composite structure in various components. As shown in Table 21, use of composites in these components results in a weight saving of over 3000 lb (1350 kg) when compared to an all metal structure. This represents about 5% of the structural weight of the orbiter.

TABLE 21. APPLICATIONS OF ADVANCED COMPOSITES
ON THE SPACE SHUTTLE ORBITER (Ref. 14)

Component	Material	Weight Savings, lb	(kg)
Ti Aft Thrust-Structure	Boron/Epoxy Reinforcement	900	(410)
Payload-bay Doors	Graphite/Epoxy	1,070	(486)
Purge and Vent Lines	Aramid/Epoxy	200	(90)
Mid-Fuselage Frame Tubes	Boron/Aluminum	180	(82)
Orbital Maneuvering Pods	Graphite/Epoxy	300	(136)
Pressure Vessel Overwrap	Aramid/Epoxy	435	(198)
TOTAL		3,085	(1,402)

19. Naval Ship Applications

The Navy is currently funding a number of programs to develop various advanced composite structural elements for naval ships. These include hydrofoils, hydrofoil flaps, and submarine sensor masts. Two full-scale [48-in.-long (122 cm)] experimental graphite-epoxy box beams representative of the main-load carrying structural box of the forward foil of the Navy's experimental Patrol Craft hydrofoil (PCH-1, HIGH POINT) are being procured by the David W. Taylor Naval Ship Research and Development Center for evaluation and as possible replacements for stainless steel or titanium alloy beams. Two graphite-epoxy hydrofoil control flaps developed under contract by Boeing Marine Systems will also be evaluated by the Center. These control flaps will be approximately 7 ft (2.1 m) long and 1.5 ft (0.5 m) in chord. One of the flaps will be tested for fatigue and the other flap will be installed on the aft inboard foils of the PCH-1, replacing one of the steel flaps, and will be put into service for evaluation on the ship. It is anticipated that the composite flaps will provide the Navy with its initial demonstration of the use of advanced composites in a marine environment. Aramid composites are being used successfully in racing boats. The speed and endurance record of aramid boats in offshore power races has served to spark the interest in Coast Guard and military patrol boats. Several prototypes are presently in building/testing stages. It is likely that these small patrol craft will be the first production use of advanced composites in military marine structures.

20. Research Support and Utilization

In 1965, the Materials Advisory Board of the National Academy of Sciences recognized the need for a coordinated effort for the fast-growing composites industry and appointed an Ad Hoc Committee on Composites to recommend research and development approaches. The DoD, the Services, NASA, industry, and the university community participated. The Committee's report (Ref. 19) highlighted the types of coordinated research needed to advance composite technology. The following points were considered:

Internal mechanics and modes of failure.

Fabrication.

Environmental effects on properties, including degradation of glass strength, interface strength, and water permeability of plastics.

Interface bonding and chemistry, in general.

Testing and characterization of constituent materials and composites.

Multiaxial load performance.

Quantity production techniques for high modulus fibers at low cost.

Brittleness of ceramics.

Processing short fibers.

Brazing of titanium.

Improvement of polymer matrices.

Multidisciplinary approach to composites research and development.

Testing techniques.

Liaison between Government groups guiding composites development.

Role of composites in future weapons systems.

Limitations of composites.

Regarding DoD and NASA composite R&D planning, Ref. 19 stated the following in 1965:

"The availability and usefulness of composites for DoD and space applications in the future depend on the kind, quality, and amount of research that will be accomplished in the period between now and then. This may seem obvious, but it is significant in the case of composites because, as of now, with the exception of glass reinforced plastics and sandwich construction, and a few specialized applications, there are no high strength structural composites in use in weapons systems or space vehicles. The only hope of realizing the potential of composites is through research and development. Further, since there is very little commercial market for the kind of composites we are concerned with here, most of this research and development will have to be planned, or at least guided, and supported by the Government. The point is that the future of composites, to an extent far greater than with most other materials of construction, will be determined largely by the capability and foresight of those persons in the Services and NASA who are responsible for planning and executing composites research and development programs."

"Obviously, progress in the years to come will be measured by the totality of the results achieved through the work supported by all three of the Services and NASA."

Indeed, Government-sponsored composite programs have guided the systematic growth of the composites technology through its formative years. The Government's development of good quality control technique, raw material specifications, and standardized testing procedures have kept in bound this "sprawling" technology that has many diversified Service and space applications. For example, the Navy has spent millions of R&D dollars on "finger printing" composite materials by studying:

- Reproducibility
- Effect of ingredient sources
- Aging of polymers
- New analytical techniques
- Upgrading quality control
- Determining allowable variations, etc.

Even after the development of a composite, the Services had to characterize the material in the environment peculiar to that Service's application. For example, the Air Force was responsible for funding the development of graphite-epoxy composite. However, in order to apply this material to Navy uses, the Navy had to further characterize the material for compatibility, flamability, attachment capability, water absorption, etc. (Ref. 20).

DoD funding has been the key to the all-important transition for advanced composites and fibers from R&D technology through manufacturing technology (ManTech) to production. A National Academy of Sciences report (Ref. 21) entitled "Accelerating Utilization of New Materials," discusses the transition problem in detail. New materials must have a test bed on which a practical data base can be developed. Also, funding of ManTech programs has been especially important in the relatively young composites field.

Figure 29 from Ref. 22 gives an example of Air Force funding of graphite filament/composite materials programs from R&D through ManTech to applications in aircraft, missile nozzles, etc. The three Services are now funding such programs. For example, the Navy is presently funding ManTech programs to automate the labor intensive procedure of weaving carbon fiber preforms in the manufacture of carbon-carbon composites. Also, the Air Force has been funding ManTech programs to optimize the impregnation and densification processes for carbon-carbon composite manufacture. These programs will help facilitate the utilization of carbon-carbon composites.

It is safe to say that without Government support of the composites technology in the last 20 years, much of the high performance equipment of today (both military and commercial) would not exist.

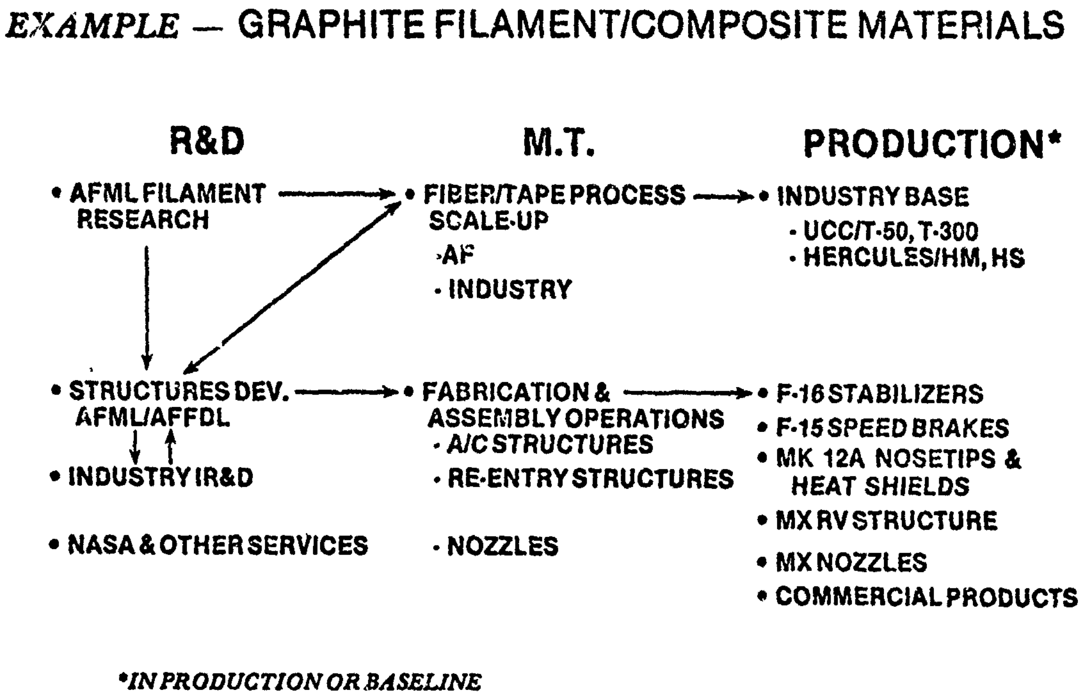

FIGURE 29. Transition of R&D Through Manufacturing Technology to Production (Ref. 22)

C. MISSILE-GRADE GRAPHITES

This section is concerned with graphitic materials which are monolithic in nature, e.g., those that do not utilize high strength fibers. There are basically two types of such graphites, Acheson and pyrolytic, which have DoD applications. Probably the most critical, and certainly the largest, such uses involve thermal protection for reentry vehicles and rocket nozzle structural components. The nature of these two graphites are described below.

The carbon and graphite industry in the United States is a mature one whose sales volume to DoD is quite small compared to the sales to the private commercial sector. The authors roughly estimate the annual sales of commercial graphite materials to be in the neighborhood of $2 to $3 billion while the

DoD probably doesn't buy more than about $20 million of graphite products per year. The largest graphite producer in the U.S. is Union Carbide followed by (not necessarily in order) Great Lakes Carbon, Speer Carbon, Carborundum, and Stackpole. These companies are known for their high degree of interest in proprietary products and processes, and generally are reluctant to accept government R&D contracts that might result in releasing any private know-how into the public domain. Naturally, this has resulted in some rather lively negotiations between DoD and the companies over the years. Nonetheless, the private R&D facilities (at least those with which the authors are familiar) are good and well-staffed and any DoD contractual funding exhibits a great deal of leverage as far as research output is concerned.

1. Missile-Grade Acheson Graphite

Much of this section is excerpted from an early (1966) review of the subject by E.M. Glass (now deceased) for an MAB committee (Ref. 7). It is based largely on a case history of DoD interactions with only one of the graphite companies, the Union Carbide Corporation.

History of the Carbon and Graphite Industry

The industrial uses of carbon and graphite products are based on properties that enable uses as terminal conductors for the conversion of electrical energy to other forms of energy. Electrolytic anodes of graphite are used in the conversion of energy from electrical to chemical; and electrothermic electrodes of graphite are used in converting electrical energy to heat.

The nuclear and aerospace uses of graphite are based on unique combinations of nuclear and physical properties, such as neutron moderation and high strength at high temperature.

Around 1800, Cruickshank discovered that a salt solution can be electrolyzed to form sodium and chlorine. About the same time, Davy demonstrated the electric arc between two carbon rods, forming a concentrated source of radiation for light or heat. These early experiments used primary batteries. From Faraday's discovery of the principles of electrical induction in 1831, the dynamo was developed in the 1860s.

With the advent of the dynamo, major industrial uses of electricity were soon developed. In the early 1870s, arc lamps were introduced for lighting. In 1878 to 1886, Siemens and Herroult invented electric-arc furnaces which were soon adapted to metallurgical processes such as production of ferrous alloys and calcium carbide. In 1886 Hall invented the electrolytic process for producing aluminum. In 1890 alkalichlorine plants were started. In the late 1880s, streetcars were developed that used electric motors, for which carbon brushes were found to be necessary.

The fabrication of carbon and graphite shapes for industrial uses is a comparatively recent invention. Because carbon has no distinct melting point at reasonable pressures, the usual metal-working techniques, such as casting or rolling, cannot be used. The graphite industry uses processes similar to those of the ceramics industry, but differing because of the thermoplastic binder and the need to protect the articles from slumping and oxidation while they are being baked.

The original physical experiments used small rods cut from charcoal or retort carbon. Formed rods were made later by pulverizing charcoal and mixing it with syrup, forming the rods, and baking them. In 1842, Bunsen made carbon electrodes for batteries. During the years 1846 to 1858, inventors developed processes for making carbon plates, using powdered coke and binders such as sugar or tar. In 1878, Brush first used the process of calcining petroleum coke as a preliminary step in the manufacture of arc carbons.

Acheson* invented his process for graphitizing formed carbon shapes in 1895. Before this, the only graphite shapes available were clay-bonded natural graphite, whose properties were only adapted to such uses as crucibles and writing-pencil leads. The availability of Acheson graphite has enabled a major expansion of electric steel and chlorine production, as well as many uses of graphite in the metallurgical, chemical, electrical and mechanical industries.

Until World War II (and even thereafter), the higher volume of defense requirements reflected primarily a greater demand for industrial carbon and graphite products used in producing alloys, steels, chemicals and electricity for war production. Special new needs were generally met with standard processing methods, but using specially selected raw materials and additional purification or impregnation. A case in point was the development of high-purity graphite for reactors in 1942. Another was the use of ATJ graphite in missiles, although this was developed primarily for die applications in the metallurgical and metal-working industry.

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*Acheson's name is now commonly used as a generic description of any bulk graphite made from a carbonaceous binder (such as pitch) and a filler (e.g., coke or lampblack particles) which is then baked (to get rid of volatiles) and finally graphitized at very high temperatures (typically above 3800 °F).

The emphasis of the industry was mainly on the commercial markets. Commercial objectives were more easily defined, and the economics and timing of these markets permitted adjustable trade-offs. Wartime and subsequent defense requirements were not as easily or as well defined nor as flexible with respect to time. Defense work represented less than 2 percent of the industry's business, and this provided little incentive for the industry to invest heavily in scaled-up programs aimed solely at satisfying the DoD's needs. Fortunately, research activities aimed at improving commercial products and processes were equally applicable to defense uses.

Special Emerging Defense Needs

The first major use of graphite for missiles was for rudder vanes, which guide a missile during the firing period by direct action in the jet blast. The German V-2 rocket (at its stage of development in 1944) used rudder vanes of high-density graphite (1.8 grams per cubic centimeter).

Graphite nozzles were first used in 1942-45 for aircraft JATO (jet--assisted takeoff) rockets. Available industrial graphites performed satisfactorily in this application. The JATO nozzles were relatively small, about 2 inches in outside diameter. By about 1952, the outside diameter of nozzles had grown to 5 inches, and erosion conditions had reached the performance limits of industrial graphites.

A major problem facing both the users and the industry was their inability to write realistic, meaningful specifications which related measurable properties to performance characteristics. In a number of Defense programs, this had resulted in unnecessarily high scrap rates and a general lack of confidence in this class of materials, which offered so many unique possibilities for use in very high-temperature applications.

Between 1955 and 1957, several situations developed which were to have a long-range effect on graphite utilization for military missile applications in the ensuing decade. The development of higher energy solid propellants indicated the potential of solid systems for intercontinental ballistic missiles (ICBMs). This led to the establishment of two missile programs, the Navy's Polaris and the Air Force's Minuteman. Early development testing of the new propellants showed that available commercial graphites were only marginally acceptable. This jeopardized the inherent reliability and growth potential of both systems. During the same period, the Army, in developing the Redstone missile, was experiencing great difficulty in obtaining producible lots of graphite materials for jet vanes. The reason was that strict acceptance standards were being imposed on commercial graphite materials not developed for missile uses. About that time, the DoD transferred prime responsibility for land-based long-range missiles to the Air Force, so the development of adequate materials for the associated missile systems became the direct concern of that military department.

DEPARTMENT OF DEFENSE INVOLVEMENT

By virtue of its responsibilities in connection with work for the DoD, the Materials Advisory Board (MAB) sensed the impending emergence of new materials problems and opportunities. In 1956, Dr. J. R. Lane, an MAB staff scientist, recognized a potential problem in the graphite area and suggested to Mr. Verne Schnee, Executive Director of the MAB, that an MAB study of the graphite problems would be appropriate and potentially helpful to the DoD. Approached on this matter by Mr. Schnee, Mr. John Garrett, Staff Specialist, Office of the Assistant Secretary of Defense (Research and Engineering)[1], recognized the importance of such a study. Mr. Garrett then secured the approval of Mr. William Holaday, Deputy Assistant Secretary of Defense (Research and Engineering), for the study to proceed on 7 January 1957. Dr. Lane completed the MAB staff study and published a report on 9 May 1957. The report's conclusions are as follows:

> "1. Better graphite than is now available will be required as components of missiles now being designed.
>
> "2. While the uniformity of graphite now commercially available is considered satisfactory for power reactor applications and most civilian uses, a much higher level of uniformity is needed to satisfy certain reactor and missile structural applications. The adoption of realistic, meaningful specifications (including such as strength, density, grain size, etc.) for critical applications would mark a logical advance in its application. Improvements in nondestructive testing are needed to enable the routine production of grades to specification.
>
> "3. Manufacturers have significantly improved their product over the years, and it is to be assumed that such effort will continue. It may not be reasonable to expect that the present commercial process is capable of yielding a product of adequate uniformity and reproducibility for some future critical military applications. Other approaches to making graphite are known, but apparently are not being actively pursued. Commercial incentives for this work are small, and the government support would be required to achieve the rate of progress which weapon designs call for.

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[1] The Office of the Assistant Secretary of Defense (Research and Engineering) - OASD(R&E) - was the predecessor of the Office of the Director of Defense Research and Engineering - ODDR&E.

"4. The development of superior grades of graphite for use in high temperature applications in such weapons as missiles will lag until performance requirements have been spelled out. This itemizing is clearly within the province of the military and weapon contractors. Involved are not only the accumulation of numerical data, but the determination of the special physical and mechanical properties which control performance.

"5. Specifications for pertinent grades of graphite need to be evolved by the concerted action of producers and users. Designers will be encouraged to specify graphite rather than possible alternate materials, if it can be produced to guaranteed narrow variations in limits on properties. It should be recognized that such practice would raise the cost.

"6. The ability to detect flaws and other undesirable characteristics in graphite is not highly developed. Sponsorship by appropriate agencies of research on nondestructive testing methods would enable the selection, with much more certainty than is now possible, of sound, strong specimens.

"7. By far the greater production of graphite is for grades (electrodes, anodes) inappropriate for use at high temperatures in weapons. Since the commercial incentive for the development of grades which might at some time be of interest to the Defense Department is small, it appears highly desirable to increase the support for research on basic studies and unique manufacturing methods. This should be done, if necessary, at the expense of sponsored research on modifications of the standard production procedure. Applied research, such as on oxidation - and erosion- resistant coatings seems to be needed. Because of its many desirable characteristics, the use of graphite should be encouraged to the fullest extent commensurate with its capabilities.

"8. In view of the extensive AEC program on improved graphite for special reactor applications, new military programs on high temperature graphite would require coordination with those of the Atomic Energy Commission."

The DoD Coordinating Committee on Materials reviewed the staff study at its eleventh meeting, 26 July 1957, and decided to send the study to producers in the graphite field for comment. The Committee also set up an informal working group with the military services and DoD staff to review the present government research and development programs in graphite, in order that an adequate study of manufacturer's comments might be made.

The Working Group's review of government work and its study of replies from graphite companies led to the conclusion that lack of progress in obtaining graphite of proper quality for military application was due mainly to the inability of users to specify the properties needed. It also became clear that university-type research did not fulfill all requirements because properties of the relatively small pieces of graphite involved could not be extrapolated to larger pieces; and that there was a large source of know-how within the graphite industry-heretofore untapped by the DoD-that should be brought to bear on the general problem of obtaining better and more uniform graphite for Defense purposes.

Accordingly, the Working Group proposed to the Coordinating Committee on Materials that a tri-service program in graphite research and development be established, consisting of contracts with the companies manufacturing graphite. It was suggested that the program be managed by a single military department and that, if desired, an MAB technical panel could assist. The Working Group stood ready to establish the objectives of the program, to discuss with the graphite industry the feasibility of a cooperative effort, and to make reccomendations to the Committee on the amount of effort required, including funding. The Working Group, in particular, proposed the establishment of an experimental facility for the pilot production of "missile grade graphite".

The Committee considered and approved this proposal at its thirteenth meeting, 6 December 1957, establishing, in place of the Working Group, a Steering Group for Tri-Service Program in Graphite Research and Development.

At a meeting on 12 December 1957, the Steering Group agreed with the suggestion of the Army member that an Army technologist make a survey of the user field in all three military services and report his findings to the group. Mr. E.J. Dunn, of the Ordnance Materials Research Office, was appointed by the Army member for this task. On 7 January 1958, Mr. Dunn outlined to the Steering Group his proposed action, which included submitting a questionnaire to graphite users. The proposal was approved by the Group. Subsequently, Mr. Dunn visited many plants in the user field and also, with members of the Steering Group, companies in the graphite industry.

Membership in the Steering Group shifted from time to time, but it was essentially as follows:

Mr. J.C. Barrett	OASD(R&E)
Mr. E.L. Hollady	Army Ordnance Corps
Mr. T.E. Hamill	Bureau of Naval Ordnance
Capt. I.K. Holdener	Air Force Materials Laboratory

Mr. Dunn's report, and recommendations arising from it, were discussed by the Steering Group at meetings in June and July 1958, at which time the report was accepted.

As a result of this policy decision and certain interservice decisions as well, the Air Force turned out to be the only military department with a direct interest in the recommended graphite program. In the Polaris program, the Navy selected refractory metal nozzles and laid primary developmental emphasis on pyrolytic graphite. Since its requirements for rocket nozzles were not severe owing to the shorter duration of their firing, the Army was more interested in the technology of solid-rocket-motor cases and devoted its resources to that area.

Already the Air Force Materials Laboratory had independently started to support several small programs related to many aspects of the recommended program. Subsequently, the Air Force continued to analyze the recommendations of the Steering Group to get a better understanding of the problems involved and the level of funding that would be required to carry out the program successfully. During routine management activities a number of studies were completed. Among them were the following:

(1) the reassessment of DoD requirements and their relationship to Air Force needs,

(2) the solicitation of outside expert opinions,

(3) a reanalysis of industrial capabilities,

(4) an analysis of the effect of the graphite industry's proprietary attitudes on program goals,

(5) the determination of the goals' feasibility,

(6) the planning of a program approach, and

(7) an investigation of the availability of funds.

These studies were made during the year September 1958-1959. Final DoD approval and authority to proceed with negotiations for a nominally $12-million, 3-year program were granted early in October 1959. Concurrently, the need for such a program was reconfirmed by the Air Force's hardware, component and other laboratories.

In the September 1958-1959 period, there was considerable interplay within normal Air Force channels to DoD for program and funding approval. It involved the Air Force Materials Laboratory at Wright Air Development Center and the Manufacturing Methods Division of the Air Materiel Command (AMC), both of which had a technical desire to initiate the program. To sell the program as a candidate to start in FY 1960 required the support of three DoD organizations - the Air Research and Development Command (Major Krug) in Baltimore; Headquarters, U.S. Air Force (Colonel J. Hearn and Lt. Colonel J. Shipp); and the OASD(R&E) (Mr. J. Barrett), both in Washington, D. C. This necessitated that Mr. Barrett secure the assignment of $2 million from the OSD (Office of the Secretary of Defense) Emergency Fund to the Air Force for use in the program.

As evidenced by the fact that the anticipated program cost was more than 10 times the total annual budget of the Air Force for exploratory development in ceramics and graphite, funding was a problem. It should be said here that the technical salesmanship of two highly interested Air Force Officers, Lieutenant Giancola and Captain Holdener, was fundamental to the DoD's acceptance of this program. Both were members of the Technical staff of the Air Force Materials Laboratory, Dayton, Ohio.

Funds were also obtained from a number of organizations in the Wright-Patterson Complex that recognized the need and unstintingly provided resources. The program was started with $3.5 million from the Air Materiel Command facilities people, $2 million from the OSD Emergency Fund, $500 thousand from the AMC's Manufacturing Methods Division, and $275 thousand from the Air Force Materials Laboratory. Further, because of the manufacturing technology aspects of the experimental facility, the AMC committed itself to an additional $1 million of support during the program. While the Materials Laboratory prepared to furnish from its normal budget the required balance, $4.6 million, to carry the 3-year program to its conclusion, this was done at the expense of other materials programs in the Laboratory.

After October 1959, the activities of primary interest involved negotiations concerning work areas, program costs, rights to data, special facility requirements, and the securing of deviations from normal Air Force contracting practice, particularly with respect to proprietary information and balance between government and company capital investments-all culminating in the DoD's award of a contract (effective 1 May 1960) to the National Carbon Company, Union Carbide Corporation, for the development of advanced graphite materials and the operation of an experimental facility for the Pilot Plant Production of Missile-Grade Graphite.

The National Carbon Company-now the Carbon Products Division of the Union Carbide Corporation-had by 1958 developed for its own line of products and processes several unique process concepts which could be adapted and scaled up to the quality and size required for defense applications. These included processes for-

(1) producing fine-grain, high-quality graphite ATJ
(2) pressure molding-baking
(3) pressure curing
(4) pressure hot working-graphitizing

The pressure molding-baking process, now covered by U.S. patent 3,001,237, was licensed from the Cuban inventor in 1955. After a major development program, the process was adapted to the production of carbon bricks in minutes, instead of days with conventional processing. In 1957, an automated plant using the new process went into the production of carbon bricks.

Pressure molding-baking is practicable only for baking carbon shapes to around 1800°F, which is suitable for furnace-liner bricks or for carbon shapes that will subsequently be heated to about 5000°F for conversion to graphite. Later developments to adapt this process to carbon blanks for fine-grain graphites resulted in some limitations because of binder-level restrictions imposed by the extreme speed of the process.

The technical work on the molding-baking process was extended to the development of other processes that used pressure with induction heating-such as pressure-curing as the first step for large sizes of fine-grain graphites, and pressure hot forming-graphitizing for recrystallized high-density graphite and for graphite-refractory composites.

It should be pointed out that the use of National Carbon Company Trade Names, such as ATJ, ZTA, etc., to identify the products mentioned results simply from the role of National Carbon Company as the contractor under this particular development program. Use of these designations is not intended to imply that National Carbon Company products are the only graphite products suitable for missile or other DoD or nuclear applications. Other products of other producers have been tested successfully and are available.

High-Density Graphite

During World War II, the postwar build-up and the Korean War (i.e., from about 1941 to 1953), graphite research and development (R&D) was focused primarily on specific needs for nuclear reactors, electrodes and anodes. Graphite-density requirements were met with available process technology, the two principal alternatives being pitch impregnation or the use of superfines in the mix.

In 1909 Acheson had developed the pitch-impregnation process to increase the density of graphite by filling the pores of the carbon blank with a carbonizable material, subsequently graphitized. For special needs, this process could be repeated to reach a graphite density of around 1.75 g per cc.* Another method of making graphite of improved density is to incorporate superfines or furnace black in the mix to fill the voids between coke particles. With a combination of this mix and multiple pitch treatments, it is possible to make graphite with a density of about 1.85 g per cc.

The R&D program maintained by National Carbon since 1900 to expand industrial uses of graphite has built an organization with interdisciplinary scientific and technical skills, equipped with the most modern scientific and special experimental facilities. Related to the management decision to adapt graphite to DoD uses, one of the general R&D objectives was higher density.

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*The theoretical density of graphite is 2.26 g per cc.

During 1944-47 and 1950-52 experimental studies were made on hot and cold pressing of purified natural graphite powders. This hot pressing used graphite tube furnaces for 4500°F to 5400°F, with carbon equipment for pressures to about 6000 pounds per square inch (psi). Though graphites up to about 2.0 density were produced, their engineering properties were not attractive.

In National Carbon's study of the triple point of carbon, small graphite boules of 2.0 density were made by recrystallization from the liquid phase of carbon in an arc furnace under 2000- to 3000-psi gas pressure. These boules had high strength as compared with natural graphite, and their electrical conductivity was about three times higher than that of good artificial graphite. This process, however, would not be practicable for producing engineering sizes of graphite.

The intensive development of the pressure molding-baking process that started in 1955, provided a fund of engineering know-how and stimulated technical awareness of the possibilities in a high-temperature pressure process for carbon and graphite.

During this initial period, about 1952-57, the National Carbon Company's specialists in engineering service to missile and aerospace builders improved the understanding of special technical requirements for graphite in these fields. It was evident that no available graphite would answer all needs; but, because its properties may be varied over such a wide spectrum, graphite represents a family of unique materials for space uses.

In 1957, National Carbon men visited the technical people of major contractors to get their thoughts about graphite-performance needs for current and future designs. This survey revealed great interest in graphite with improved and more reliable high-temperature properties for services involving high degrees of strength, resistance to severe erosion, oxidation and thermal shock. There was also considerable interest in higher density, as possibly related to improvements in properties affecting performance of nozzles, cones, edges and reactors.

In view of this interest in higher density graphite, C.E. Larson, Vice-President for Research of National Carbon, asked V.C. Hamister, Associate Director and Senior Scientist, to study available technology and ideas and to carry out research-type experimental work to establish a principle suitable for development scale-up.

Hamister assigned this project to his engineering assistant, E.L. Piper. After consideration of the principles previously evaluated and the subsequent advances in pressure technology, Hamister recalled observations that he and his colleague, J.W. Gartland, had made in 1937 concerning the plastic flow of carbon beginning at 3100°F and of graphite at 4000°F. He suggested that Piper study the hot pressing of carbon preforms.

Piper first made graphite of 2.06 density by hot pressing a piece of 1.56-density carbon (3/8 inch in diameter by 1 inch) at about 8500 psi at 4700°F in a tube furnace. This key experiment in 1957 demonstrated that hot pressing was a feasible method for producing high-density graphite.

The small size of the research piece did not allow the measurement of engineering properties, and it was realized that it would remain a lab curiosity until scaled up by a major development effort.

The possibility of scaling up Piper's research finding was explored at National Carbon's Fostoria (Ohio) Development Laboratory, which had facilities and know-how for the molding-baking process to around 1800°F by resistance heating of the work piece. New tooling had to be developed to reach graphitizing temperatures in the experimental hot pressing of carbon or graphite preforms. It was found, however, that this process was not as practicable as an alternative developed at the Niagara (New York) Development Laboratory which had facilities and know-how for induction-heated processes. The development engineer there to whom this study was assigned, K.J. Zeitsch, built a small experimental unit to enable hot pressing to 5400°F.

National Carbon continued through 1959 with development work to establish engineering data on the hot-pressing processes for producing high-density graphite. The first scale-up was to 2-inch-diameter blanks for subscale test nozzles, which were first delivered to Aerojet-General Corporation in March 1960. The next scale-ups were to diameters of 14 inches in September 1961 (on contract AF33(616)-6915) and to 30 inches in July 1963.

Although the principle of the process to make high-density graphite is quite simple, the engineering aspects are far from simple because of the extremes of temperature and pressure involved. Thus, major problems have been the development of materials and construction to enable practical and safe operation.

Along with its effect on densification, the hot working of graphites results in a highly oriented recrystallized graphite with a crystal structure intermediate to premium and pyrolytic graphites and with characteristics that cannot otherwise be obtained. The process allows the use of a wide variety of base materials to make graphites with a broad spectrum of properties.

ZTA graphite is only one grade of this high-density type of material; its properties were selected for present service needs. With this hot-forming process,[2] it will be possible to develop other

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[2] Wright Air Development Division, Technical Documentary Report No. WADD TR 61-72, Vol. VII and Supplement, "High Density Recrystallized Graphite by Hot Forming."

high-density graphites with optimum combinations of properties to meet specific needs of new weapon designs. Among the applications of ZTA graphite are the following:

(1) Approved by Aerojet-General Corporation for nozzles of both stages of the Skybolt missile.

(2) Approved by Atlantic Research Corporation for nozzle of the Redeye missile.

(3) Tested by Aerojet-General and Hercules Powder Company for second-stage nozzle of the Polaris A-3 missile. In the tests, its erosion rate was almost as low as that of pyrolytic graphite, but ZTA developed cracks that were due to design factors. For the stacked washer design, pyrolytic graphite was adopted. ZTA was not fully evaluated in the washer-type design.

(4) Approved and used by Westinghouse Astronuclear Division for structural components of NERVA nuclear propulsion reactors. Its properties and uniformity meet these particular service needs better than those of any other available graphites.

Improved Large-Diameter, Fine-Grain Graphites

Flexibilities in graphite fabrication enable to a considerable degree the optimization of properties to meet specific performance needs. For example, graphite for furnace electrodes is designed for maximum resistance to extreme thermal shock, high-temperature strength and electrical conductivity. Similarly, graphite for chlorine anodes is designed for maximum resistance to chemical-reaction conditions and electrical conductivity. The numerous other specialty applications of graphite require a wide range of characteristics in structure and properties; for many of these uses, the technical requirements allow the use of relatively normal grades, whereas other applications are sufficiently critical to justify the use of premium types of graphite.

Graphite processing becomes more difficult as the size of the product increases. Thus the size of the piece needed for an application is a critical factor in the production of a graphite article whose structure and properties meet performance requirements.

Commercial graphites for specialty uses have been developed in three general types:

Low-priced (e.g., AGSR, AGSX)	**Medium-grain structure to 12 inches in diameter; coarse-grain in 14-to 35-inch diameters. May have minor internal voids and laminations. Has good thermal and electrical properties. As size increases, grain becomes coarser, with a reduction in strength.**

Medium-priced (e.g., CS, ATL)	Medium-grain structure in 30- to 50- inch diameters. Has better structural quality and strength in larger sizes.
Premium-priced (e.g., ATJ)	Extremely fine-grain structure to 17 inches in diameter. Structure is essentially free of flaws. Has high strength. Can be machined to very close limits and sharp detail.

In adapting graphite for the most critical applications, the real need is for structural homogeneity and the absence of fissures or localized areas of lower density, strength, etc. Thus, it is a perennial objective of the R&D program to find means for improving structural homogeneity and texture for a larger size range and at a cost consistent with the economics of the material's end use.

The low- and medium -priced specialty graphites were developed from the electrode type, with gradual improvements in quality from general advances in graphite technology. These graphites are satisfactory for the majority of industrial uses.

The premium-priced ATJ specialty graphite was developed to meet the need for highest quality and finest grain in critical uses such as dies for continuous casting of metals and nuclear-reactor parts. The ATJ process could not be practicably extended to pieces beyond about 17 inches in diameter.

For larger sizes of specialty graphite 30 to 50 inches in diameter, it was necessary to use the ATL type of medium-grain structure, which has a lower strength.

As part of the management decision to adapt graphite to new uses, a general R&D objective was to develop graphite of maximum homogeneity and uniformity of properties. In 1950-54 studies of thermosetting binder systems were extended to new resins and sulphur-modified pitches. The expanding development of the pressure molding-baking process (started in 1955) stimulated thinking toward the possibility of using pressure processes in the production of graphites with greater structural homogeneity.

New levels of technical and economic factors associated with graphite were introduced in the early 1950s by missile uses, which emphasized the need for graphites of improved reliability with properties of the highest possible level. About 1955, similar needs were indicated for nuclear reactors that called for large pieces of graphite impermeable to molten metals.

During mid-1955, in view of these product needs and the already promising development of the pressure molding-baking process, C.A. Odening, Director of Development, National Carbon Company, authorized a group of development engineers, led by R.C. Stroup, to experiment with pressure curing of the thermosetting resin binder systems studied in the early 1950s.

The first experiments with pressure curing used a resin binder for test pieces 3 inches in diameter. Although these pieces could be cured, they could not be baked. In 1956 the work shifted to pitch binders with sufficient sulphur to effect thermosetting at around 600°F to 700°F. After successful trials with 4-inch pieces, equipment was improvised to make graphite pieces 26½ inches in diameter by 13 inches.

This experiment showed that the principle of pressure curing could be used for developing a process to make a homogeneous carbon article at around 700°F, which then could be baked and graphitized. The advantage indicated was the ability to process relatively large pieces of fine-grain structure with bond levels optimized for physical properties of the graphite.

As an initial step toward scaling up the pressure-curing process, National Carbon set up an experimental facility at its plant in Lawrenceburg, Tessessee, to do trial work on 17- and 40-inch-diameter test pieces for the Babcock & Wilcox LMFR program. During 1957-1959, this work showed that the process had a potential capability of producing graphite of excellent structure and improved uniformity-if optimum control of the process could be developed, particularly to avoid gradients that caused cracking.

Thus, the National Carbon-supported development of pressure curing, together with precursor work on pressure baking and thermosetting-binder systems, made it possible to undertake a specific development project aimed at the goal established by contract AF 33(616)-6915, "...fabrication of large size pieces of graphite which are equal or superior to ATJ grade and have approximately one-third the standard deviation of the properties of strength and bulk density of ATJ."

For this contract, the development work on improved process equipment, materials and techniques produced RVA graphite in sizes up to 30 inches in diameter by 40 inches in length that had (1) average physical properties close to those of ATJ and (2) standard deviations in strength and density not more than one-third those of ATJ. Under contract AF 33(657)-11304, RVA has been scaled up to a diameter of 58 inches.

This process was also used to develop an even finer grained graphite, grade RVD, for improved resistance to erosion.

Another graphite developed with this process is grade RVC, whose thermal expansion characteristics closely match those of silicon carbide to around 2000°F. Thus, RVC is an excellent substrate for silicon-carbide coatings to protect against oxidation.

(1) RVA was approved by Aerojet-General to be used in nozzles for Algol motors used on the Little Joe and Scout programs.

(2) CFZ(RVA with impregnation) was Aerojet-General's leading candidate material for the first-stage nozzle of Poseidon.

(3) RVD, an all-flour RVA, was tried once unsuccessfully by Thiokol Corporation and Picatinny Arsenal for Minuteman's first-stage nozzle. The failure was due to a misunderstanding in the initial production of RVD, which was later removed.

(4) RVC, a high-thermal-expansion form of RVA, was developed as a machining substrate for silicon-carbide coatings. It was used successfully for the Dynasoar nose by Ling Temco Vought.

The RV series made available large monolithic articles of graphites with fine-grain structure and much more uniform physical properties than previous graphites had. The process is flexible and enables the development of graphite grades with new combinations of physical properties as required to meet new service needs.

Graphite has excellent properties for uses requiring high-temperature strength, thermal-shock resistance, electrical or thermal conductivity, machinability, etc. These properties are particularly valuable for applications such as furnace electrodes, metallurgical process equipment, nuclear reactors and rocket components.

A characteristic restricting some uses of graphite is oxidation, beginning at temperatures over about 700°F. In many uses, such as electrodes, the rate of oxidation even up to 3000°F is not prohibitive, considering all other factors of use. However, there are applications for which oxidation and erosion conditions are extreme, e.g., for rocket nozzles or other high-temperature uses in which structural integrity and strength must be maintained within critical limits. The development of increased resistance to oxidation, therefore, is a natural consideration in the R&D program for expanding the uses of graphite.

Many approaches have been studied for the development of oxidation-protective barriers on the surface of graphite. The first attempts, from about the 1930s to 1951, investigated mainly coatings, such as boric oxide and other ceramic materials.

Beginning in the early 1950s, the need for rocket-nozzle materials greatly intensified the search for protective coatings. During 1953-1957, much work was done on many types of coatings, impregnating or infiltrating graphite with metals to form coatings such as carbides, oxides, nitrides or silicides. The major difficulty with coatings is in getting an adherent and oxygen-impermeable coating which is really effective and mechanically practical. Only a few of these coatings showed a limited usefulness, for example, silicon carbide and silicon nitride.

In its general R&D program, National Carbon has studied the mechanism of carbon oxidation and possible controls. For example, in 1924 protective oxide coatings for arc carbons were studied; in 1935 metal coatings for electrodes; and in 1943 boric-oxide coating for metallurgical molds. In 1948, coatings were classified with respect to their end-use requirements and whether they were solid, plastic or liquid in the operating temperature range. This recognized the value of self-healing for the coating. Studies were made of the oxidation rates of graphites as affected by composition and processing. Fire-proofing impregnations, such as the phosphoric-acid type, were investigated. In 1951, under contract, Armour Research Foundation developed a technique for spraying fused oxides on the surface of graphite, but the oxide films were too permeable. In 1952, a carbon brick composite with boric oxide and silica was developed which formed a self-healing glaze when the brick oxidized. In 1955, under contract, Stanford Research Institute studies zirconium/zirconium-oxide coatings for graphite.

Since the late 1950s, an extensive R&D program has been maintained for the study of properties and the fabrication of high-temperature refractory compounds such as boron nitride, titanium diboride and zirconium diboride. In 1957-58, as the result of progress with the pressure molding-baking process, and the jet torch nozzle test, conventionally processed composites of graphite with titanium carbide, zirconium carbide and boron carbide were studied for possible use as rocket-nozzle materials. Silicon-carbide and silicon-nitride coatings were developed.

The key action leading to the recognition of the possibility of developing the new graphite-base composites was the 1957 decision to study high-density graphite, which led to development of the hot-forming process.

By 1959, the hot-pressing process was making high-density graphite blanks 2 inches in diameter for [illegible] nozzle tests.

In 1960, K.J. Zeitsch, the development engineer working with the hot-pressing process, was trying to make 3-inch-diameter graphite over 2.0 g per cc in density, starting with coke flour. He was able to make pieces of about 2.0 density, but most of them had a conchoidal fracture. To strengthen the graphite, he added metal compounds, which melted during the hot pressing and were squeezed into the pores of the plastically compressed carbon matrix to produce a semialloyed graphite-base composite.

Zeitsch first tried boron carbide as an additive. Physical-property testing showed the first pieces of this composite to have remarkably low permeability. Previous tests with the National Carbon jet torch and [illegible] nozzle tests with Aerojet-General had indicated some correlation between permeability of the graphite nozzle throat and resistance to flame erosion. In view of the low permeability of the composites, Zeitsch tested them for oxidation and found outstanding resistance to oxidation in terms of weight loss. With this promising finding, further investigation was undertaken as part of one of the goals of contract AF 33(616)-6915, described as "...investigation of impregnants, coatings, carbides, fibers, unique processes and unusual carbon base composites."

Working with 2-inch-diameter pieces, Zeitsch carried out tests with a wide variety of additive materials, proportions, and processing conditions to establish general guiding principles for selecting materials to give optimum properties for various operating temperature ranges. In general, the additives can be borides, carbides and silicides of refractory metals. The carbon content of the graphite composites generally exceeds 70 percent by volume, in order to keep the basic properties of graphite.

Special test equipment was developed so that effects of high-temperature oxidation on properties of the graphite composites could be measured. Weight loss by arc image and levitation furnace tests, flexural strength, thermal expansion and thermal and electrical conductivities were measured at exposures up to around 4500°F.

As a basis for size scale-up, a graphite/zirconium-diboride/silicon composition was selected as representing a reasonable balance of properties for present uses. The first scale-up was to 5 inches in diameter to enable initial hardware evaluations. Scaleup to 14 inches in diameter has now been accomplished.

These graphite-base composites are a unique type of high-temperature material that is homogeneous and intrinsically resistant to oxidation. When these composites are exposed to oxidizing conditions, they form a glazelike surface of highly refractory solid solutions of oxides which protects against further oxidation; this surface is self-renewing if damaged. Since the composites are homogeneous, they can be machined without destroying their oxidation resistance. It is possible to mold articles roughly to final shape. These composites have excellent physical properties at high temperatures.

The JTA graphite-refractory composite was used by Rocketdyne in attitude control motors for Gemini and Apollo. These motors must maintain controlled thrust in start-and-stop pulse-type operation over many cycles of varying duration. The hypergolic fuel is so highly oxidizing that conventional graphites do not stand up. JTA can be used in motors with flame temperatures as high as 5000°F with suitable heat-dissipation designs.

JTA was successfully tested for Surveyor by Space Technology Laboratories.

Special Comment

It is of interest to note the assumption, without justification, made in the section above (on "Improved Large-Diameter Fine-Grain Graphites") that large billets of graphite were needed by the large diameter solid motors, particularly for the 33-inch nozzles of the TITAN-III solid strap-ons. The conventional thinking at that time did not seem to recognize that the absolute erosion rate becomes progressively less important as the throat diameter increases. Very large diameter graphite billets were, in fact, developed and manufactured under the Air Force program but proved to be useless for the TITAN-III solid application because of thermal stress failure (which should have been easily predictable by then available codes). Efforts were then devoted to segmenting the large graphite billets but motor testing failure still resulted. Finally, polycrystalline graphite was abandoned for tape-wrapped graphite fabric in a phenolic matrix for the TITAN-III solids.

2. Other Improved Conventional Graphites

All of the information in the remainder of Section VI-C was obtained by probing the memories and old file papers of the following individuals: Jerome Persh (OUSDR&E), William Smith (Super-Temp), James Pappis (Raytheon), Jack Bowman (Union Carbide), Edward Epremiam (National Research Council), Robert Meyer (Aerospace), Robert Feldhune (Navy Surface Weapons Center), Edward Pacquette (Atlantic Research Corporation), Stanley Channon (Aerospace), Donald Schmidt (AF Materials Laboratory) as well as the authors themselves.

As far as the authors can determine, none of the three graphites discussed previously (the ZT, RV, and JT series) were ever utilized in a major DoD system although there were some NASA applications as APOLLO propulsion components. However, one of the other tasks (mentioned previously but not discussed) led to what came to be designated ATJS graphite. As noted, one of the goals was to upgrade the quality control of ATJ so that the deviation of its thermal and mechanical properties would decrease by a factor of two-thirds. Although the production of a more uniform product was the main driving force, a second impregnation and graphitizing step was added so that ATJS has somewhat higher density and mechanical strength than ATJ. There was little or no new science required to produce ATJS; mostly, the advances needed were much better property characterization and manufacturing technology. Union Carbide, of course, had the in-house expertise to pretty well know how these advances could be achieved, but it must be remembered that there was almost no profit incentive for them to do so on their own. The properties and uniformity of the ATJ grade, which was already being produced in 1960 at the Lawrenceburg plant, were more than adequate for the commercial sales which were the overwhelming bulk of Union Carbide's business.

The authors have not yet delved into the details of the ATJS development, but it appears that limited (to DoD) production began about 1964. This was the date when the development work done at Lawrenceburg was transferred to Union Carbide's Niagara Falls facility for manufacturing. Although Aerojet bought some large billets for solid rocket motor nozzle-testing, they didn't work out. As mentioned previously, there seems to be no successful applications for any polycrystalline graphites to solid motor throats, except for noncritical uses such as heat sinks underlying refractory metal throat inserts (a possible exception is the POLARIS A3 second stage, discussed in Section VI-A as a question that the authors were unable to resolve). Some billets were sold for reentry vehicle nose tips to be used on experimental ABRES flight tests, but no deployed Air Force vehicle has ever used ATJS. During 1966 (see Section VI-A) the Navy and LMSC started design on the Mark 3 reentry vehicle, which no longer had compatibility requirements in the Air Force Mark 12 and Mark 17. The Mark 3 had a more stringent operating environment than the previous Navy RVs and a beryllium tip could not be used. Also, the Navy had volume constraints (i.e., the submarine launch tubes) which could not readily be designed away. Thus it was undesirable to use a combination of charring and low-temperature ablators for the nose tip as did the Air Force. Hence, almost by default, the Navy had to go to a noncharring ablator, i.e., graphite. ATJS was the only graphite available that came close to meeting the requirements (especially resistance to thermal stress) and, in about 1968, LMSC started to buy billets for this purpose. ATJS finally obtained a home in a deployed weapon system, the first of the DoD improved graphite developments to do so.

However, by about 1970, Mark 3 flight tests had indicated that ATJS was less than fully satisfactory and greater improvements in both thermal stress resistance and uniformity (to ensure a greater design confidence) were required. With at least partially this motivation, three more-or-less independent development programs started in about 1971, the TS-994, the CMT, and the Graphnol efforts. The way in which each program evolved was different.

The TS-994 had Air Force money put into it in 1971 by AFML (Clarence Pratt), but the work at Union Carbide was actually started in early 1969 by IR&D funded purchase orders out of McDonnell-Douglas Aircraft Corporation (Robert Meyer). McDAC's interest in the subject was, at that time, due to their involvement in two ABRES programs in which it appeared that ATJS would not be satisfactory. The TS-900 series was the internal Union Carbide designation for experimental graphites assigned to the McDAC request (994 was evidently the formulation with the best properties, especially strain-to-failure, to resist thermal stress) and was kept when the Air Force picked up the efforts. TS-994 had characteristics that were very close to CMT (described below) but it doesn't appear that 994 was ever scaled up and used for anything.

CMT graphite development was started by LMSC sometime in early 1971 using Navy Strategic Systems Project Office funding. The initial work was done at Union Carbide's Parma facility. The precise nature of the Navy funding has not been determined. It probably was started as part of the MSAT 6.3 program (the only technology program ever to creep into SSPO's budget) and was then picked up by the 6.4 EDP for the ULMS program which got funded about January 1972. It may also have been financed by monies out of the POSEIDON C3 procurement budget or, conceivably, gotten started out of LMSC IR&D funding. In any case, CMT development was entirely under SSPO and LMSC auspices and meant exclusively for the TRIDENT C4 nose tip and retrofit for the POSEIDON C3. CMT is a double-impregnated graphite like ATJS but with much more stringent manufacturing quality control procedures. It also has a smaller average grain size than ATJS by a technique still proprietary to Union Carbide. It has undergone flight tests and apparently behaves satisfactorily for Navy needs; it is now being produced in a dedicated controlled line facility, at Parma, which was built by the Navy at a rough cost of $10 million. CMT was evidently not made available to the Air Force.

The Navy improved graphites, under the generic title of Graphnol, were the only class (of the three new types) which involved Technology Base (6.2) funding from the beginning. The program was first funded in mid-1971 under the management of the newly established REVMAT project at the (then) Naval Ordnance Laboratory. The major contract was at Oak Ridge's Y-12 (under Walter Eatherly) although other contracts were let (e.g., Oak Ridge National Laboratory, Parma, Los Alamos, and ITT Technology Institute). An estimated $2.4 million in 6.2 funding was spent over the length of the program; since about late 1976, the major efforts have been in a Manufacturing Technology program (at Great Lakes Carbon) to produce billets of 5" diameter and 9" length (of what is designated Graphnol N3M). Unlike the previous two graphite types, Graphnol is not an improved ATJS but is made by an entirely different process not involving discrete impregnation steps. Its resistance to thermal stress cracking (e.g., its strain-to-failure) is better than either CMT or 994 and its overall processing time is two to three times faster than the others (which implies a potentially cheaper billet cost). It is also the only one of the improved graphite types which is completely nonproprietary. A successful ABRES flight test of Graphnol has been made (on a HEART vehicle) and there is a small ground-swell within the Air Force missile community to push it as a satisfactory (and much cheaper) competitor to carbon-carbon composites for future reentry vehicles. The authors will follow this with interest.

Figure 30 shows an illustrative sketch of graphite work (FY 1960-FY 1968) taken from the Proceedings of the DoD Hearings before the House DoD RDT&E Subcommittee of the Appropriations Committee of the 90th Congress (1973) (Ref. 23). It was given by Dr. Malcolm Currie (then DDR&E) as an example of the orderly progression of R&D to satisfy a mission need. The "Advanced RV" application is never defined and the Mark 18 program has been cancelled. For completeness, the pages VI-108 through VI-115 show a detailed breakdown of this 1960-1968 funding exactly as it was presented to the subcommittee. Note that this includes some work not pertinent to the above discussion, such as efforts on pyrolytic graphite and fibers. The funding is almost all Air Force (as mentioned previously); the $10 million of Navy 6.3 money has not been identified by the authors. Apparently the graphite Air Force funding starting in FY 1969 was cut back. A directive from ODDR&E (J. Persh, June 1968) recommended the initiation of a $1.0-1.5 million graphite 6.3 effort in the Air Force ADP 63211F. The authors have not determined the subsequent funding history. Presumably Air Force 6.2 funding on polycrystalline graphite was not kept going, since it was the following year (FY 1970) that the Navy REVMAT project was started with urging from the ODDR&E.

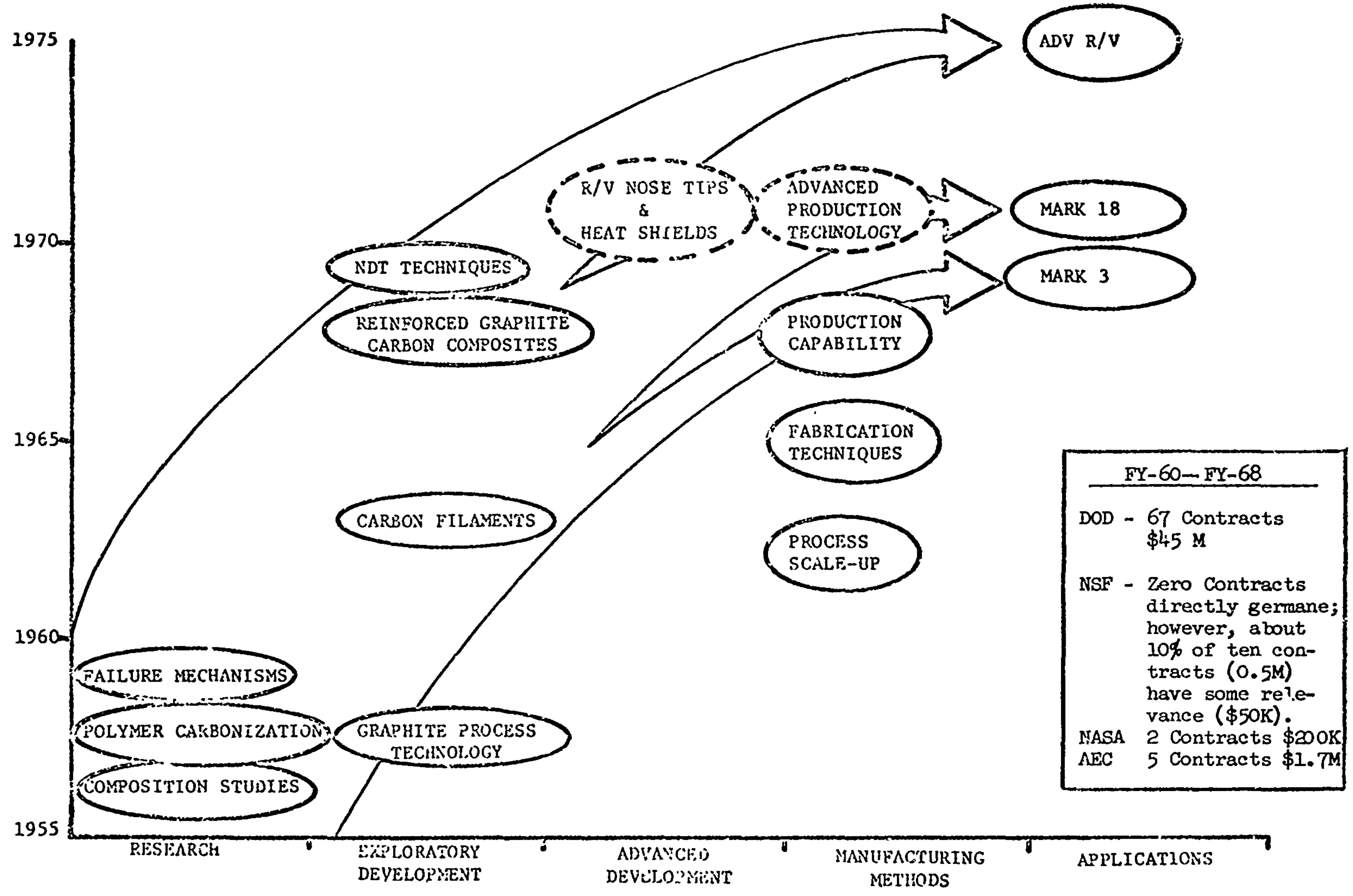

FIGURE 30. Graphitic Materials for Advanced Reentry Vehicle Applications (Ref. 23)

BRIEF DISCUSSION OF TECHNOLOGICAL AREAS

RESEARCH (Funding FY-60 - FY-68 $2.7M)

1. Composition Studies - 7 Contracts $0.8M

Objective: Determine effects of various processing techniques and ingredients on final properties.

2. Polymer Carbonization - 3 contracts $0.5M

Objective: Determine procedures for pyrolizing and processing polymeric fibers into carbonaceous mats, felts, and simple shapes.

3. Failure Mechanisms - 7 Contracts $1.4M

Objective: Determinations of loading and stressing situations which cause failure. Establishment of failure criteria as a function of material characteristics.

EXPLORATORY DEVELOPMENT (Funding FY-60 - FY-68 $16.0M)

4. Graphite Process Technology - 23 Contracts $11.7M

Objective: Development of processing techniques for producing graphites with properties desired - reproducibility studies.

5. Carbon Filaments - 9 Contracts $2.0 M

Objective: Development of techniques for uniformity in thickness and properties - long lengths - preservatives.

6. Reinforced Graphite - Carbon Composites - 12 Contracts 1.8M

Objective: Development of processing for pyrolyzing of organic fibrous shapes and infiltration with pyrolytic graphite. Porosity vs. strength trade-offs. Shape stability under loading.

7. NDT Techniques - 6 Contracts $0.5M

Objective: - Development of procedures for detailed examination and assessment of finished parts without affecting integrity.

ADVANCED DEVELOPMENT (Funding FY-60 - FY-68 AF $18 M) (Navy $10 M)
(New FY-69 - FY-71 $12M)

8. R V Heat Sinks and Heat Shields

a. Application Analysis

Objective: To generate conceptual designs of operational and flight test R/V's and utilize these designs for guidance and selection of technological efforts.

b. Development

Objective: Advance technological development of those graphitic components which hold promise for achieving design requirements, using data from previous exploratory and research areas.

c. Demonstration - Test and Evaluation

Objective: To demonstrate, by laboratory and ground tests, the adequacy and potential of the fabricated components to meet the design requirements.

MANUFACTURING METHODS (Funding FY-60 - FY-68 $9.1 M) 11 Contracts

9. Fabrication Techniques - 3 Contracts $2.6 M

Objective: To develop technology to scale-up fabrication methods developed and verified on small components to determine effects of scale-up on characteristics and properties.

10. Process Scale-Up 6 Contracts $5.1 M

Objective: To develop standardized techniques for fabrication of reliable components. Develop inspection procedures to verify quality and performance of hardware.

11. Advanced Production Technology 2 Contracts $1.4M

Objective: To develop manufacturing methods for producing full scale components on a quantity/quality basis. Develop mechanized procedures for verification of quality, properties and reliability.

TOTAL FUNDING: FY-60 - FY-68 $45 M

(Exclusive of R/V Flight Test Hardware, Rocket Nozzle Applications, Operational R & D and Hardware - Estimated $50 M)

6.1 Research

1. Composition Studies

Title	Contractor	Funding
Binder/filler interactions, synthetic binders, pitch characterization impregnants.(AF)	Union Carbide	$ 250 K
Investigation of carbon base materials (AF)	U of Buffalo	$ 50 K
Evaluation of graphite composites (AF)	General Electric	$ 85 K
Compositional Studies and evaluation of new JT-type materials (AF)	National Carbon	$ 200 K
Hardening additions to pyrolytic graphite (AF)	General Electric Union Carbide	$ 80 K
Studies on hardening additions (AF)	Douglas	$ 25 K
Development and characterization of carbide materials (NASA)	ITTRI	$ 90 K
		$780 K

2. Polymer Carbonization

Carbonization and graphitization studies (AF)	Union Carbide	$ 300 K
Reinforced Pyrolyzed Plastics (AF)	Ling-Temco	$ 160 K
Graphitization studies (AF)	Douglas	$ 60 K
		$ 520 K

3. Failure Mechanisms

Dislocation studies, impurity effects, oxidation mechanisms, thermal stress and shock studies (AF)	Union Carbide	$ 200 K
Thermal shock evaluation (AF)	Space Technology Lab	$ 25 K
Thermal shock behavior (AF)	TRW	$ 40 K
Pyrolytic graphite heat transfer, delamination stress analysis, thermal stress behavior studies (AF)	General Electric	$ 120 K
Radiation effects on graphite (AEC)	Batelle-Northwest	$ 300 K
Mechanisms of radiation effects (AEC)	Gulf General Atomic	$ 125 K
Correlation of microstructure with mechanical properties (NASA)	Los Alamos	$ 100 K
		$1410 K

6.2 Exploratory Development

4. Graphite Process Technology

Title	Contractor	Funding
Process studies in pilot coker (AF)	UCC	$ 3900 K
Coking mechanisms (AF)	UCC	$ 2500 K
Mechanism of pyrolysis (AF)	UCC	$ 1306 K
Graphite base materials (AF)	UCC	$ 955 K
Carbon base materials (AF)	U of Buffalo	$ 44 K
Coatings for graphite (AF)	UCC	$ 632 K
New graphites (AF)	UCC	$ 75 K
New graphites (AF)	Great Lakes Res	$ 76 K
Protective coatings (AF)	Aerojet General	$ 77 K
Coated graphites (AF)	MAN Labs	$ 175 K
Iridium coatings (AF)	Battelle	$ 119 K
Iridium & Iridium alloy coatings (AF)	General Tech Corp	$ 110 K
Graphite design date (AF)	Southern Research	$ 89 K
Thermal shock evaluation (AF)	Space Tech Labs	$ 25 K
Graphite base materials (AF)	UCC	$ 476 K
Data on JT series graphite (AF)	ITTRI	$ 49 K
Thermal shock behavior (AF)	TRW	$ 38 K
Thermal protection materials (AF)	Space General Corp	$ 24 K
Ceramic & Graphite Info Center (AF)	BMI	$ 220 K
Thermal property measurement (AF)	A. D. Little	$ 50 K
Coatings for fuel elements (AEC)	Batelle-Columbus	$ 150 K
Uranium oxide coatings (AEC)	Oak Ridge	$ 200 K
Coating uranium carbide (AEC)	General Atomic	$ 400 K
		$11.7 M

5. Carbon Filaments

Title	Contractor	Funding
Refractory fibers for reinforcement of ablative composites (AF)	High Temp Materials, Inc.	$ 30 K
High modulus carbon fibers (AF)	UCC	$ 999 K
High modulus carbon fibers (AF)	Celanese Corp.	$ 695 K
Weavability of new fibers (AF)	Fabrics Res Labs	$ 29 K
Development of graphite fibers (AF)	Hitco, Inc.	$ 40 K
Refractory reinforcements (AF)	High Temp Materials, Inc.	$ 120 K
Processing of Glassy Carbon Filaments (AF)	Tokai Electrode	$ 16 K
Ceramic & graphitized fibers (AF)	General Electric	$ 35 K
Ceramic & graphite fibers & whiskers (AF)	General Electric	$ 30 K
		$ 2.0 M

6. Carbon Composites and Reinforced Graphites

Title	Contractor	Funding
Graphitic matrix composites (AF)	UCC	$ 200 K
Reinforced pyrolyzed plastics (AF)	Ling-Temco	$ 160 K
Ablative specimens (AF)	Hughes Aircraft	$ 60 K
Reinforced ablative composites (AF)	Avco	$ 166 K
Reinforced ablative composites (AF)	Hughes Aircraft	$ 337 K
Experimental composites (AF)	Monsanto	$ 120 K
Graphite composites (AF)	General Electric	$ 86 K
JTA composites (AF)	Bell	$ 44 K
Evaluation of Refractory Composites (AF)	UD	$ 374 K
Refractory composites (AF)	Solar	$ 40 K
Ceramic & graphite reinforcements & composites (AF)	General Electric	$ 30 K
Refractory composites (AF)	UD	$ 225 K
		$1.8 M

7. NDT Techniques

Title	Contractor	Funding
Radiographic techniques (AF)	OSU	$ 50 K
NDT for graphite (AF)	Avco	$ 158 K
NDT for graphite (AF)	Avco	$ 154 K
Coatings NDT (AF)	Avco	$ 75 K
NDT of graphite (AF)	Avco	$ 30 K
NDT graphites (AF)	Avco	$ 64 K
		$ 531 K

6.3 Advanced Development - 7 Contracts $28 M

8. (a) Applications Analysis New

(b) Development New

(c) Demonstrations - Test and Evaluation New

(Funding FY-60 - FY-68 (AF $18 M) (Navy $10M))

(Estimated $12 M FY-69 - FY-71)

CONTRACTS

MANUFACTURING TECHNOLOGY PROGRAMS

9. Fabrication Techniques

Title	Contractor	Funding
Porolytic graphite nose-tip fabrication (AF)	General Electric Union Carbide	$ 400 K
Pyrolyzed composite prototype nose-tip fabrication (AF)	Lockheed, Douglas	$ 100 K
Monolithic cast 260-inch diameter graphite(AF)	Aerojet	$ 2130 K
		$ 2.6 M

10. Process Scale-up

Title	Contractor	Funding
Process studies and scale-up of fibrous carbon and graphite, graphite refractory metal composites, pressure cured large graphite bodies, high density recrystallized graphite (AF)	Union Carbide	$ 3900 K
Graphite yarn (AF)	Hitco	$ 280 K
Refractory Graphite Base Composites (AF)	Union Carbide	$ 340 K
Pyrolytic graphite deposition parameters and mandrel materials (AF)	General Electric Union Carbide	$ 300 K
Pyrolytic graphite process studies (AF)	Douglas	$ 200 K
Pyrolyzed composite process development (AF)	Lockheed, Douglas	$ 100 K
		$ 5.1 M

11. Advanced Production Technology

Title	Contractor	Funding
Pyrolytic graphite shapes and structures(AF)	General Electric	$ 500 K
Missile grade graphite (AF)	Union Carbide	$ 860 K
		$ 1.4 M

NATIONAL SCIENCE FOUNDATION CONTRACTUAL EFFORTS IN CERAMICS AND GRAPHITE
(FY-60 - FY-68)

MATERIAL	TYPE OF EFFORT	LOCATION	$K
Carbides, nitrides	Diffusion of C & N atoms into Nb and Ta carbides and nitrides. Mechanical and electrical properties.	Minnesota	27
Borides	Bonding in transition metal borides.	Minnesota	48
Binary borides	Crystallography - nature of bonding. Single crystals to be tested for elect. and physical properties	Brooklyn Poly.	30
Ceramics	Effects of high pressure on structure and deformation in crystalline solids.	Case Inst.	91
Carbides	Deformation behavior of sintered carbides under high stress and elevated temperatures	Lehigh	63
Ceramics	Comminution of brittle materials	Penn State	16
Oxides	Diffusion and Defect Studies in high temperature oxides	Minnesota	150
Oxides	Solid state reactions of oxide materials	Penn State	32
Carbon	Interaction of oxidizing Gases with Carbon surfaces	Penn State	27
Ceramics	Metal to ceramic adhesion	U. of Utah	23
		Total	$507 K

About 10% effort relevant -

3. Pyrolytic Graphite (PG)

PG is a form of bulk graphite that is entirely different from any of the graphites discussed previously, having very nearly the characteristics of a single crystal. In general, PG processing consists of depositing carbon from the thermal cracking of a hydrocarbon gas onto a mandrel (usually polycrystalline graphite) which is induction-heated to temperatures of 3600 °F or more. The entire process is at low pressure and generally it takes a long time (of the order of days) to make an end product of practical size. The deposition rate can be as high as about 150 mils per hour but a much slower rate is usually necessary to obtain a material of the best quality. High purity of the mandrels, susceptors, and nozzles is essential and they must be heated in a vacuum prior to the start of PG processing in order to drive off gases and other volatile impurities. After the desired deposition is completed, a slow cooling process is required to minimize residual thermal stresses in the PG component.

PG has nearly the maximum possible density that graphite can have and does not exhibit grain boundaries in the usual sense of the word. There are discrete boundaries, of course, because the material does not grow homogeneously over the surface of the mandrel, but nucleates at preferred spots and then grows outward in sort of an inverted cone. These cones meet each other as growth continues, form discontinuity surfaces and thus create what, in a literal sense of the word, can be called grains. However, unlike conventional graphites, these boundary regions are not generally a second phase and do not show significant differences in density or other properties from the predominant material. This means that PG is nearly impervious to diffusion of most gases or liquids since penetration through polycrystalline materials preferentially occurs in the grain boundary regions. The Atomic Energy Commission had considerable work going on to utilize PG-type materials as a protective coating for nuclear fuel particles long before the DoD started any serious efforts to grow thick shapes for missile use. Another distinguishing feature of PG is its extreme anisotropy of transport and expansion properties. Considering a flat plate, the thermal conductivity in the plane of the plate can be higher by about a factor of 10^3 than that through the thickness of the plate. This property, in particular, coupled with the refractoriness of any graphite, created a great deal of interest in the possible use of PG as a reentry vehicle heatshield or nose tip and a rocket nozzle throat. After serious development work began in the mid-to-late fifties, it became sort of a "wonder material" with a wide variety of potential applications. An offshoot of the PG work was pyrolytic boron nitride (PBN) which has nearly the same thermal properties as PG but is a good electrical insulator. Despite its seeming potential, PBN has had no DoD use (in a monolithic form) to the best knowledge of the authors.

The DoD investment in PG development seems to have largely come from the Navy, more specifically the SP (later the SSP) Office. Although there may have been some earlier, the only Air Force money identified since 1960 was a total of about \$1.6 million in Manufacturing Technology funding. An appreciable but unknown sum was also invested by industry both as R&D and private funds. The principal companies involved were, at first, General Electric and Raytheon. As DoD interest grew, there arose several spin-offs from these two concerns by the simple process of knowledgeable people quitting to set up their own companies. In this fashion, Edward Keon left Raytheon in the late fifties to start High Temperature Materials (later bought by Union Carbide after some disastrous financial experiences) and later William Robba formed Space Age Materials which today is an operating part of Pfizer. Sometime around 1960, William Smith left GE to form Super-Temp, the only one of these spin-offs to still remain in its original form. Both Raytheon and GE first put their own research efforts into processing PG shapes in early 1958. GE did, a little later, set up a production facility at its Metallurgical Products Department in Detroit. Raytheon apparently was in the process of doing so in late 1960 when a number of their people left to form High Temperature Materials. After that, as far as the authors know, Raytheon was never a major factor in PG production (what there was) but maintained a strong research interest.

The Navy SPO sponsored its early PG development in two fashions. The contracts for reentry vehicle use were let directly out of the SP Office while those for rocket nozzle applications were mostly let out of Lockheed. It should be realized that the present-day categorization of R&D into 6.1, 6.2, 6.3, and 6.4 did not exist in those days. These did not come into being until about 1961. The absence of such R&D segmentation undoubtedly contributed to the relative speed with which the SP Office got things done. The research need only be justified by what was needed, not according to an organized set of boundary conditions.

Considering first the reentry vehicle efforts, an applied research program on aeroballistics and materials was initiated by the SPO in July 1957 with the goal of advancing the state of the art in specific areas in the most rapid manner possible. To quote from the "Semi-Annual Report on the Status of the Reentry Body Applied Research Programs" (Jerome Persh, 28 February 1958):

> "The concept of the Mk 2 reentry body is derived from the approved Navy requirements for a higher yield warhead for the follow-up design of the Polaris missile. While the shape of this warhead is completely undefined at this time, it is estimated that the diameter, length and c.g. location will be sufficiently different from the present warhead to necessitate a complete redesign of the body.... The materials program is directed towards the development of new materials which have thermal properties superior to beryllium for reentry

> body application. The philosophy utilized is to determine first what thermal properties can be achieved. The contractor then attempts to make samples of the speculative materials for thermal and other property measurements. These materials are then 'evaluated' by typical trajectory thermostructural computations. These new materials are intended to be utilized in a heat-sink design and therefore exclude materials which ablate, sublime, or vaporize. The latter materials are adequately covered in other ballistic missile projects [i.e., Air Force] and should the Mk 2 design be directed towards ablation, the results of these other projects will be utilized."

Note that, except for the use of beryllium as a baseline and the restriction to heat-sink designs, the RFPs for this work were quite free-wheeling. In effect, the SPO was searching for ideas and did not specify what materials were to be investigated. For some standardization, the SPO utilized NRL for thermal measurements and NOL for trajectory aerothermal computations, although other tasks were assigned to these organizations as well. PG was not emphasized in the very early work; in fact, the first materials contract to a commercial firm was to National Beryllia Corporation to investigate beryllium-beryllia composites (started on 29 November 1957). In about February 1958, the initial PG contract was let to Raytheon to "extend the development which it has begun on the process for making pyrolytic carbon." Raytheon remained the sole contractor for pyrolytic work until the latter part of 1960 when High Temperature Materials was funded to investigate pyrolytic boron nitride and GE to investigate PG with perpendicularly placed refractory metal wires (the latter idea never did work out). In the latter part of 1958, Raytheon also received a contract (evidently from the SPO directly, not through Lockheed) to investigate PG for rocket nozzle use. The total SPO R&D money spent on PG has not been determined but was probably fairly modest since PG was only a part of the overall program. It may be noted that the Mk 2 did go the ablation route and none of the above R&D was directly utilized on that system.

The first significant application of PG to a DoD system was for the nozzle throats of the POLARIS A2 second stage (there were four nozzles). The authors have not been able to trace the DoD development funding other than the SPO late-1958 contract to Raytheon mentioned above. It should be recalled that the A2 program was essentially begun in April 1958 and flight testing started in November 1960. In an informal, undated document out of GE there is a reference to "a large program under joint U.S. Navy and Lockheed sponsorship [which] is now underway to further develop and evaluate PG as well as alloys and other materials made by the same process .... GE is playing a major role in this program as well as carrying out a large amount of work on company funds or for other contracts." From the text references in this GE document, it was probably written in mid-to-late 1960. By this time, GE already had several (perhaps six or seven) furnaces in operation at its Detroit facility and the program referred to (out of Lockheed) was most likely a combination of laboratory R&D and "production" buys for A2 evaluation. Again, it should be remembered that SPO monies were not divided into well-defined boxes in these days (or perhaps even today).

According to the recollections of William Smith,* the A2 nozzle PG buys were started in the late 1950s when Smith was still at GE. The components were washer shapes about 4 inches in the inside diameter. Initially, the procurement was spread equally between GE and High Temperature materials. Then Smith left GE to form Super-Temp which created three suppliers. Shortly after, however, GE dropped out because they couldn't (or wouldn't) meet the price of their two smaller competitors. This may have been sometime in mid-1961 (the first A2 deployment was in June 1962).

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*Personal communication, August 1978.

As far as the authors are aware, GE never again became a factor in PG production.

With the caveat that there is some confusion about the A3, the next (and only other) major application of PG was for all stages of the TRIDENT C4 missile. The work started in the early 1970s and the reader is referred to Section VI-A for details of the configuration. For the C4 first stage, the size of the PG washer required was 9.60-inch inner diameter and 13.55-inch outer diameter with a thickness of 0.385 inch. Each engine has three such washers. It appears (see Section VI-A) that the use of PG was critical to the success of the C4 (it was not clear that it was all that critical to the A2) and new manufacturing technology was required to make such large plates. By now, the only companies in the field were Super-Temp, Pfizer and, to a lesser extent, Union Carbide (because of their acquisition of High Temperature Materials). Union Carbide apparently had little serious interest in the C4 efforts and soon dropped out of the running. It appears that no Navy or Lockheed (actually the Thiokol/Hercules Joint Venture, who was Lockheed's boost engine developer) money went into the large plate scale-up. The main problem consisted of altering the furnace geometry to ensure an even distribution of deposition over the larger area and both Pfizer and Super-Temp invested of the order of $100,000 of their direct money. While each company used a somewhat different growth technique (continuously nucleated for Super-Temp versus surface nucleated for Pfizer), the technical differences in the end product were not significant. Actually, these investments were no great risk since there were only two companies involved and both knew that the customer wanted two suppliers.

It is interesting to speculate on what might have happened to the C4 if PG technology had not been practically ready for use, since the missile itself had no design leeway in volume. One possibility is that the chamber pressure and temperature would have been decreased with a consequent drop in range/payload capabilities. Another possibility is that a crash effort would have been carried out on developing carbon-carbon composites. Besides the out-of-pocket cost of this crash effort (which would have been considerable), there probably would have been a considerable delay in the C4 Engineering Development Program. Since the EDP cost ran around $400-500 million per year, there obviously would have been some major financial penalty for holding up the EDP, but the authors do not now know how to assess it. This might be an important item to try to evaluate since other DoD uses of PG have been minimal. The NIKE-ZEUS used PG as an insulating layer on its graphite hot-gas vane guidance system and the SPRINT used a similar scheme. PG was considered for PERSHING II but was not used. Pyrolytic boron nitride has only had minor uses in specialized electronic vacuum tubes, thus, except for the C4 (and the A2), PG never seems to justify the early enthusiasm displayed.

D. CARBON-CARBON COMPOSITES

1. Introduction

High performance carbon fibers have been used as reinforcements since 1965. They have made available a new class of lightweight materials which have high strength and stiffness. The history of the development of carbon fibers is described in detail in Section B of this Chapter on "Advanced Composites and Fibers." This section discusses carbon-carbon composites and their applications.

The development of high strength carbon fibers and new carbon composite processing techniques has added a whole new line of potential carbon products to the existing applications of molded and extruded bulk graphites. Carbon and graphite occupy a unique position as a class of materials because of their unusual combination of physical and chemical properties at both low and very high temperatures.

Included among the useful properties of bulk graphite are mechanical stability and an increase in strength at elevated temperatures, low thermal expansion, resistance to severe thermal shock, and a wide selection of thermal conductivity. Chemically, they are inert to most elements and practically all corrosives over a wide temperature range. They have abrasion and erosion resistance, are not wet by most molten metals, and are available in porous to impervious grades. Furthermore, they are relatively light in weight, easily handled, and readily machined and fabricated.

Thus, there is a broad field of utility for products made of fabricated carbon and graphite. They have diverse applications in nearly all industrial fields because the unique physical and chemical properties may be altered in various directions by selection of raw materials and control of the manufacturing procedures.

Illustrating the amazing property ranges for well-known grades of carbon and graphite, the electrical conductivity may be varied by a factor of 100, heat conductivity by a factor of 30, and thermal expansion by a factor of five or more. Such wide variations have presented certain problems to investigators. But, conversely, it is this degree of variation that makes fabricated carbon and graphite so adaptable (Ref. 24).

The Air Force Materials Laboratory (AFML) had been supporting the young graphite fiber technology since the early 1960s. The development of commercially available quantities of textile forms of carbon and graphite provided a better means of overcoming many of the limitations of bulk graphite. Resin systems could be reinforced with these fibers and processed much the same as glass fiber reinforced resins. These early carbon-reinforced resin composite systems solved many of the chemical compatibility and fabricability problems experienced with bulk graphite, in spite of relatively low stiffness and disappointing interlaminar shear strength. Developments in both fiber properties and carbon composite technology have since been rapid (Ref. 24).

2. DoD Research and Development

DoD research and development* interest increased greatly when Union Carbide Corporation made available high performance graphite fibers in 1965. The new carbon-carbon composite combined the thermal stability of graphite with the excellent structural properties of a composite. The technology matured during the early 1970s under exploratory development funding for reentry material development (Navy, Air Force) and Anti-Ballistic Missile (ABM) materials tasks. This effort made available a material that could withstand high dynamic loads at high temperatures. Carbon-carbon's high strength and stiffness properties and dimensional stability at elevated temperatures are unique. These characteristics made carbon-carbons a prime candidate for many high temperature applications such as missile nose tips and rocket motor nozzles, etc. The advantages and limitations of carbon-carbons are shown in Figs. 31 and 32.

The thermal properties of graphite and carbon-carbon stand alone in their capability to survive temperatures from 5000 °F to 7000 °F, as experienced under some reentry and rocket nozzle conditions. They have relatively low ablation rates at elevated temperatures. The reinforcement in carbon-carbons makes them more shock-resistant than bulk graphite. The fibers in carbon-carbon act as crack stoppers and prevent catastrophic failure of the material.

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*Excerpts from information received from AFML (Ref. 25) are presented in this section.

THERMAL

- HIGH TEMPERATURE STABILITY
- HIGH SUBLIMATION TEMPERATURE
- LOW ABLATIVE RECESSION
- THERMAL SHOCK RESISTANT
- THERMAL STRESS RESISTANT

MECHANICAL

- PSEUDO-DUCTILE BEHAVIOR
- CRACK PROPAGATION RESISTANT
- NONBRITTLE FRACTURE
- IMPULSE ATTENUATING
- HIGH STRENGTH AND STIFFNESS PARALLEL TO FIBERS
- PROPERTY RETENTION AT ELEVATED TEMPERATURES

GENERAL

- LIGHT WEIGHT
- RADIATION RESISTANT
- TAILORABLE PROPERTIES

CHEMICAL

- INERTNESS

FIGURE 31. Advantages (Ref. 25)

MATERIALS

- LOW OFF-AXIS MECHANICAL PROPERTIES
- LOW STRAIN-TO-FAILURE
- VOID CONTENT AND DISTRIBUTION
- LOW FIBER/MATRIX BONDING
- HIGH THERMAL CONDUCTIVITY
- LOW OXIDATION RESISTANCE
- LOW PARTICLE EROSION RESISTANCE
- HIGH COSTS

PROCESSING

- LONG FABRICATION AND PROCESSING TIMES
- REPRODUCIBILITY

FIGURE 32. Limitations (Ref. 25)

The quality and characteristics of carbon-carbons can be varied greatly. Some of the significant variables are:

- The processing procedures, especially the processing temperature.
- The fiber type, direction, spacing, straightness, and volume fraction.
- The quality of the bond between the fibers and the matrix.
- The number, size, and shape of voids.

There has been an active learning process since 1965 to optimize the manufacture of carbon-carbons and to better understand the critical parameters in their makeup. AFML, the Naval Surface Weapons Center (NSWC), White Oak Laboratory, and the Army Material and Mechanics Research Center (AMMRC) have been active in sponsoring work to advance the carbon-carbon technology. Parametric evaluation of many carbon-carbon variables have been studied by the Service laboratories. One major goal has been to evaluate the range of attainable properties.

3. Preform Construction

The construction of the reinforcements that have been used are listed in Fig. 33. Random, continuous filaments, and 2-D (two-directional) yarns are the simplest methods of reinforcement. These materials give properties that satisfy components with mild requirements. Regarding multidirectional construction, the complexity of manufacture (and the cost) increases with the number of fiber directions. Multidirectional construction requires considerable ingenuity to fabricate and can create a spectrum of properties from nominally the same structure. However, multidirectional construction gives special properties to carbon-carbons that satisfy the demands of many high performance applications. The 3-D material gives much better mechanical properties than, for example, 2-D material, and eliminates delamination which is prevalent in high performance applications with 2-D material. The 4-D, 5-D, 7-D, and 11-D configurations minimize off-axis mechanical weaknesses, which can be important in certain applications. These complex configurations can be useful where there are strong oblique forces encountered or where there are critical requirements for shape stability. A schematic of 7-D construction is shown in Fig. 34.

Two types of 3-D weave are shown in Figs. 35 and 36. The fine weave pierced fabric configuration (Fig. 35) was one of the earliest used. The 3-D orthogonal configuration (Fig. 36) is one of the most popular designs. Refinement of these and other 3-D carbon-carbon fabrication methods has been the subject of considerable R&D effort. The tightness of weave, the fiber volume fraction, the damage to the fiber during weaving, etc., have been important considerations. 3-D preform manufacturers (FMI, Avco, HAVEG, etc.) have optimized their weaving operations through the years to better ensure the output of reproducible and undamaged billets.

The Navy, aware of the labor intensive nature of the 3-D weaving process, started a manufacturing technology (ManTech) program to automate the weaving process. Successful completion of this program promises to significantly reduce the cost of carbon-carbon fabrication. The Air Force has sponsored ManTech programs on the impregnation and densification procedures for carbon-carbons. Optimized procedures are being utilized as a result of these studies.

Another useful preform shape is the cylinderical weave preform. Figure 37 shows the yarn orientation in a 3-D cylinderical weave configuration. This method of weaving is used to make cylinders with large IDs (inside diameter), such as large rocket nozzles. Cylindrical preforms eliminate the need for "hogging out" a large amount of expensive 3-D billet material for large ID shapes. However, the art of making a 3-D carbon-carbon cylinder weave is not as advanced as the art of making orthogonal

RANDOM

- FELTS
- CHOPPED FIBERS
- FOAMS

CONTINUOUS FILAMENT

- FILAMENT WOUND
- SPIRAL WRAP

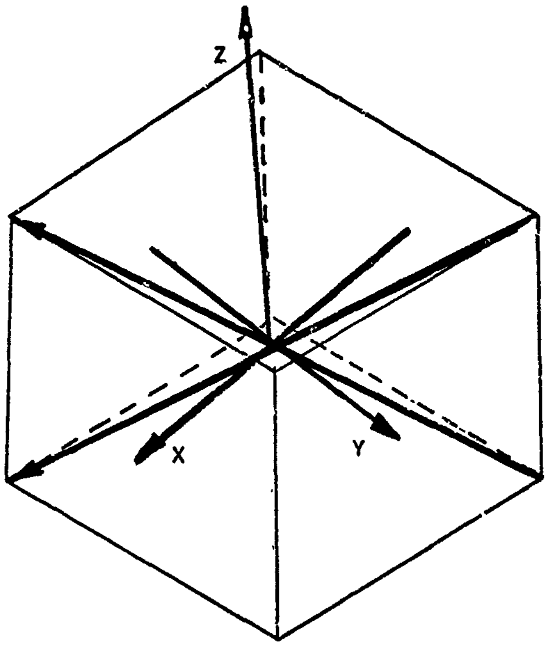
TWO-DIMENSIONAL

- WOVEN TAPES
- FABRIC-WOVEN AND MASCERATED

MULTIDIRECTIONAL

- ORTHOGONAL
- PIERCED FABRIC
- INTERLOCKED WEAVES
- 4-D, 5-D, 7-D AND 11-D
- BRAIDS
- NEEDLED FELT
- STITCHED FABRIC

FIGURE 33. Reinforcement Constructions (Ref. 25)



7-D CUBIC WEAVE
(4 CUBE DIAGONALS AND XYZ)

FIGURE 34. Seven Directional (7-D) Construction (Ref. 25)

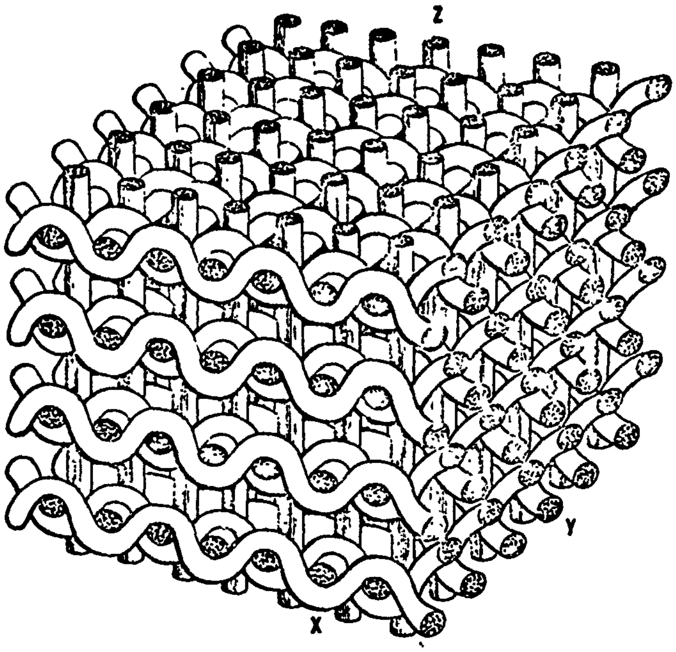


FIGURE 35. Pierced Fabric
(Courtesy of S.L. Channon, Aerospace)

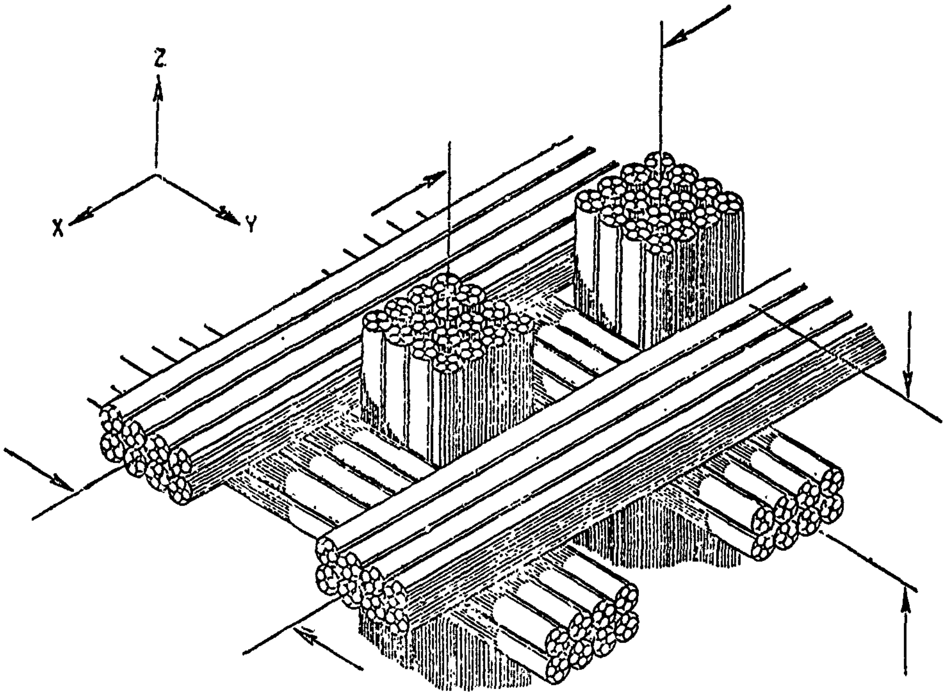


FIGURE 36. 3-D Orthogonal Weave

weave and therefore the time and cost are greater for fabricating cylinders. The costs equalize at about a 7-in. ID nozzle configuration at this time. As the cylindrical weave technology develops, the cost advantage will move toward making smaller nozzles by the cylindrical method.

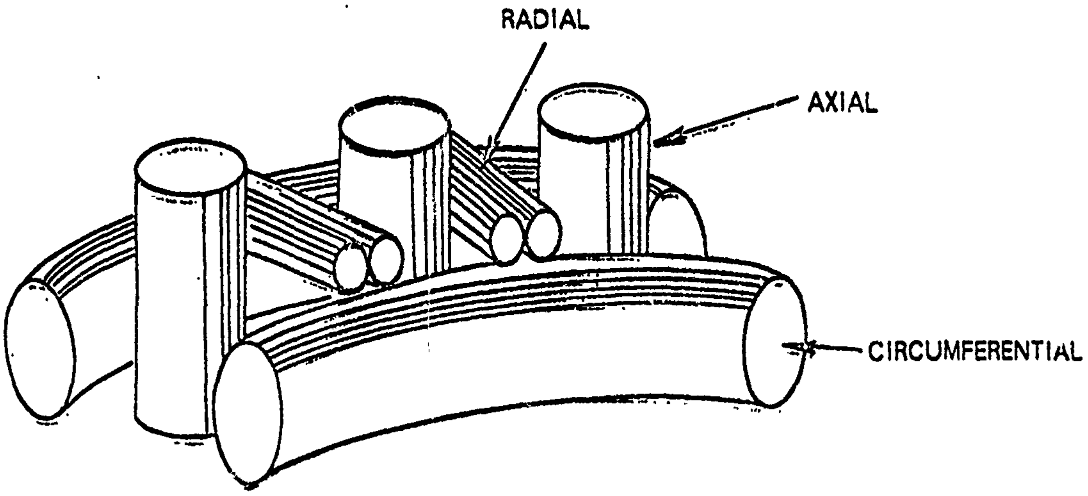


FIGURE 37. 3-D Cylindrical Weave Yarn Orientation (Ref. 26)

4. Matrix

The matrix material used to impregnate or densify carbon-carbon preforms are usually coal tar pitch, petroleum pitch, resins, or CVD pyrolytic graphite. These materials are often used in combination with one another. Considerable variation in properties has been experienced with these materials and with the many process variations that have been used with them.

A typical 3-D carbon-carbon densifying process is shown in Fig. 38. In the process of densifying billets of carbon-carbon, the properties of the fibers can be seriously degraded. The carbonizing cycles at about 800 °C and the graphitizing cycles at 2700 °C take their toll. To densify a carbon-carbon preform to a density of about 1.9 g/cc requires approximately six carbonizing cycles and three to six graphitizing cycles. These temperature excursions degrade the fiber properties. Considerable R&D has been done to reduce the number of cycles required and to thereby minimize this degradation.

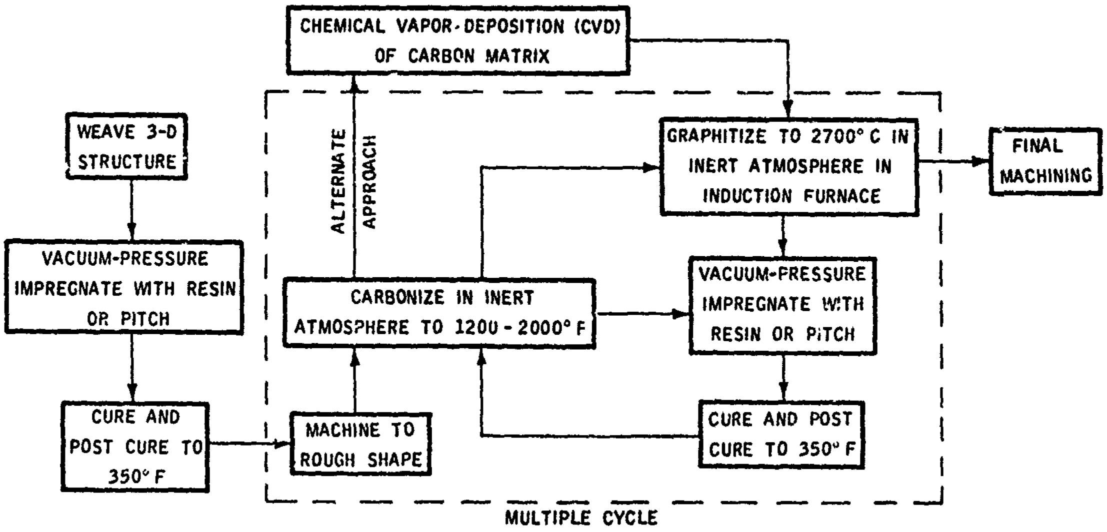


FIGURE 38. Typical 3-D Carbon-Carbon Process (Ref. 25)

The two major procedures for densifying carbon fiber preforms--by infiltration with pitch or resin at elevated temperature and pressure or by chemical vapor deposition (CVD)--are sometimes used together to achieve desired billet properties. There is also a great deal of variation possible in each method, so that optimization of densifying procedures requires considerable R&D effort.

The goals of preform densification are (1) to achieve the desired billet density in the least number of densification cycles and with minimum fiber property degradation, (2) minimize pores that degrade billet properties, and (3) obtain a strong fiber-to-matrix bond. Some ideal matrix characteristics are shown in Fig. 39.

PRECURSORY STATE

- FLUID AT AMBIENT TEMPERATURE
- PENETRATE FIBER BUNDLES
- WET FIBER SURFACES
- WELL CHARACTERIZED, REPRODUCIBLE, COMMERCIALLY AVAILABLE AND INEXPENSIVE
- CURES WITH LITTLE OR NO VOLATILES
- THERMAL EXPANSION/CONTRACTION MATCH FIBERS

RESIDUE

- HIGH CARBON WEIGHT
- DENSE AND STRONG
- LOW AND OPEN CELL POROSITY
- UNIFORM PORE DISTRIBUTION
- REPRODUCIBLE CARBON
- CAPABLE OF GRAPHITIZATION

FIGURE 39. The Ideal Matrix (Ref. 25)

5. Processes

Considerable R&D effort has been devoted to low- and high-pressure impregnation methods for densification. The attributes and limitations of both methods are listed in Figs. 40 and 41. Results of studies are still being assessed to determine the tradeoffs between cost, processing time, and product quality. A sketch of a high-pressure autoclave used for densification is shown in Fig. 42.

The carbonizing and graphitizing procedure must be carefully designed to avoid cracking of the billet. During carbonizing the volatiles must be allowed to escape from the inner cells of the billet. If this is done too quickly, the billet may distort or crack. The high temperature of graphitization (2700 °C) must be regulated carefully to avoid overstressing

ATTRIBUTES	LIMITATIONS
• HIGH DENSITY CHAR	• LARGER NUMBER OF IMPREGNATION/ CARBONIZATION CYCLES
• ADAPTABLE FOR THERMOPLASTIC PITCHES AND THERMOSETTING RESINS	• LARGER PORES
• VERY LARGE SIZE PARTS POSSIBLE	
• LOW FACILITY OPERATING COSTS	
• FASTER EQUIPMENT REPAIRS	

FIGURE 40. Low Pressure Impregnation/Carbonization (LOPIC) (Ref. 25)

ATTRIBUTES	LIMITATIONS
• HIGH CARBON YIELD	• SPECIALIZED, EXPENSIVE EQUIPMENT
• HIGH DENSITY CHAR	• LIMITED PART SIZES
• SMALL PORES	• SAFETY PRECAUTIONS
• UNIFORM DISTRIBUTION OF PORES	
• MINIMUM IMPREGNATION/ CARBONIZATION CYCLES	

FIGURE 41. High Pressure Impregnation/Carbonization (HIPIC) (Ref. 25)

the billet and cracking it. R&D efforts have virtually eliminated cracking or orthogonal billet, but the cylindrical billet stresses are not yet completely understood and sometimes cracking does occur.

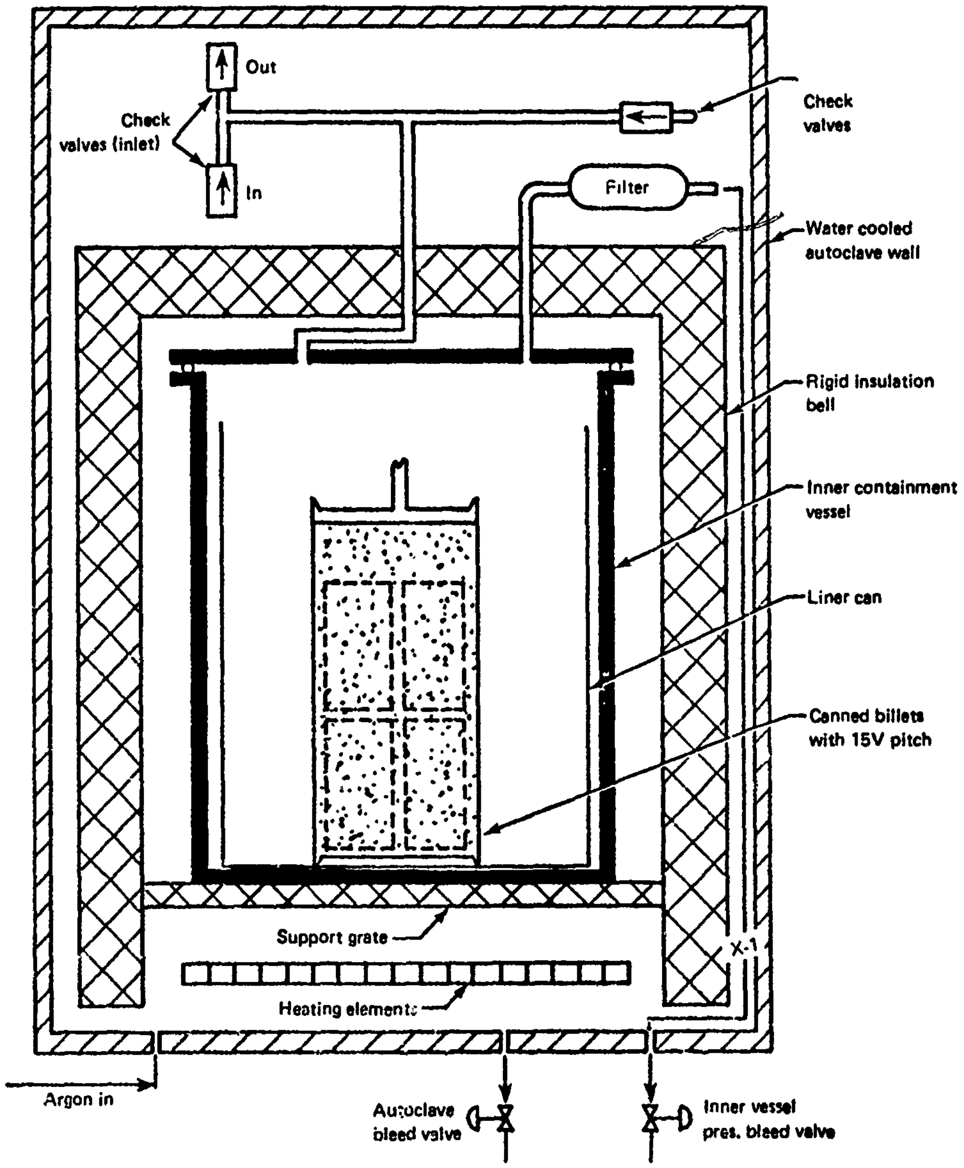


FIGURE 42. High Pressure Gas Autoclave for High Pressure Impregnation/Carbonization Process (Ref. 25)

One of the most important properties affecting the performance of carbon-carbon is the quality of the bond developed between the fibers and the matrix. The high performance properties of the fibers are transmitted through the matrix. If the bond is not adequate, the resultant billet properties will be degraded. One hundred percent translation of properties is not expected. However, through considerable R&D effort, including detailed microanalysis studies and process variation studies, the translation of properties has been increased from approximately 30% to 70% in the past six years (Ref. 26). Gaining this understanding of the interface bonding parameters has not only upgraded the properties of carbon-carbons but has enhanced the quality of the product and its reproducibility.

The need for good fiber matrix bonding justifies the extensive effort that has been expended in attempting to optimize the densification procedures. It is evident that the impregnation/densification procedure and the CVD procedures do complement one another. For example, when the pitch is carbonized, it shrinks. This tends to draw it away from the fibers, thereby eliminating some fiber matrix bonds. On the other hand, the CVD process deposits the pyrolytic graphite (PG) on the fibers and forms an excellent bond. However, because a PG coating is built on all exposed surfaces simultaneously, the spaces between fibers can be gradually sealed off, stopping further penetration of the PG to the interior. So care is required to prevent premature sealing during the CVD process. Considerable R&D effort has been carried out to combine the processes and produce a superior product. One recent study on carbon-carbon procedures (Ref. 27) has concluded that combining the CVD and impregnation processes does give superior billet properties.

6. Applications

Since high performance carbon fibers became available in 1965, carbon-carbon technology has advanced to the application stage in spite of many adverse situations.

- T-50 carbon fiber, which had become the industry's standard, became unavailable.
- Original high cost of fiber.
- Need to develop brittle fiber handling techniques.
- Labor intensive methods required to fabricate multidirectional preforms.
- Relatively high cost and long lead time for delivery of billets.
- Many parameters to be considered in optimizing weave design, matrix materials, and processing techniques.
- High temperature applications of carbon-carbon demand costly and sometimes difficult testing procedures (high temperature testing, rocket motor firings, flight tests).

Technological advances in carbon-carbon technology which counteracted the adverse situations were:

- High performance pitch and polyacrylonitrile (PAN) fibers became available at low cost (pitch fiber costs about 7% of the cost of T-50 fiber).
- Techniques were developed to weave preforms with relatively brittle carbon fiber.
- Automation of weaving process is progressing well (semiautomation has already been accomplished).
- Carbon-carbon billet delivery time has been reduced by 30%, and the cost has been reduced by about 60% in the last 6 years.
- Extensive parametric studies have been sponsored by the Services that have identified many preferential parameters for billet design and processing procedures.

- Credibility of carbon-carbon materials in high temperature applications was established through highly developed test methods. The results qualified carbon-carbons for rocket motor and flight-test evaluation.

7. Reentry Vehicles

Reentry vehicle environment presents one of the most severe conditions that materials are required to withstand. This includes temperatures up to 7000 °F, high velocity conditions, axial and side forces, possible particle (rain, ice, etc.) impact, and high stress buildup due to transient conditions. As the missile performance requirements changed through the years, material for RV heat protection components (nose tips and heatshields) advanced from metals to ablative resin composites to graphitic-type materials, such as graphite and carbon-carbons for nose tips and carbon phenolics for the heatshields. Graphite is used on the current Navy C4 nose tip but may be replaced by a carbon-carbon nose tip for future systems.

Although both graphite and carbon-carbon nosetips perform well in present-day RV environments, graphite requires more nondestructive testing (NDT) and inspection than does carbon-carbon to establish the same margin of safety. Carbon-carbon preforms can be inspected during weaving for flaws, and missing fibers can be detected on the finished billet. Critical flaws in graphite are difficult to detect.

Regarding rain and particle erosion of RV nose tips, no satisfactory material has been developed that has superior erosion resistance to carbon-carbons and that also can meet the ablation requirements for the RV. Considerable R&D effort has been sponsored by the Services to develop materials combining the properties of erosion-resistant carbides (e.g., tantalum carbide, hafnium carbide) with carbon-carbons. A product with satisfactory ablation/erosion properties is the goal.

Some typical properties for a carbon-carbon billet are shown in Table 22.

TABLE 22. TYPICAL 3-D CARBON-CARBON PROPERTIES (Ref. 26)

Tensile Strength	X-direction	20 ksi
	Z-direction	44 ksi
Compressive Strength	X-direction	16 ksi
	Z-direction	23 ksi
Thermal Expansion	X-direction	5.4 mils/in.
	Z-direction	4.8 mils/in.
Thermal Conductivity	X-direction	$320 \ \frac{\text{BTU - in.}}{\text{hr-ft}^2/^\circ\text{F}}$
	Z-direction	$480 \ \frac{\text{BTU - in.}}{\text{hr-ft}^2/^\circ\text{F}}$

8. Nozzles

The years of development work on carbon-carbons under the Service-sponsored RV and ABM programs, made an advanced carbon-carbon technology available to the nozzle community. This technology transfer was affected through concerted effort and cooperation, especially on the part of Navy and Air Force Command and laboratory personnel. The years of RV experience have highlighted several potential advantages of carbon-carbons over other candidate nozzle materials. In comparison to most of the materials presently used, the 3-D carbon-carbon has the potential for (1) improved strength, (2) greater strain-to-failure, (3) unitized construction, (4) a lower coefficient of thermal expansion, and (5) a reduced susceptibility to gas leaks. Additionally, the erosion rates (with acceptable roughness) on the order of 2 to 10 mils per second are within the range where grain and throat geometry modifications can be made without an appreciable loss of I_{sp} (Ref. 28). The advantages of carbon-carbon over the existing system and the other primary candidate material are shown in Table 23.

TABLE 23. NOZZLE MATERIAL COMPARISON (Ref. 28)

	Operational	Developmental	
	Tungsten	Pyrolytic Graphic	Carbon-Carbon
Weight	Heavy	Medium	Light
Survivability	Temperature limited	Medium-high corrosion	Low erosion
Reliability	Acceptable	Questionable	Good
Design Complexity	Complex	Complex	Less complex
Efficiency	Very low	Low	High
Cost	High	Very high	Moderate

The Navy Fleet Ballistic Missile (FBM) program has upgraded the nozzle throats, through the years, to withstand the increasingly harsh environment. The propellant advances, which have given major performance increases for each new generation of ballistic missiles, also produce an extremely harsh environment which requires better materials to ensure nozzle survivability. The current Navy C4 missile uses pyrolytic graphite (PG) inserts in the throat. Due to earlier failures of this design, however, the Navy had evaluated 3-D carbon-carbon throats that performed well. Changes in the PG nozzle design corrected the problem and it was approved for use (see Chapter VI-A).

Reference 29 discusses comparative nozzle tests conducted on a conventional PG nozzle design and experimental 3-D carbon-carbon simplified nozzle designs. The prefired and postfired measurements at each location were compared to determine eroded depth. Figures 43 and 44 present the pretest/posttest erosion profile for typical designs. The conventional PG nozzle developed flow discontinuities. The 3-D carbon-carbon nozzles had more uniform erosion and retained their original shape better than the PG nozzle. The throat section of the PG nozzle eroded less than the 3-D carbon-carbon nozzles but the efficiency of the 3-D was greater.

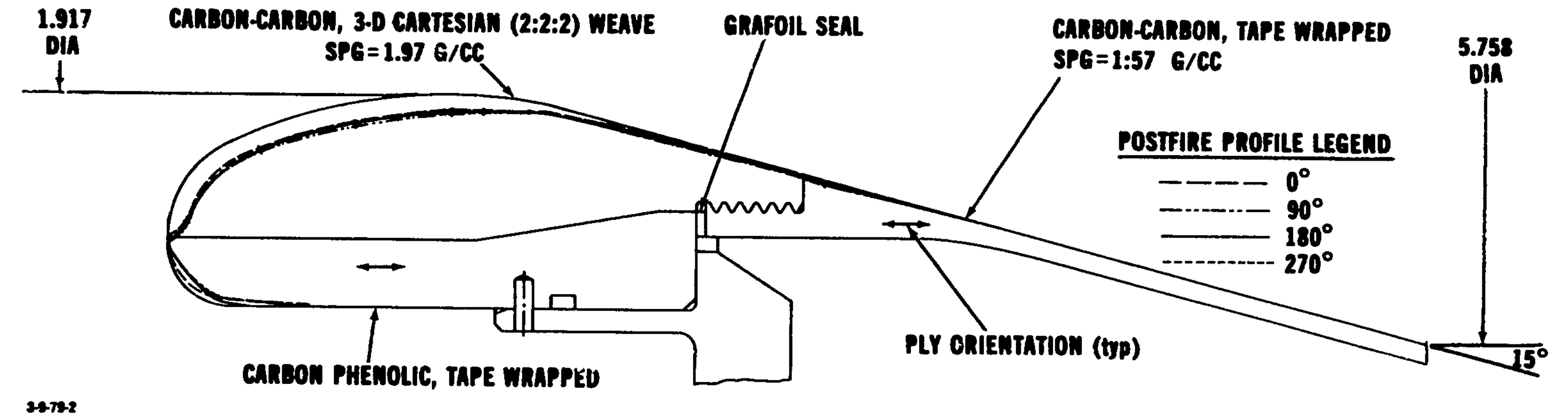


FIGURE 43. Prefire/Postfire Profile, Carbon-Carbon Throat Nozzle (Ref. 29)

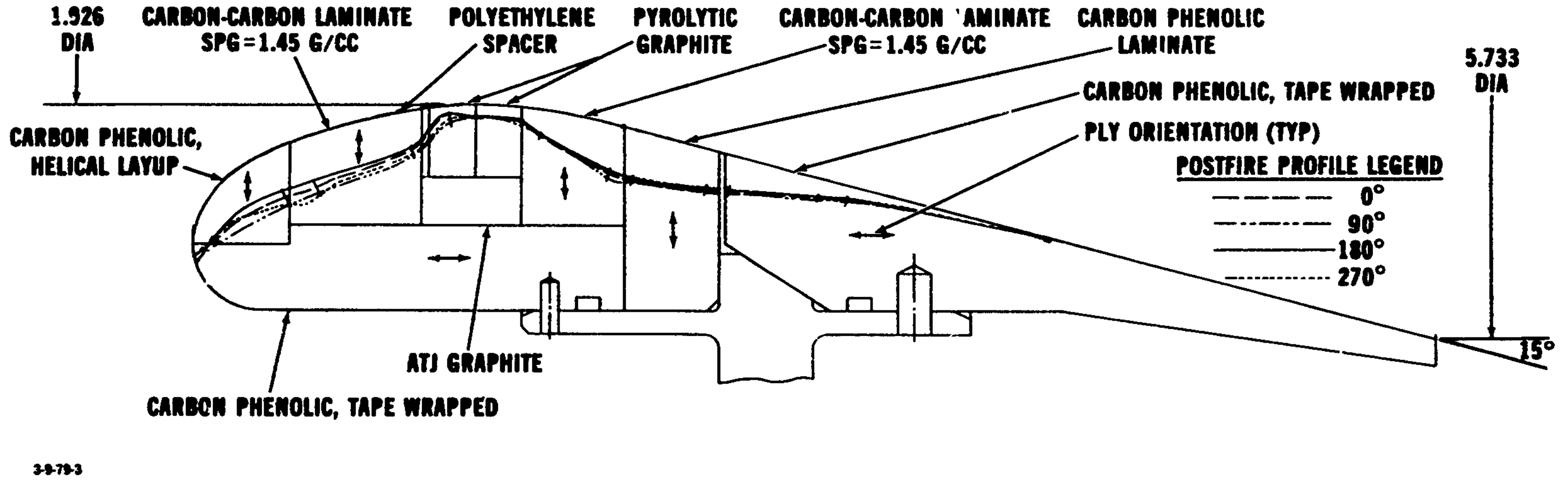


FIGURE 44. Prefire/Postfire Profile, Conventional PG Throat Nozzle (Ref. 29)

Because of the simple design of the 3-D nozzle and its efficiency, designers feel that the 3-D carbon-carbon integrated throat and entrance (ITE) will be used in future designs to replace the more complicated PG nozzle.

The severe environment, coupled with an increase in nozzle throat size and the desire to reduce nozzle weight, have driven the Air Force nozzle designers toward the use of high density multidirectional woven carbon-carbon composites. The Air Force invested some $3.5 million between 1968 and 1975 in developing nozzle throat inserts with pyrolytic graphite or pyrolytic graphite/silicon carbide coatings. Small-scale (3.5-in. inside diameter) nozzles were built, tested, and were extremely successful. However, attempts to scale to larger (7 in.) coated parts led to difficulties in manufacturing nozzle parts with sufficient structural integrity. At that time, carbon-carbon 3-D materials became the most promising materials for that generation of rocket nozzles (Ref. 28).

9. Funding

The funding expended in the development of the carbon-carbon technology to date is approximately as follows:

Navy	$10 million
Air Force	$10 million
Army	$ 2 million
Other	$ 3 million
Total	$25 million

E. TITANIUM DEVELOPMENT

In the mid-1940s titanium was just a laboratory curiosity, but its potential was recognized by both government and industry. The massive support of the government (mostly DoD) is evidenced by the 1970 R&D contract years expended on titanium development from 1945 to 1965 (Fig. 45)*. The impetus for this effort was the potential of titanium high temperature alloys to become the strongest structural alloys known in proportion to their weight, with the added bonus of high corrosion resistance. In the 1940s NAVSEA, NAVAIR, AMMRC (Watertown Arsenal), Bureau of Mines, some universities, and industry were involved in an effort to develop the potential of titanium.

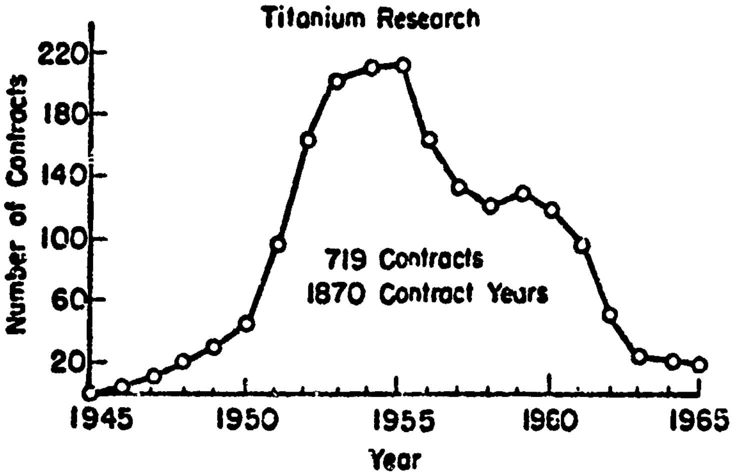


FIGURE 45. Distribution of U.S. Government-Supported Research Contracts from 1946-1965 (Ref. 30)

Some facets of the intense government support that gave the titanium development an early boost were:

- Early plants were built by industry assisted by fast write-off by the government.

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[*] Excerpts from Ref. 30 are indicated in the material quoted in the following paragraphs.

- Sponge production was subsidized by government purchase contracts.
- Research on all facets of titanium metallurgy was conducted by government laboratories (physical metallurgy) and industry (process metallurgy) with assistance from universities and research institutes (Ref. 30).

"During the period of the 1950s, the extensive government support of physical metallurgy provided a large body of information on phase diagrams, crystal structure, and mechanical properties as affected by composition, heat-treatment, and processing history. Industry support (metal and aerospace) concentrated on alloy development, product and process optimization, and fabrication problems. The Titanium Metallurgy Laboratory* was funded by the DoD to assist in the all-important process of disseminating the accumulating titanium technology to hasten the process of utilization. The DoD titanium sheet-rolling program was a response to the needs of the aircraft industry that was responsible for the development of fabrication processes for producing flat, thin-gage sheet without defects and within commercial thickness and flatness tolerances."

"On the other hand, the diminished government support for titanium research and development during the 1960s has been more strongly oriented to current applications and is characterized as applied research. The titanium industry has carried the brunt of alloy, product, and process development, and the aerospace industry has carried forward with the practical but detailed research in production processing and fabrication, as well as detailed characterization of new and old alloys in the titanium family. This latter effort is an important one and is tied closely to the problem of evolving structural design methods and confidence."

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*Forerunner of the Defense Metals Information Center.

Titanium is widely used in the aircraft industry, both military and commercial. It is used in airframe construction and in jet engines where titanium's corrosive resistance at high temperatures has been exploited.

"To illustrate the growth in use of titanium in airframes, Tables 24 and 25 [identified in Ref. 1 as Tables 2 and 3] provide a brief history taken from more extensive case histories provided by Boeing Airplane Company and Lockheed-Georgia. In both cases the footnotes to the tables clearly show that economic factors have played a major role in determining the rate of incorporation of titanium in commercial and military aircraft. In addition, special properties such as heat- and corrosion-resistance and fracture toughness and a good strength-to-weight ratio provided continued reasons for using titanium. Many applications in the mid-1950s also were the result of USAF stimulus, even though it was not cost-effective. With regard to the use of titanium in cargo and transport jet aircraft, with but one exception, each succeeding airplane had a larger use of titanium than did the previous one. Sometimes the reasons for additional use were a consequence of the special properties of titanium. The major reason, however, was an economic one."

Cancellation of the Supersonic Transport (SST) and the B-1 bomber programs considerably reduced the expected titanium requirement. However, the high quantity use of titanium in the F-14 and F-15 aircraft was not contemplated. If the SST had been built, it would have used about 140,000 lb of Ti per aircraft. Aluminum could not survive the aerodynamic heating on the SST so that the use of Ti for the frame would have been mandatory. The Concorde uses about 16 tons of Ti milled products per aircraft. Twenty-five percent of the F-14 aircraft's weight is Titanium--some in the frame and some in the engine. Thirty-four percent of the F-15 frame is titanium and 25% of

TABLE 24. UTILIZATION OF TITANIUM IN PRODUCTION IN BOEING AIRCRAFT (Ref. 30)

Year	Aircraft	Pounds/ Aircraft	Why Used?
1950	B-17	100	Weight savings, heat resistance, experience
1952	B-52A	660	Weight savings, heat resistance
1956	B-52G	2,000	Same as B-52A, push by USAF
1958	707	180	Weight savings, heat resistance
1963	727	650	Weight savings, heat resistance
1967	737	750	ight savings, heat and corrosion resistance
1969	747	8,150	Weight savings, heat and corrosion resistance, cost-effective
1970	SST*	140,000	Temperature eliminated aluminum structure, titanium most effective material

*Program cancelled.

During the period 1950 to 1970, the average cost per pound decreased from $18 to about $6. From 1963 to 1970, the utilization factor (ratio of amount purchased to amount used) decreased from 7 to about 3.5.

TABLE 25. UTILIZATION OF TITANIUM IN PRODUCTION IN LOCKHEED-GEORGIA AIRCRAFT (Ref. 30)

Year	Aircraft	Pounds/ Aircraft	Why Used?
1954	C-130	300	Weight savings, heat resistance
1959	C-140	100	Weight savings, fracture toughness
1961	C-141	600	Weight savings, heat resistance
1965	C-5A	9,950	Weight savings, heat and corrosion resistance, fracture toughness

During the period 1954 to 1965, the average cost per pound decreased from about $18 to about $7. In the same period of time, the utilization factor decreased from 6 to 3.

the engine weight. Titanium is used in parts of some military frames simply because it does not give a weight disadvantage.* This will ensure survival of these areas of the structure if more severe exposure is required in the future.

NAVSEA has continued to fund titanium effort through the years with NRL and NSRDC, Annapolis, supplying the laboratory effort. Titanium was considered for hull application for deep-diving submarines because it is nonmagnetic and corrosive resistant and has deep-depth capability. Serious effort to study candidate hull designs submitted by titanium producers was started in 1959. This led to the development of alloy Ti821 (8Al-2Cb-1Ta) and then to Ti721(7Al-2Cb-1Ta). Ti721 had better weldability than Ti821 but it was susceptible to stress corrosion cracking (SCC). The SCC of Ti721 was found to be dependent on its aluminum content and the absence of the beta phase. An alloy more resistant to SCC, Ti100(6Al-2Cb-1Ta-0.8Mo), was subsequently developed. A 1.9-inch-thick Ti100 hull on the submersible ALVIN gave it twice the depth capability and payload of a hull made of 1.33-inch-thick HY-100 steel. In 1969, two 3-inch-thick hulls were made, one for ALVIN and one for testing. Another submersible, SEACLIFF, uses 2.87-inch-thick Ti100 which replaces a 1.5-inch-thick steel HY-100 hull and increases the depth capability by about three times. The SEACLIFF work is under way and will be completed in 1982.

The application of superplastic forming techniques to titanium in the last three years and the diffusion bonding capability have made feasible the fabrication of complex shapes from titanium. NAVAIR funded early work in superplastic forming but industry picked up the effort and has made great strides in perfecting the procedures. This should expand the future application for titanium.

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*The information contained in this paragraph was provided in Ref. 31.

The following excerpts are taken from Ref. 32.

"Subsonic transportation applications of titanium have been gratifying but modest. Highlight items on the L-1011 include electron beam welded forgings in the pylon boxes, center engine support forging, fuselage fail-safe straps, APU compartment structure, and a low cost precision forged tail bumper support. These applications have been predominantly Ti-6Al-4V."

"On the other hand, the SR-71 is a very high-performance aircraft which holds the world's speed record and is a titanium airframe. It is interesting that the SR-71 is, in a sense, the future that happened yesterday since it is now midway in its second decade. Ti-6Al-4V, Ti-5Al-2.5Sn, and Ti-13V-11Cr-3Al were employed in sheet, plate, extrusion, and forging product forms. Classic manufacturing technology (hog-out, hot form, spot weld, fasten) was used throughout."

"But the high performance vehicles of the future will require a far more avant-garde production technology of economic viability. The cold-rolled, cold-formable, age-hardenable beta alloys represent a long-awaited breakthrough (Fig. 46). This study shows the results of a cost-benefit analysis based on use of beta alloy materials in fabricating 27 APU compartment sidewall parts presently fabricated in alpha-beta materials. Where stiffness in compression dictates the design, use of the beta alloys would impose a weight penalty. However, even in stiffness-critical designs, such a penalty must receive a very rigorous tradeoff against the many advantages. In addition to lower part fabrication costs, the beta materials excel in raw-stock tolerance and finish control, raw-stock costs, stress corrosion resistance, thermal stability, toughness and crack growth resistance. They are compatible with the superplastic forming and diffusion bonding (SPFDB) and low-cost titanium (LCT) weld bond and isothermal brazing advanced assembly concepts now under government and industry development."

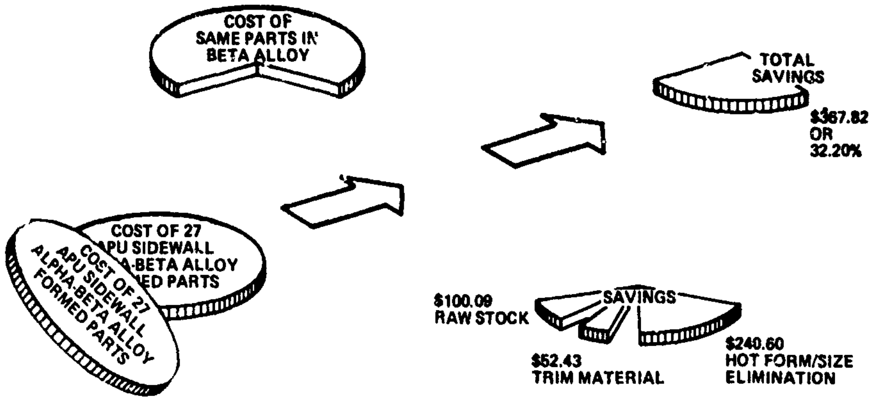

FIGURE 46. Cost Payoffs from Application of Beta Titanium Sheet (Ref. 32)

Titanium consumption in the U.S. has fluctuated considerably through the years as shown in Fig. 47. In the early days DoD consumed most of the Ti production, therefore, the consumption rate was dependent on military aircraft production. The early peak production in 1957 was greatly reduced when the emphasis was changed from manned aircraft to missiles. The production has steadily increased to a new peak of 32 million pounds in 1969 only to be drastically reduced by the cancellation of the SST supersonic transport. Civilian aircraft use and a diversification to other civilian uses has caused the consumption rate to increase again in the 1970s.

The percent market share of mill product used in the U.S. up to 1973 is shown in Table 26 (Ref. 33). A marked increase in civilian use of titanium is apparent in the 1970s. Titanium's corrosive resistance makes it desirable in food processing, desalination plants, heat exchanger applications, medical surgery, and recently in sea based oil rigs. Table 27 (Ref. 33) gives many examples of civilian uses of titanium and its alloys. A trend toward an increase in titanium production is expected by industry in the future. "We look to an increased market in the next 5 years of about 40-60% over the 1974 market" (Ref. 34).

Estimates of DoD R&D funding used in titanium development is in the order of $150 million.

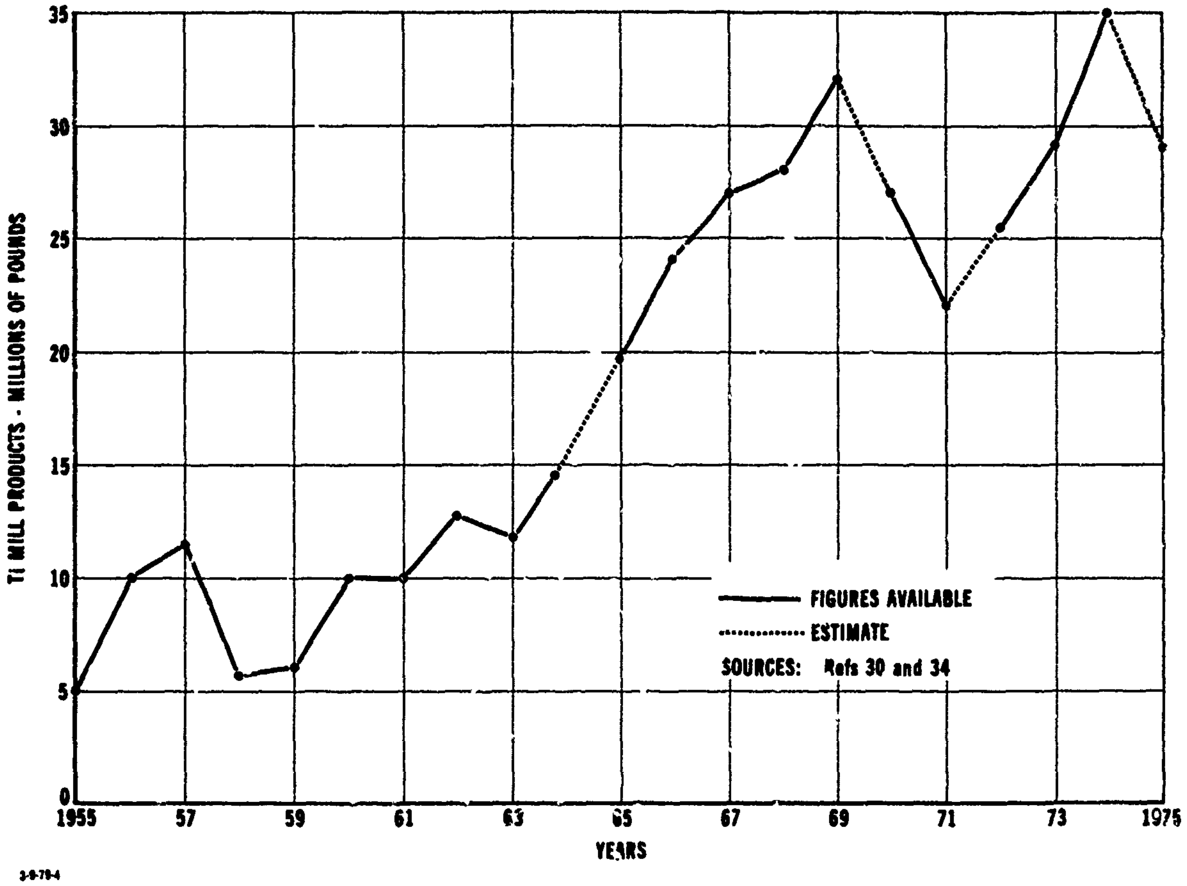

FIGURE 47. Titanium Consumption

TABLE 26. TITANIUM APPLICATIONS IN UNITED STATES
(% market share of mill product sales) (Ref. 33)

	1955	1959	1960	1961	1962	1963	1971	1973
Military aircraft-engines	63	46	38	39	35	31	21	15
-airframe	33	29	30	26	25	23	10	21
Missiles and space	1	15	17	16	25	31	7	7
Civil aircraft -engines	--	4	8	10	7	6	31	23
-airframe	3	4	5	4	3	3	15	17
Chemical and general engineering	--	2	2	5	5	6	16	18

TABLE 27. APPLICATIONS OF IMI* TITANIUM IN CHEMICAL AND GENERAL ENGINEERING (Ref. 33)

Industry	Environment	Alloy used**	Types of plant	Reason for using titanium
Chemical	Organic acids; chlorides/bromides; chlorine, chlorine compounds; chlorinated solvents; nitric acid; chromic acid; fertilizers—e g. urea, ammonium nitrate etc; sulphuric, hydrochloric and phosphoric acids	CP Ti IMI 260 IMI 261	Loose lined, solid and clad plate vessels; tubular and plate type heat exchangers; mixers; pumps; valves and pipework; dimensionally stable anodes for chlorine production.	Excellent corrosion resistance in a wide variety of oxidizing and neutral solutions, particularly in chloride environments. Good corrosion resistance in non-oxidizing solutions in the presence of inhibitors, with anodic protection or as IMI 260 or IMI 261. Unique electrochemical properties for anode use.
Paper Pulp and Textile Bleaching	Chlorine dioxide; sodium hypochlorite	CP Ti	Tanks; J-boxes; heat exchangers; heating elements; rollers.	Good corrosion resistance in chlorine containing compounds such as chlorine dioxide and sodium hypochlorite.
Metal Finishing	Nickel, chromium, copper plating solutions; sulphuric acid anodising solution.	CP Ti	Tanks; heat exchangers; heating coils; immersion heaters; anode baskets; anodising jigs; anodes.	Good corrosion resistance in most metal finishing solutions. Unique electrochemical properties as anode basket, jig or anode material.
Metal Refining	Various, mainly sulphuric acid based solutions.	CP Ti	Heat exchangers; coils; starter blanks; cathodes;	Good corrosion resistance. non stick properties; electrochemical properties.
Power Generation	Seawater, brackish and polluted estuarine waters.	CP Ti	Steam condensers; ancillary heat exchangers.	Good corrosion resistance to seawater and other polluted, naturally occurring waters.
Desalination	Seawater and various sodium chloride brines.	CP Ti	Heat exchangers for multi-stage flash distillation units.	Good corrosion resistance to seawater and sodium chloride brines.
Steel	Hydrochloric acid and ferric chloride; hydrochloric acid gas.	CP Ti IMI 260	Various plant items.	Good corrosion resistance.
Nuclear	Nitric acid.	CP Ti	Vessels for nuclear fuel reprocessing.	Guaranteed good corrosion resistance in nitric acid
Surgery	Body fluids.	CP Ti IMI 318	Prostheses; joint replacements; bone plates; screws; pins, etc.	Good corrosion resistance and compatibility with body fluids.
Marine	Seawater.	CP Ti IMI 318	Tubular and plate type heat exchangers; scrubbers; sonar yacht fittings, etc.	Good corrosion resistance. High specific strength.
Oil Production and Refining	Seawater; brackish water; sulphide containing hydrocarbons.	CP Ti	Tubular and plate type heat exchangers.	Good corrosion resistance to seawater and to hydrocarbons.
Engineering	Various	CP Ti Ti alloys	Various.	Good specific strength.

*Imperial Metal Industries

**CP = Commercially Pure

IMI 260 = 0.15 palladium alloy
IMI 261 = modification of IMI 260
IMI 318 = 6 Al, 4V alloy

F. HIGH-YIELD STRENGTH STEELS FOR NAVY USE

In the late 1940s, the steel in general use in ship construction was a high-tensile steel (HTS) that had a yield strength of 47,000 psi. The Navy planned to develop a higher performance submarine which required a high performance steel. The following excerpts are taken from Ref. 35.

"To make a new steel worthwhile in terms of improved military characteristics, its yield strength should be significantly higher than the steel then in general use, and should also be consistent with the attainment of good ductility, toughness, and resistance to low-cycle fatigue. A yield strength of about 80,000 psi was desired. NAVSEA's (BuShips) efforts to modify the chemistry of the HTS steel met with little success."

"The modified versions of HTS, which were characterized by modest amounts of alloying elements, produced only small increases in strength and toughness. This result persisted in both the as-rolled and normalized conditions. Although this series was a failure, it demonstrated clearly that a different approach was needed. The calendar was figuratively turned back more than half a century to the excellent Krupp quenched and tempered armor steel originally produced in 1884. This Krupp product was characterized by modest use of nickel (Ni), 3.5%, and chromium (Cr), 1.5%, and possessed excellent toughness. This basic Krupp steel, with modifications in the carbon and nickel content and with the addition of molybdenum (Mo), appeared to have the most attractive combination of all desired properties. This modified Krupp steel, originally known as low-carbon Special Treatment Steel (STS), was the forerunner of that which is now known as HY-80 steel."

"Unlike HTS, which possesses its strength in the as-rolled or milled condition and has its toughness enhanced by normalizing, HY-80 is a low-carbon alloy steel which acquires strength and toughness through quenching and tempering. HY-80 is, therefore, a hardenable steel, the properties of which are achieved by heat treatment. Its composition must be balanced to give the desired strength, toughness, and hardenability."

"HY-80 steel is made by either open hearth or the electric furnace process. The resulting steel is fully killed and fine grained. Plates are rolled directly from either ingots or slabs. To achieve the desired strength and toughness, a final heat treatment of quenching and tempering is required. This is the step in the manufacture of HY-80 which is distinctly different from the usual process of making structural steel."

Forming and welding problems could have been expected when HY-80 steel was brought out of the "laboratory."

"It was obvious, even before HY-80 steel became available in production quantities, that a steel with a yield strength much in excess of the steel then in general use would pose fabrication problems. The first, and most obvious, such problem was that involving forming equipment. Since forming involves stressing the materials in excess of its yield strength to obtain a permanent change of shape, the forces required to be exerted by the forming equipment are, for constant geometry, directly proportional to yield strengths. But the shipyards in the United States possessed, for the most part, rolls and presses that had been in constant use throughout World War II when the common submarine hull materials were medium- and high-tensile steels (yield strengths of 33,000 and 47,000 psi, respectively). Now these same shipyards were about to be called upon to form steel with a yield strength of about twice that previously used--and with the same equipment. This prospective burden was patently too great for existing equipment. Hence retooling was necessary."

"A second, and almost as obvious prospective difficulty in fabrication was that of weldability. From antiquity, when welding was confined to hammer and forge methods, the introduction of a 'new' material has been accompanied by weldability problems. The usual manifestation of the problem has been the appearance of fissures in the heat-affected zone of the base material adjacent to the welds--in the vernacular, cold cracking. As has been ably and clearly stated by Griffin (Ref. 36), such conditions prevailed in the fore-welding era, persisted in both bare-wire and covered-electrode welding, and stalked rampant through our submarine building yards of World War II. It would be extremely fortuitous, then, if the introduction of a new material in mid-Twentieth Century was not accompanied by similar troubles." (Ref. 35)

Reference 37 provides the following excerpts.

"In 1952, HY-80 was approved for use in the side protection system in the aircraft carrier USS FORRESTAL and other similar applications. No problems were encountered in these applications. Based on the impressive characteristics of HY-80 and encouraging test results, it was approved for use in submarine construction in 1955. The USS SKIPJACK was the first combatant submarine with structure subjected to submergence pressure specified at the outset to be of HY-80 steel. Later, extensive cracking was discovered in another submarine in an earlier stage of construction. It was subsequently found that weld cracking was also occurring in the SKIPJACK."

"Very few defects were discovered and those were minor. SKIPJACK was considered structurally sound. Nevertheless, inquiry into the difficulties encountered on the other submarine was begun. During the early stages of this inquiry, cracks were found in the connections of frame webs to shell. When this problem was probed further, it was discovered that cracking persisted in the repair welds. In a few cases as many as six cycles of welding and inspection were required before successful repair was achieved. Furthermore, welds previously inspected and found satisfactory were, on reinspection as much as three weeks later, found to have severe cracks. Sleuthing pointed to reheating to make a weld in the vicinity as the culprit. Still later, boundaries of 'hard' tanks were reexamined and found to have cracks although previous inspections, some as much as four months earlier, had resulted in acceptance. These defects were attributed to high restraint. All the forecastable difficulties were appearing belatedly."

"The inquiry was broadened to include all submarine building yards and the cognizant Supervisors of Shipbuilding. As might have been expected, each 'expert' interviewed espoused a different factor as contributing to the problem and each proposed a different remedy. The consensus was, however, that compliance with BuShips Notice 9110 of 2 July 1958 would be a 'big step toward eliminating the problem.' The guidance contained in this BuShips Notice 9110 included controls for:

1. Preparation, storage, and issue of electrodes
2. Preheat and interpass temperature
3. Heat input
4. Welding sequence."

"In addition, from all the contributing factors cited and the remedies proposed during the inquiry, came the realization of the need for:

1. Protection from the weather
2. Avoiding highly restrained construction
3. Standardization of inspection procedures and records
4. Better training and qualifications of welders and inspectors."

"In summary, all concerned with submarine construction became convinced of the necessity of close control of the entire fabrication process."

Submarine building had come practically to a standstill (Ref. 38). Intensive studies were initiated to better understand the controls required to successfully weld high performance steel in the shipyard environment.

"The total fabrication process was attacked on a broad front. Base material specifications were improved and made more definitive, and electrode development was intensified. Nondestructive inspection techniques were improved and standardized; inspectors were carefully trained in their conduct and interpretation. Extruded and rolled shapes, castings, and forgings of HY-80 steel were produced, qualified, and authorized for use. Construction details were redesigned to avoid high restraint and stress concentrations. Finally, all of these improvements were combined with the required controls into a single document, NAVSHIPS 250-637-3 of November 1960, which became the gospel for fabrication, welding, and inspection of HY-80 steel in submarine construction. Later revisions to this publication have removed ambiguity, closed loop holes, improved clarity, and incorporated results of research and development." (Ref. 37)

"The effectiveness of this concerted effort is graphically presented in Fig. 48 where the decline in the incidence of weld defects is related to time. The significant event--issue of BuShips Notice 9110 in July 1958, the inquiry into construction practices of 1958-9, and the publication of NAVSHIPS 250-637-3 in November 1960--have been indicated in Fig. 48. The most encouraging finding

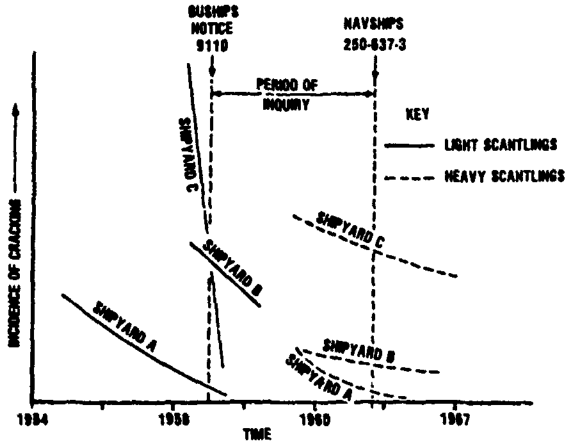

FIGURE 48. Incidence of Weld Defects Discovered During Submarine Construction (Ref. 37)

shown in Fig. 48 is that the incidence of weld defects at every submarine building yard declined and this continued even when heavier scantlings were incorporated in later classes. The most annoying finding shown in Fig. 48 is that a structure entirely free from flaws has not yet been achieved. Clearly this was never really expected although it was devoutly to be desired. Certainly the very low incidence of weld flaws at the present time time [1970] is ample evidence that submarine construction can be and is 'under control.' Equally obvious is that careful attention to details, vigorous inspection, and constant vigilance are required." (Ref. 37)

Research funds (equivalent to current 6.1, 6.2, 6.3A monies) in the amount of approximately $2 million were spent in the development of HY-80 steel. This product has made the building of present day high performance submarines possible. The work was done by teams of Navy Laboratory personnel working in close cooperation with shipyard personnel. The program was funded and coordinated by BuShips (NAVSEA).

HY-100 was developed as a follow-on to HY-80. Its chemistry is essentially the same as HY-80 but its heat treatment causes it to attain higher strength. HY-100 has been used for several years in surface ship applications and is now being considered as a replacement for HY-80 in submarine pressure hull applications (Ref. 39).

Work on higher strength steels was started in 1962. Work on an HY-180 steel was stopped because of serious fabrication problems and stress corrosion cracking tendencies. Development of HY-130 steel has been successful. "The major known technical risk of the HY-130 program involves stress corrosion crack propagation in the weldment areas. This refers to the growth of existing cracks under the combined influence of salt water and local high residual tensile stress fields. In practice, the residual stresses will usually vary from tension to compression both along the surface and through the thickness, so that stress corrosion cracking is very unlikely to be catastrophic. In addition, the HY-130 weld area has a good fracture toughness, which further resists long-range crack propagation." (Ref. 40). However, steps are being taken both to minimize flaws and to reduce residual stresses in HY-130.

From a material and supply standpoint, there are available qualified producers of plate forging, extrusions, and castings in both HY-100 and HY-130. HY-100 is considered similar enough to HY-80 so that application to submarine use would be done without extensive hardware evaluation (Ref. 39). The HY-130 material is chemically much different from HY-80 and HY-100, and a test vehicle is planned to fully evaluate the materials in the intended environment before it is considered for submarine pressure hull application.

G. ALUMINUM ALLOYS

1. Background

Looking back through the years, it is evident that DoD-sponsored research was at the forefront in aluminum alloy technology development. The critical needs of the Services dictated the direction of research and development in this area. The surface Navy of the late 1940s required corrosive-resistant lightweight metal that could be satisfied at that time only by a new aluminum alloy. In the 1960s, Service aircraft were experiencing difficulties with stress corrosion in high strength aluminum that caused loss of aircraft. NAVAIR and AFML efforts to correct this problem ranged from basic research studies to manufacturing technology development. Brief case histories on the development of the 5000 series aluminum and the 7050 aluminum alloy illustrate typical DoD R&D effort on aluminum.

2. 5000 Series Aluminum

Development of 5000 series aluminum (Ref. 41) was started about 1948 by BuShips (NAVSEA). There was a need for a lighter weight material, resistant to sea environment, which could be used in superstructure construction. Radar and other equipment were being attached to the ship's superstructure and were making the ship top-heavy. The available aluminum alloys in the 1940s had poor weldability, poor corrosion resistance, and poor pitting resistance. BuShips initiated R&D programs to develop an aluminum alloy to satisfy the Navy needs. This work eventually produced the currently available 5000 series aluminum.

BuShips directed the aluminum research program, receiving excellent cooperation from aluminum companies in the fabrication of small test specimens for little money. The specimens were passed on to the Navy laboratories, supported by BuShips, which tested and evaluated the product. In this way, many experimental approaches could be assessed in a short period of time for a relatively small amount of money. A series of alloys was developed that met the Navy requirements. The successful alloys were evolved through logical metallurgical formulation development and did not involve a "breakthrough." The 5000 series alloys are now used in substantial quantities aboard all Navy surface ships.

The funding required in the development of the 5000 series aluminum alloys has been lost in history, but the amount was very small by today's standards.

3. 7050 Aluminum Alloys (Ref. 42)

The Fleet, from 1964 to 1966, reported extensive trouble with stress corrosion in high strength aluminum. Such problems has caused loss of aircraft and loss of millions of dollars of unsatisfactory equipment. The extent of the problem prompted NAVAIR to turn their R&D efforts toward solution of the problem in 1966. Close contact was maintained with concerned companies such as Alcoa, North American, Boeing, etc., and with countries such as Australia, the U.K., and Italy, who were actively working on this problem.

NAVAIR sponsored work (6.1) on the microstructure of various Al alloys in order to understand the basic mechanism of stress corrosion cracking. The failure mode was found to be related to the microstructure. Studies showed that certain heat treatments made the existing alloys resistant to exfoliation corrosion and stress corrosion cracking but with about 10% loss in strength. Further studies were conducted to restore the strength. To do this, the chromium was replaced with zirconium, and the level of iron and silicon was lowered. This work resulted in the development of the 7050 aluminum alloy in 1971. Much of this work was done under contract with Alcoa.

A data base on 7050 was developed from 1971 to 1975 in Navy laboratories and under the Air Force's Manufacturing Technology (MT) program. The Navy had no MT program at that time and the Air Force was cooperative in developing thick extrusion technology for 7050 under their MT program.

The introduction of 7050 into aircraft use started in 1974. It is now used in all new Service and commercial aircraft and is proposed for use in cruise missiles.

During the 6.1 work, NAVAIR spent about $30K per year (1966-1968). The 6.2 work cost about $50K per year (1968-1971). During scale-up, $100K per year was spent (1971-1974). The total expenditure for the 7050 development was about $500-600K.

4. New Trends

In the 1970s, aluminum alloys have been facing intense challenges for military applications from titanium, Kevlar epoxy, graphite epoxy, metal matrix composites, etc. Advances in superplastic forming and other fabrication techniques are making more materials viable options for the designers. Also, efforts to supply materials to near net shape make higher cost materials more competitive to low-cost materials like aluminum. The Air Force, Army, and Navy have been supporting powder metallurgy R&D since the concept was discovered about 15 years ago (Ref. 42). Recent advances in powder metallurgy and other manufacturing techniques have caused aluminum manufacturers and those tooled-up to use aluminum (e.g., the aircraft industry) to "fight back" with the new fabrication technologies and increase the utilization of aluminum.

Reference 32 discusses new advances in aluminum and excerpts are included in this section. "Figure 49 highlights aluminum applications in the L-1011 as a benchmark for consideration of anticipated aluminum improvements. Note that the fuselage skins are the classic pure clad 2024-T3, that 7075-T73 and -T76 are used in the more aggressively corrosive exposures, that the weight effectiveness of 7075-T6 was reserved for the more benign environmental situations, and that 7008 high strength clad 7075-T76 wing skins afforded both weight and corrosion payoffs."

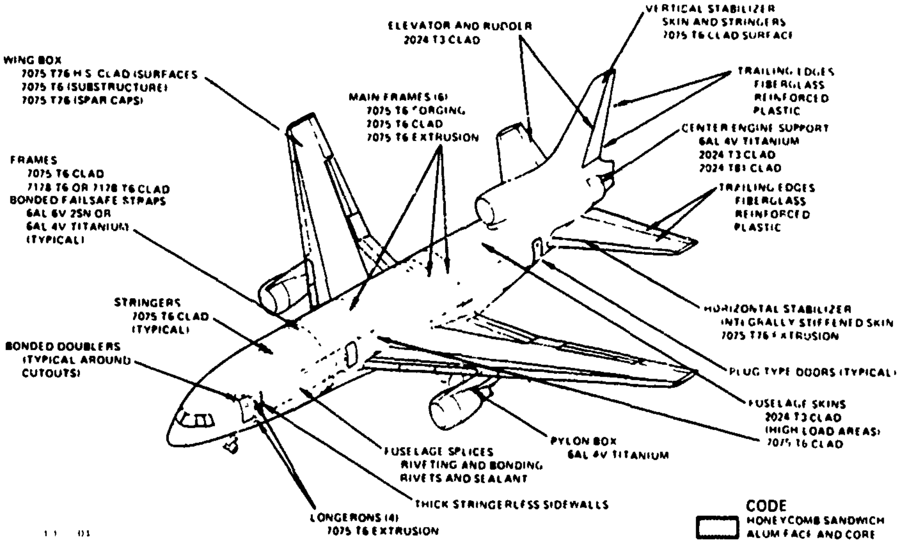

FIGURE 49. L-1011 Basic Materials and Design Features (Ref. 32)

TABLE 28. PROJECTED PAYOFFS FOR ADVANCED ALUMINUM ALLOYS IN TRANSPORT APPLICATIONS (Ref. 32)

STRUCTURE	ADVANCED I.L.O. CURRENT ALUMINUM ALLOY WEIGHT SAVED %	WEIGHT SAVED #	FUEL SAVED GAL	FUEL SAVED $
WING	13.3	6,310	225,000	90,000
FUSELAGE	11.2	5,677	202,000	80,800
EMPENNAGE	13.2	1,197	42,000	16,800
SHIP SET TOTALS	12.2	13,184	469,000	187,600

"Table 28 presents a strong case for the potentially attainable improved aluminum alloys in the future. The weight saved and the attendant annual fuel payoffs available to a typical commercial carrier by a redesign of the L-1011 wing, fuselage and empennage using 20 percent stronger, 20 percent stiffer, and 10 percent lighter advanced aluminum materials are impressive. Another advantage of using improved aluminum alloys is the ease of incorporation into the production process as opposed to the difficulties of a composite material. Similarly, the advantages to the operators in terms of inspection and repair techniques are in favor of improved aluminum alloys."

"Figure 50 shows the strength and corrosion resistance trends in high-strength 7000 series and powder alloy research and development leading up to 1990. Note that the intergranular/exfoliation/stress corrosion vulnerabilities of 7178-T6 and 7075-T6 were avoided through special aging (-T76) and that the 7075-T7X is a stride on the road to combined high strength and corrosion resistance predicted for the powder metallurgy alloy MA-87 now under development by the aluminum suppliers."

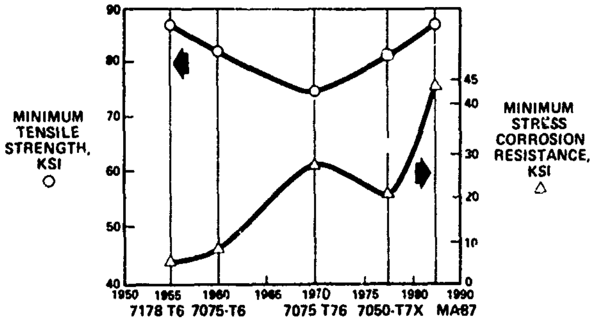

FIGURE 50. Trends in 7XXX and P/M Alloy R&D (Ref. 32)

"Figure 51 compares the strength and fatigue life of the 2024 alloy prepared by cast ingot versus controlled solidification powder densification technology. Note that controlled

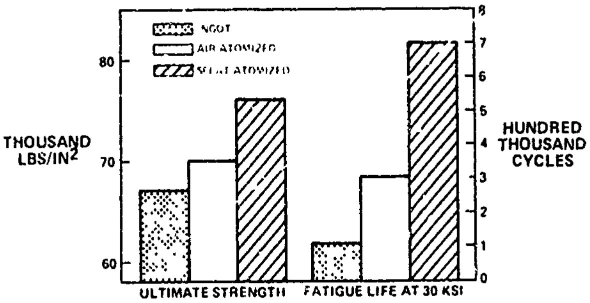

FIGURE 51. 2024-T4 P/M Payoffs (Ref. 32)

solidification involving powder produced by impingement (splat) of molten aluminum on a moving chilled surface provides a much higher cooling rate, a much finer and more homogeneous grain structure and property gains beyond those provided by impingement of molten and gaseous jets (air atomization). Controlled solidification avoids undesirable separations and precipitants inherent to ingot technology and provides an entree for uniform co-mingling of finely divided constituents of such low solubility as to defy melt alloying. Such dispersion compounding is responsible for experimental aluminum materials which in early evaluations promise to retain their yield strengths indefinitely at 500 degrees Fahrenheit. The potential for application to vehicles having cruise speeds well above Mach 2 is very attractive."

"Figure 52 shows the modulus payoff attainable from lithium as an aluminum alloy constituent. The Al-Mg-Li alloys are an evolving system which promise high mechanical properties, good corrosion resistance, and a unique high modulus/low density combination."

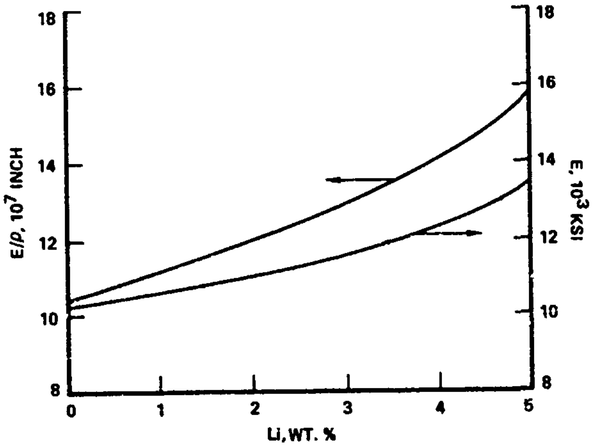

FIGURE 52. Aluminum/Lithium Stiffners Payoff (Ref. 32)

"Table 29 summarizes properties for candidate Al-Li, powder metallurgy and the classic 7075-T6 materials and reflects the substantial progress being made toward the lighter, stiffer, stronger, more durable aluminum materials of the future. Production availability of these materials is projected for the early to mid-eighties."

TABLE 29. PROPERTY COMPARISON (Ref. 32)

PROPERTY	7075-76	MA-87-T75	ALUM-Li
TENSION ULTIMATE	75	87 (15%)	75
COMPRESSION YIELD	65	80 (23%)	65
COMPRESSIVE MOD	10.7	10.7	12 (12%)
FATIGUE CUTOFF	45	55 (22%)	
KIC	30	32 (6%)	
SCC	25	25	35
DENSITY	.101	.102	.090 (11%)

H. NAVY R&D EFFORT ON MOLYBDENUM AND OTHER REFRACTORY METALS

After World War II, the materials experts at the Office of Naval Research (ONR) and the Naval Bureau of Weapons believed that there would be a service need for metals which would function well at elevated temperatures. The gas turbine was the case in point that provided motivation but the extrapolation to propulsion systems and vehicles, not in development at the time, provided inspiration (Ref. 2). The Office of Naval Research supported research programs on molybdenum and its alloys from 1948 to 1956. These programs stimulated additional development effort on other refractory metals. NAVAIR supported refractory metal R&D until 1965, seeking turbine blades to withstand operating temperatures to 2600 °F (Ref 43). Research was done on molybdenum, tungsten, tantalum, and columbium alloys. The oxidation problem has prevented the use of these materials in many applications.

The following paragraphs are mainly excerpts from the Institute for Defense Analyses (IDA) report (Ref. 2) that discusses the early work on molybdenum and other refractory metals.

"The year 1947 appears to mark the beginning of the present story. I.R. Kramer, then of ONR, in collaboration with N.E. Promisel, then of the Bureau of Aeronautics, F. Perella of the Bureau of Ordnance, and personnel from Bureau of Ships, began to plan a research and development program aimed at developing ductile metallic materials for service at approximately 2000 °F."

"Background activities may be typified as follows. In 1945, B.S. Old of the Office of Research and Invention initiated a program on heat-resistant materials aimed at extending and improving technology for aircraft gas turbines that had been imported from the U.K. during the war. Still further back lie the science and technology of molybdenum and tungsten that emerged in 1910 with the incandescent lamp industry and the development of ductile tungsten and molybdenum by Coolidge and Fink, respectively. In parallel, ONR also sponsored the study of intermetallic boron compounds at the American Electro Metals Corporation and the study of selected ceramics at Battelle Memorial Institute."

"It was clear that the flow and creep strength of existing nickel and cobalt alloys were inadequate for structures to be used in the temperature range of 1800-2000 °F. Similarly, experience showed that the ductility of the intermetallic compounds and ceramics was very likely inadequate to avoid brittle failure under loads and combustion chambers. Data showed that the strength of chromium, molybdenum, tungsten, rhenium, tantalum, columbium (niobium), and vanadium would be adequate for many uses above 1800 °F (or might be made so by alloying). In contrast to Ni, Co, and Cr, adequate supplies of molybdenum ores were then known to be available in the USA. In doubt, however, were properties and items of behavior connected with the ductility of chromium, molybdenum, and tungsten; relations defining limiting temperature and time of service, the oxidation resistance of all of these elements except chromium; and the availability and fabrication of most of them. Some of the relationships between composition, structure, properties, and behavior of these metals and alloys were known but they provided an inadequate basis for predicting behavior, use, design data, or conditions for processing and fabricating. This statement is true, notwithstanding the rather extensive experience in making and using tungsten and molybdenum lamp filaments, heating elements, electron tube parts, and scientific equipment. In fact, a legitimate criticism of metallurgy, particularly in those days, concerns the very limited range over which the knowledge and techniques of as sophisticated a technology as filament making could be extrapolated."

"The first steps taken by the program planners included a review of the knowledge concerning Mo, W, Cr, Ta, Cb, V, and their alloys. They made contracts with individuals, laboratories, and industries having pertinent knowledge and experience. During World War II, molybdenum had been found to be an excellent liner material. Pressing and sintering molybdenum powder in hydrogen, the primary means of fabrication at the time, was found to be poorly adapted for producing the required sizes and shapes. In 1943, R.M. Parke, working under an OSRD contract with the Climax Molybdenum Company, recommended and tried vacuum arc melting. The resulting ingots were too brittle for further hot working and were found to fracture along grain boundaries. Individual single crystals, on the other hand, appeared to be reasonably ductile. A fractographic analysis by J.L. Ham showed that oxide particles at grain boundaries led to fracture. Collaboration between the two workers led to the solution of this difficult problem. They added carbon to reduce molybdenum oxide quantitatively and eventually found source material of low oxygen content which, together with the proper carbon additions, yielded reasonably ductile cast material that could be extruded at about 2400 °F using molten glass as a protective lubricant (the Ulgine-SeJournet process)."

"The program began to take shape in late 1947. A key individual was R.M. Parke, who had moved to the Battelle Memorial

Institute. Key facilities were those developed at the Climax Molybdenum Company in the intervening years since 1943. Programs emphasizing powder metallurgy and elimination of impurities were already under way in other industries. Accordingly, Kramer and Harwood arranged a contract with Climax, starting October 1, 1948, with A.J. Herzig, the principal investigator. The objective of the contract was stated in ONR report as follows:

> 'The purpose of this project is to prepare experimental melts of promising alloy compositions; explore physical and mechnical properties, including hot and cold working properties, and develop work on fabrication processes and heat treatment of significant alloy compositions. The alloys are to be prepared by Arc-Casting processes.'

It is interesting to note the deliberate lack of specificity. Under another contract starting April 15, 1949, R.M. Parke undertook to direct a study described in another ONR report in the following way:

> 'The purposes of this project are: (1) to determine the causes of certain unfavorable characteristics of molybdenum and to develop molybdenum of improved characteristics; and to determine the high temperature properties of molybdenum and molybdenum-base alloys. (2) To develop molybdenum-base alloys with alloys with elevated temperatures greater than that of molybdenum and with satisfactory ductility and resistance to oxidation;' (investigations of a number of elastic, plastic, crystallographic, and chemical properties were also anticipated.)
>
> 'A Molybdenum Advisory Committee* will be established to help guide the work and to advise ONR on the course of future action to be taken with regard to the development of molybdenum for high-temperature applications. The Battelle Memorial Institute will also coordinate for ONR all of the work being done on molybdenum and its alloys applicable for high temperatures.'

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*Later to become the Materials Advisory Board.

Again, it is interesting to note the wedding of science and technology. The advisory committee was composed primarily of representatives from laboratories working in the field and willing to collaborate with the Navy program. Correlative programs, one on extrusion at Babcock and Wilcox Tubular Steel Products Division and one on isostatic pressing of powder at Westinghouse, proved equally important."

"The committee began in July 1949 to define problems and review progress toward answers to the questions essential to the production and use of molybdenum as a structural material at 1800-2000 °F. Two questions deserve special attention. One concerns ductility (a) the nature of flow and fracture in individual molybdenum crystals and (b) the way in which interstitial and substitutional solutes, grain size, and other variables affect the transition from ductile to brittle behavior common to most body-centered-cubic metals. A second concerns the physical and chemical properties of oxide layers on the surface of molybdenum and its alloys and, in particular, the question as to whether alloying constituents would lead to the formation of protective oxide films, in the desired temperature range, in analogy with stainless steels. The need for research workers interested and able to provide answers to these questions led to placing contracts, respectively, with Maddin at Johns Hopkins, Speiser at Ohio State University during 1950. Question (b) above was answered slowly and partially by a number of investigators. The initiation of this effort brought the program to an approximately steady level that was maintained for a few years. Using 1951 as a sample, this level appears to be around 10 or 11 full-time equivalent research workers with normal supporting help and facilities, with about 40% of the effort in industry, 46% in a not-for-profit laboratory, Battelle Memorial, and the balance in university laboratories. The total effort on heat-resistant materials was approximately the same. At this time, slightly over 30 projects in nearly as many universities were in progress which studied a variety of phenomena occurring in the production and use of materials. This total university effort was perhaps 90% as large as the total effort on high-temperature or heat-resistant materials."

"Funds came from several parts of the Navy, the Air Force, and the Army, and were provided by program managers on their own authority out of their budgets. More funds undoubtedly would have led to more rapid progress."

"As already mentioned, individuals responsible for materials research and development in the several branches of the Services collaborated and agreed on the objectives of the program. Within these objectives, the ONR program monitors and the committee already mentioned were able to use results as they were produced to determine the evolution of the program."

"The advisory committee was very important in analyzing and correlating results, helping principal investigators plan ways of solving problems, and, where necessary, explicitly deciding the course of the work. The committee and program monitors provided a means of coupling knowledge, capabilities, and requirements. Contributions from programs not supported by ONR were coordinated through the committee. All of the groups were kept informed through presentations and written reports."

"Because there was reasonable flexibility in the use of funds by the program monitors, and in the day-to-day decisions of the researchers, the program evolved according to its technological needs and opportunities."

"The program on molybdenum and its alloys began to taper off and to be replaced by new objectives after about 1955. The symposium sponsored by the Office of Naval Research and the resulting book, show the state of molybdenum technology in 1956 fairly. These papers, however, only partially show how individuals asked and answered questions that brought the subject to a much improved state."

"The motivation of the industrial groups towards evolving products or product lines was a key factor in developing techniques and facilities for consolidating and working molybdenum and molybdenum alloy shapes. In particular, vacuum arc melting, purification and alloying techniques and facilities developed at the Climax Molybdenum Company introduced a new and essential capability. The availability of ingots that could be extruded and then further worked, the interests and needs signified by ONR support, together with the knowledge of the need and methods for control of contamination led to the creation of facilities for fabricating molybdenum under the protection of an inert atmosphere such as the In-Fab Facility at the Universal-Cyclops Steel Corporation."

"In a parallel development, researchers at Westinghouse showed that isostatic pressing of powders provides a partially complementary and possibly more effective technique for preparing fairly large bodies of molybdenum and its alloys that may be rolled and worked further. Many other interrelated improvements in technique and knowledge took place."

"Research directed by Maddin placed the behavior of molybdenum single crystals in the context of knowledge concerning body-centered-cubic metals and extended knowledge concerning crystal plasticity. These results greatly increased the confidence of the committee and program monitors in the direction of their work and helped define conditions and limits for practical processing. Here was an excellent example of a scientific project assisting a complementary technological project. In addition, the work reported by Bechtold and by Spacil and Wulff on the effects of oxygen, nitrogen, and carbon on the ductility of wrought molybdenum further defined the role of interstitial solutes and the limits of their concentration in practice. It may be stated that some persons* directly engaged in development relied less on these clarifications than did program managers."

"Similarly, the thermo-chemical studies directed by Speiser defined the problem of oxidation and the main limitations on protective coatings. This work permitted the conclusion that no oxide coating based on the oxidation of a molybdenum base alloy could be protective in the temperature ranges of interest and led to the important decision that protective coatings such as molybdenum disilicide would have to be overlaid."

"Most of the experimental techniques and preparation and fabrication processes developed for molybdenum have been adapted to other members of the refractory metals family with considerable enrichment of both science and technology. The advent of electron-beam heating in very high vacuum in the late 1950s, stimulated by the development of electron-beam welding at the Centre d'etude del'Energie Atomique in France, taken together with the technique of zone refining developed in the semi-conductor science and technology, has further assisted the study of refractory metals. This application in turn has further stimulated and increased the power of the technique itself."

"The first large-scale application of molybdenum technology following on the heels of this program was in the manufacture of jetavators for POLARIS missiles. From the Navy's point of view, it was fortunate that the molybdenum program did not wait for this special need to develop. Many other applications have followed this one. Obviously, no single index measures the value of such a development adequately, except, perhaps, new capability."

"About 1960, a refractory metals sheet rolling program was set up under the Materials Advisory Board. This program was

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*An understandable and resolvable difference of opinion attended this matter. Those workers emphasizing development believed that research merely confirmed what they had already demonstrated and research was thus of secondary value. Managers believed that it was necessary to continue to invest in research. Appeal to persons with less conflict of interest (Advisory Committees) usually resolved this question in favor of further research).

coordinated among the Department of Defense, the National Aeronautics and Space Administration, and the Atomic Energy Commission and showed that the fabrication, joining and coating of sheet material from refractory metal alloys are now largely on a commercial basis. The MAB panel for this study also mentions that more effort by trained researchers earlier in the program to study the relationship between solutes, dispersed phases, structure and processing variables, would have produced additional knowledge that could have been factored into the program. Apparently, this effort did encounter some inflexibility due to the procurement practices and management of money by the various agencies involved."

The Services could find broader use of refractory metals if they could satisfactorily solve the oxidation problem. Efforts to do this have generally failed. Protective coating is normally unsatisfactory in the environments in which the refractory materials are required.

The authors did not have the time to investigate Army and Air Force programs in refractory metals.

I. MISCELLANEOUS INFORMATION ON MATERIALS AND SYSTEMS

This section contains useful but brief or incomplete information on materials and systems of interest. Lack of time and source material prevented a more complete account of these programs. Some of the information is included for its historical value. Many of the programs show successful completion and application of the product; other programs were terminated without solving enduring technical problems or finding application.

1. MINUTEMAN Missiles

Tables 30 and 31 give information regarding the materials used on MINUTEMAN motors and reentry vehicles (RVs). Some information on components on early missiles was not readily obtainable. Material changes on the MINUTEMAN missiles were not

TABLE 30. MINUTEMAN--MOTOR CASE AND NOZZLE MATERIALS
(Courtesy of S.L. Channon, Aerospace Corporation)

		STAGE 1	STAGE 2	STAGE 3
CASE	MINUTEMAN I	D6AC (Thiokol) 220 ksi	Ti-6Al-4V (Aerojet) 160-180 ksi	S-994 Glass/ERLA 2256 Epoxy (Hercules)
	MINUTEMAN II	220 ksi	Same as MMI	Same as MMI (Hercules)
	MINUTEMAN III	220 ksi	Same as MMI	Same as MMI (Aerojet, Thiokol)
NOZZLE	MINUTEMAN I	Blast Tube - ADHG Graphite	Blast Tube - Ti coated with MX2630A carbon phenolic	Blast Tube -
		Inlet -	Inlet - ATJ Graphite	Inlet - ATJ Graphite
		Throat - W	Throat - W in ATJ ring w. Mo sleeve	Throat - W in graphite ring
		Rings -	Rings -	---
		Exit Cone -	Exit Cone - MX2625 Graphite phenolic	Exit Cone - Chopped Graphite phenolic
	MINUTEMAN II	Blast Tube - Graphitite G over CP	Blast Tube -	---
		Inlet - Graphitite G	Inlet - Comp. molded MX2630 carbon phenolic	Inlet - Graphite phenolic
		Throat - W with G insulation	Throat - W in ATJ with Mo sleeve	Throat - W w. graphite ring
		Rings - 1 Graphitite G, 4 HLM85	Rings -	Rings - Graphite
		Exit Cone - Molded Chopped Graphite Phenolic FM5014	Exit Cone - Tape wrapped FM5014 Graphite phenolic w. silica phenolic extension	Exit Cone -
	MINUTEMAN III	Same as MMII	Same as MMII	Same except exit cone MX4926 carbon phenolic with MXC-113 carbon felt tape extension

as extensive as on the Navy ballistic missiles because the requirements did not change as drastically in the MINUTEMAN program (Ref. 44).

TABLE 31. SUMMARY OF MARK 11 AND MARK 12 RV MATERIALS
(Courtesy of S.L. Channon, Aerospace Corporation)

	Mark 11	Mark 12	Mark 12A
Nose Tip	Avco RAD 60 silica phenolic with removable windshield	Teflon over inverted dixie cup carbon phenolic 5055A	Carbon-Carbon (weave not decided)
Heatshield	OTWR silica phenolic	Carbon phenolic 5055A	Carbon phenolic 5055A (may be replaced by alternate fiber)
Antenna Window	Dynasil-quartz	Slip-cast forged silica	

2. Armor

Considerable DoD funding has been devoted to armor R&D through the years. Some interesting advances in the 1968 time frame are discussed in the following paragraphs (Ref. 45).

a. Ceramic Composite Armor (1968). A new concept in lightweight armor was developed that employs a ceramic layer to shatter small arms projectiles. The material permits armor weight reductions of more than 50% without sacrifice of protection. An entirely new national production capacity for making complex ceramic shapes and for fabricating composite armors has been established. In 1968, more than 400 tons of this armor were employed in Vietnam to protect troops and critical aircraft components.

The discovery of aluminum oxide composite armor by the Goodyear Aerospace Corporation in 1962 was recognized by AMMRC as a major breakthrough in lightweight armor technology. It was quickly followed by the development of improved ceramic composite armor materials, based on the ceramic properties, which AMMRC ordered from industry. In order of increasing efficiency, these included silicon carbide, beryllium oxide, and boron carbide for defeat of steel-cored, small arms, armor-penetrating projectiles. In 1967, AMMRC developed both titanium carbide and titanium diboride composite armor for defeat of tungsten carbide small arms projectiles.

The remarkable ability of ceramics to shatter armor-piercing bullets has resulted in body armor which, for the first time in history, will protect a soldier against bullets. Of even more significance, however, is the program that AMMRC undertook to exploit the development of these new armor materials for Army hardware. It consisted of three major phases: (1) briefing Army organizations that are concerned with hardware developments on the research progress and the significance and limitations of results, (2) presenting executive briefings to American industry to motivate the expenditure of corporate funding for production of the new armor materials, and (3) conducting armor symposia every 12 to 18 months to disseminate complete information on new materials to both the industrial and military communities. The effectiveness of this program in reducing lead time has been little short of remarkable, since new armor materials, such as boron carbide and dual-hardness steel, were employed in end items in Southeast Asia in less than 9 months after their discovery in the laboratory.

b. <u>Heat-Treatable Dual-Hardness Steel Armor (1968)</u>. A heat-treatable grade of dual-hardness steel armor has been developed with the cooperation of the steel industry. Laboratory techniques have had to be scaled up to production size, necessitating stringent production controls. The new armor is just being made available for military applications and weight savings of up to 50% over currently used homogeneous steel and aluminum armors will be possible.

In 1963, the ausformed grade of dual-hardness steel was developed by the Aeronutronic Division of the Ford Motor Company. This material had to be work hardened, which meant that it could not be formed, welding was difficult, and it could only be made in rather small pieces. AMMRC developed the concept of using low alloy steels of different composition for dual hardness such that the material could be softened, worked, welded, and then hardened by simple heat treatments.

AMMRC expedited commercial availability of the material with three major U.S. steel companies who are now able to supply the material in large tonnage quantities. Concurrently, a program is under way to make prototypes of various helicopter components, so that the armor will be an integral part of the component. A prototype scout vehicle is also being fabricated to establish manufacturing capability. This material should aid materially in the Army objective of air transportability and air dropability of combat vehicles since it will permit large weight reductions without loss of protection.

c. Aluminum Armor (1968). Under programs which started after World War II, the Metallurgy Research Laboratory of Frankford Arsenal investigated the ballistic properties of the available commercial aluminum alloys and promoted research to develop improved light alloys for armor as well as improved fabrication technology. This background knowledge proved to be valuable when the Army in the late 1950s explored new attack concepts which employed air-droppable, highly maneuverable vehicles. On the M113 family of vehicles, the use of an aluminum strain-hardened alloy (5083) resulted in a significant advantage over steel armor in resistance to shell fragments, which were regarded as the primary ballistic hazard. In addition, the extremely good weldability of this alloy led to economies in assembly that more than compensated for a slightly higher basic material cost. To date (1968), more than 10,000 M113 vehicles have been produced; this represents a consumption of aluminum approximating 100,000,000 lb.

During concept studies on a lightweight assault and reconnaissance vehicle, which evolved into the design of the General Sheridon, it was indicated that there was a critical need for increased resistance to armor piercing projectiles. Frankford Arsenal led an Industry-Army team effort which results in the development of a heat-treatable aluminum-zinc-magnesium, copper-free alloy (7039), which provided a 10 to 15 percent weight advantage over the strain-hardened alloy in AP resistance.

A key problem encountered was the vulnerability of the alloy, like other alloys of the 7000 family, to stress corrosion when surfaces with high residual stresses in the cross-fiber direction were exposed. Based on work in the Metallurgy Research Laboratory, this problem was brought under control by eliminating compositions and tempers that were highly susceptible, by educating designers to circumvent the deficiencies, and by applying remedial procedures when exposure is unavoidable.

The use of the 7039 alloy for the hull armor in the General Sheridan has successfully passed prototype evaluation and is now in limited production.

d. Alumina-GRP Composite Armor (1968). Prior to this development work, ceramics for making ballistic armor were limited to small tiles (6" x 6"). The ceramic composite armor systems fabricated for use to protect areas such as pilots torso or back regions consisted of tiles butted against each other and adhered to a glass reinforced plastic. These multiple tile arm systems had to be overdesigned to provide protection against "tile intersection impacts." The velocity to penetrate a two-tile intersection (good fit) was found to be approximately 15% below that required to penetrate the center of tiles. Protection was provided for these vulnerable areas by added weight of GRP backup material behind the joints or increased ceramic thicknesses. The added weight required for joint protection increased the areal density above the required maximum weight of 10 lb/ft^2. Another design approach was to increase the ceramic thickness at the joints only. This resulted in alumina armors at weights slightly over 10 lb/ft^2.

A concentrated effort was initiated to encourage the ceramic manufacturer (Coors Porcelain Company) to investigate fabrication and firing techniques to produce alumina in large one-piece (monolithic) curved pieces. It was believed that a one-piece alumina construction would eliminate the vulnerable areas and provide, in essence, only center-of-tile impacts. This approach would also result in a savings of weight over the multiple tile systems.

In the fabrication of alumina ceramic face plates, considerable shrinkage is encountered and usually results in warpage and nonuniform wall thicknesses. The degree of warpage and shrinkage may vary from plate to plate even though the same fabrication techniques and the same mold are used in each case. If all the backups are premolded, from the same mold, misfits between ceramics and GRP will result. This usually results in extremely thick adhesive layers. Experience in the lightweight armor program for protection against small arms indicates that thick layers of adhesive adversely affect the ballistic performance of the composite armor. In light of this evidence, it was decided that this Laboratory would select a fabrication technique that would result in a near perfect fit between the contoured ceramic and glass reinforced plastic. This was accomplished with a vacuum bag under pressure and heat in an autoclave. It was this concept, i.e., utilizing each ceramic breastplast as a mold for an individualized backup plate, which made the one-piece monolithic alumina-GRP composite armor practical.

The total cost savings resulting in the utilization of one-piece (monolithic) constructions over the multiple tile arrangements cannot be accurately evaluated at Picatinny Arsenal. However, cost estimates on one-piece breastshields and on the multiple tiles required to fabricate a shield of equal size were tabulated for sets of 20 and 500 as estimated by Coors Porcelain Company. It is believed that the cost differential would be greater today since more experience in making one-piece ceramics has been accumulated. Some of the advantages over multiple tile are:

1. Increased production of armor
2. Faster delivery dates
3. Eliminated high cost of grinding tiles
4. Simplified inventory
5. Lower weight for a given threat
6. Low-cost fabrication of backup and composite
7. Gave incentive to other ceramic manufacturers (B_4C, etc.) to produce one-piece monolithic ceramics.

While the work was initiated for body armor, seat panels and other flat sections are now made in larger sizes. This reduces the number of vulnerable areas.

e. Personnel Armor (1968). Intensive effort was initiated during World War II to create a practical system and materials for protecting the soldier from high velocity missiles generated by shell and grenade fragmentation. Studies of energy absorption and dissipation in oriented extensible materials led to the attainment of "soft" armor made of multiple layers of ballistic nylon fabric. Protective vests incorporating this system were used in the Korean conflict with considerable success.

Continuing research to improve the ballistic efficiency and decrease the weight and bulk of these soft armor materials is being made at all levels. Studies range from polymeric and supra-molecular response phenomena, through translational effects in fiber, yarn and fabric structures, to after-treatments and assembly systems. The information gained has shown the way to practical improvements both in the protective levels obtainable and in the controls necessary to realize these benefits consistently and economically. One change made in the basic yarn form in 1962 made a significant increase in ballistic performance and resulted in savings of approximately 5 million dollars in one procurement alone.

The principles of soft armor have been extended to use of the ballistic fabric in helmets and helmet liners where the plastic matrix provides the necessary firmness for the desired shape and contributes to low velocity impact resistance.

3. Aircraft Canopies and Windshields

Stretched Acrylic Development (Ref. 46). NAVAIR and AFML sponsored research on materials for aircraft canopies and windshields from 1950 to 1959. This effort resulted in the development of stretched acrylic which has for several years been used in almost all military and commercial aircraft. Excerpts from 1968 notes concerning the development of stretched acrylic follow.

Acrylic plastic, more commonly known by the trade names Plexiglas and Lucite, was the preferred glazing for aircraft windows and canopies at the end of World War II. By 1950, inflight failures were frequent and were often attributable to crazing, which consists of patterns of small cracks, interacting with the notch sensitivity of the acrylic. The problem was aggravated by growing pressurization and aerodynamic load requirements. Laminating two sheets of acrylic together with a soft interlayer, as is done with glass, reduced inflight failures but added cost and weight to the canopy and delamination to the causes of high replacement rates.

By this time there were recurrent observations that severely formed parts, such as the crowns of gunner's bubbles, did not craze and appeared to resist cracking. BuAer initiated a project at the National Bureau of Standards to investigate in-plane stretching of acrylic sheet as a means of combating crazing. At about the same time, the Naval Research Laboratory was investigating a technique for determining the toughness of materials by measuring the resistance to crack propagation. Among the materials included in the program was stretched acrylic, which was found to be many times tougher than as-cast acrylics. This was the beginning of a program of ten-year duration at NBS and NRL (and the Air Force and the aircraft glazing industry).

Molecular orientation was responsible for the increase in toughness and for the substantial reduction in crazing, with essentially no loss in other properties. Factors such as the optimum amount of stretching, the optimum temperature and stretching rate, and the techniques for handling and forming stretched acrylic were determined, with the crack propagation test as a monitoring tool to assure acquisition and retention of the desired toughness. Ultimately, the crack propagation test and later a shrink-back test were developed as quality control procedures.

As early as 1955, stretched acrylic won acceptance as an aircraft glazing material and as further information and service experience were obtained, it won further acclaim.

The Navy and Air Force R&D costs for development of stretched acrylic is estimated at $700K (Ref. 46).

4. Metal Processing

Outstanding advances in metal processing have been achieved, in recent years, through Government and Industrial funding of R&D and manufacturing technology programs. Superplastic forging powder metallurgy, directional solidification, hot isostatic pressing (HIP), and near net shape processing are some of the techniques that are causing major changes in manufacturing procedures.

In the light of these advances, it is interesting to review the participation of the Services in the early R&D which led to advanced fabrication processes. Following are excerpts from Ref. 45 documents.

a. Research on Superplasticity (1968). ONR investigators, such as Professor W. Backofen, MIT, and Dr. E.E. Underwood, Lockheed-Georgia, continue to elucidate the variables and mechanisms of superplastic behavior, i.e., the ability to plastically stretch an alloy by vast amounts without failure. A complete understanding of superplastic behavior has many technological implications. Among them are increases in the faithfulness of coining operations, the ability to form alloys with much smaller and inexpensive machinery, and the production of wire without the use of short-lived expensive dies. Progress on this research has been necessarily slow and difficult, but results look promising for the application of this phenomena to the metal working industry.

Note (1979): NAVAIR's interest in superplasticity began in 1962, sparked by a paper by Dr. Underwood. NAVAIR started work in 1963 and spent about $500K on the technology (Ref. 47). The technology advanced significantly in recent years and is being used in many applications.

b. **Metal Processing (1970)**. A few years ago, it became evident that the success of efforts to develop (for advanced weapons systems) alloys of higher strength/weight ratio, higher temperature resistance, and greater resistance to deformation presented a paradox. The better the materials, in fact, the more difficult they were to deform. It was obvious that there was a need for improvement of processing equipment to fabricate these new materials into useful shapes. The Services banded together in what was called the Metal-Working Processes and Equipment Program to investigate new methods of fabrication based on scientific principles. Methods found to be particularly fruitful are high pressure containment of the work piece, superplasticity, and use of ultrasonic vibration.

The imposition of high pressure on the work piece during metal processing increases the ductility of the metal and allows it to be shaped more readily. Brittle metals and alloys incapable of being fabricated to a certain shape without fracture now can be--through the application of pressure. The phenomenon was quickly adapted to the extrusion process, since containment by one means or another is part of the process, and now success has been achieved in the extrusion of the relatively brittle refractory metals by this method. By use of this principle, one company is now production-forming parts such as deep shells, expanded tubes, and flanged components. Copper electrical connectors are an example of an end-item made by this process. The somewhat more complicated tooling and additional expense are more than compensated for by the reduction of necessary secondary operations, and by the higher strength and quality of the resultant parts.

The term "superplasticity" is used to denote such phenomena as drastic increases in ductility or decreases in deformation resistance in certain metals and alloys when they are undergoing a phase change. The phenomenon has been observed in iron and steels and in certain aluminum alloys and copper alloys. Practical use is now being made of superplasticity. It is being employed in the fabrication of stainless steel, and International Nickel Company has developed a series of stainless steels which exhibit superplasticity. One such alloy elongates up to 600% during tension in the 1600-1800 ^{o}F range. This is considered to be a breakthrough in this field. Some new zinc-aluminum alloys exhibit improved plasticity and can be easily fabricated into complicated automotive parts by the forging process; also, these alloys are not as prone to brittle fracture as are the zinc die castings normally used for these applications. For superplastic forming operations for aerospace applications, titanium alloys have the greatest potential. Not only can difficult-to-form metals be easily deformed by use of superplasticity, but also much lighter and cheaper equipment can be utilized, or on the other hand, the working range of the equipment can be expanded and its speed increased. Efficiency is the keynote of this means of deformation.

The superimposition of ultrasonic vibration on the applied stress during deformation of metals has been found to have two effects: (1) the marked reduction of the yield stress in the material and (2) the reduction of friction between two contract surfaces. These two effects suggested that the fabricability of metals could be improved by the use of ultrasonic energy during processing. This prediction has been proved to be correct, and several laboratories and plants are now fabricating materials with the aid of this technique which is particularly adaptable to wire drawing and tube drawing. This effect has also been shown to be beneficial in the compaction of ceramic materials.

c. Directional Solidification of Metal Alloys (1968). The Naval Air Systems Command has pioneered in the support of research and development in a new method of producing high strength alloys for use in aircraft and missiles. It has been found that with the alloys of special composition (eutectic), very careful control of the way they solidify from the melted state, can cause the precipitation within the alloy of fibrous or plate-like precipitates which are very high in strength. Proper solidification conditions (usually freezing progressively from one end) will cause orientation of these high strength precipitates and can strengthen the metal in a manner somewhat similar to the way glass fibers strengthen plastic matrices in glass-reinforced plastics.

Thus far, the above research has led to development of a series of "controlled eutectic" alloys. The most promising of these is a nickel-aluminum-columbium alloy that has a tensile strength of 115,000 psi at 2000 °F and a 600-hr stress rupture strength of 20,000 psi. These values are more than twice as high as the best superalloys now available. Attention will now be given to characterizing and, if necessary, to developing the other necessary attributes in this alloy, and to exploring other alloy systems for advantageous compositions.

d. Research on Metastable Phases (1968). The ability to develop nonequilibrium phases by rapid or ultrarapid cooling (splat cooling) continues to stimulate the research community. Implications of this research range from the development of structures with new and improved mechanical and electrical properties to the possibility of developing superconducting materials with transition temperatures near room temperature.

Among ONR contractors active in this field are Professor M. Cohen, MIT, whose interests are in the mechanical properties of metastable hexagonal iron-carbon alloys and Professor J. Mackenzie, RPI, whose work on metastable oxides could lead to new improved ferrite materials for computer memories.

e. Current Metal Processing Technology (1979). Today, the use of superplastic processes to optimize fabrication of components made of certain metals and alloys is expanding rapidly. Gatorizing, a patented Pratt & Whitney process, is described as a superplastic forming or isothermal forging process. The dies and the part being forged are held at the same temperature (2150 °F). TZM molybdenum dies are used because their physical properties can be maintained at the elevated temperatures. However, the process is carried out in a vacuum or in an inert atmosphere because Mo will vaporize in the air at the processing temperature. The metal is brought under pressure and heated to the "superplastic state" where it flows under the pressure but is not truly at its melting point. IN100, a nickel alloy, has been used in the gatorizing process.

Powder metallurgy is at the heart of the advanced forming processes, and when used with hot isostatic pressing (HIP) makes a one-step forming process that will probably replace the two-step gatorizing process at Pratt & Whitney. IN100 is not HIP-able, so a new alloy (MERL 76) has been developed that may be used in the advanced process.* This new Pratt & Whitney process will enable turbine blades to operate at 100 °F higher temperatures than the strongest cast turbine blade made today. If the Pratt & Whitney F-100 engines were fitted and retrofitted with the new blades, the higher operating temperature would translate into a saving of $120K over the engines' lives through a decrease of nearly 1% in specific fuel consumption.**

Near net shape is the art of manufacturing a forging, casting, or pressing that requires considerably less final machining than is required by conventional processes. Near net shape actually is the goal of most innovative manufacturing today because, during the past 5-6 years, high temperature, high technology alloy raw materials have increased in price 60-100% or more.

The General Electric CFM56 turbine shaft illustrates the payoff in near net shape technology. The finished piece is a 10-lb structure made of Rene 95, a nickel-base superalloy. The conventional forging delivered to GE weighs 450 lb of which 431 lb is an envelope that was machined away. Starting with Rene 95 as a powder rather than a billet or metal preform, the delivered piece weighs 120 lb. The industry still is not happy with this ratio, but it represents a substantial gain in near net shape.

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*<u>Aviation Week</u>, November 20, 1978.

**<u>Aviation Week</u>, October 16, 1978.

Rene 95 was developed by General Electric. A new nickel-base alloy, which is called AF115, is under development by the Air Force and General Electric, and the material is expected to have superior properties to Rene 95 and IN100--both of which now cost approximately $15/lb in powder and powder/billet form.*

Directional solidification is now being used in the production components of gas turbine engines. In the order of 200,000 components have been fabricated by one company using this process.

The rapid solidification rate (RSR) powder process holds promise of producing superior high priority powders for the advanced manufacturing processes. The authors did not have time to investigate this process fully.

5. Other Technologies

There are several systems or technologies which the authors did not have time to puruse but which may have contributed to this study.

a. The B-1 Bomber Program. Before it was cancelled, the B-1 program included many high technology items and it made broad use of titanium, advanced composites, and advanced structural design. Many of the items planned for the B-1 may be used in other new aircraft construction (see Section VI-B).

b. Radomes and Antenna Window. Development programs included interesting work on boron nitride reinforced with boron nitride fibers (Bn-Bn) for windows and environmental resistant coatings for radomes.

c. Paints, Lubricants, Elastimers, and Seals. Important advances have been made in these areas especially for aircraft,

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*Aviation Week, October 16, 1978.

ship and submarine applications. These advanced materials reduced costly maintenance and/or increased capability and reliability of the components.

d. Cermets and Energy-Absorbing Materials. Programs were not reviewed.

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GLOSSARY

AAH	Advanced Attack Helicopter
ABM	Anti-Ballistic Missile
ABRES	Advanced Ballistic Re-Entry Systems Program
ADP	Advanced Development Program
AEC	Atomic Energy Commission
AFASD	Air Force Air Systems Division
AFFDL	Air Force Flight Dynamics Laboratory
AFML	Air Force Materials Laboratory
AFOSR	Air Force Office of Scientific Research
AMMRC	Army Material and Mechanics Research Center
ARC	Atlantic Research Corporation
ARO	Army Research Office
ASD	Aeronautical Systems Division
ASME	American Society of Mechanical Engineers
ASTD	Advanced Structures Technology Demonstrator
ASTM	American Society for Testing and Materials
ATJ	Union Carbide Corporation Graphite (Company Designation)
ATJS	Union Carbide Corporation Graphite (Company Designation)
AVSCOM	Army Aviation Systems Command
AWS	American Welding Society
BIE	Boundary Integral Equation
BuAer	Bureau of Aeronautics (Navy)
C-C or C/C	carbon-carbon
CLF	Controlled Live Facility
CNO	Chief of Naval Operations

CVD	chemical vapor deposition
DARPA	Defense Advanced Research Projects Agency (formerly ARPA)
DDR&E	Director of Defense Research and Engineering
DSRV	Deep Sea Rescue Vehicle
ECOM	Army Electronics Command
EDP	Engineering Development Program
EEC	extendable exit cone
ERDA	Energy Research Development Administration (now DoE)
FM	frequency modulation
FYDP	Five-Year Defense Program
GAO	General Accounting Office
GEMS	Generalized Energy Management System
HDL	Harry Diamond Laboratories
HiMAT	Highly Maneuverable Aircraft Technology
HIP	hot isostatic pressing
HTS	high-tensile steel
IAC	Information Analysis Center
ICBM	Inter-Continental Ballistic Missile
ICM	Interagency Council for Materials
IITRI	Illinois Institute of Technology Research Institute
IMA	intermediate maintenance activity
IRBM	Intermediate Range Ballistic Missile
IR&D/B&P	Independent Research and Development/Bid and Proposal
I_{sp}	specific impulse
ITE	integrated throat and entrance
JTA	Union Carbide Corporation Graphite (Company Designation)
JV	Joint Venture

LCT	low-cost titanium
LMSC	Lockheed Missile and Space Corporation
MAB	Materials Advisory Board (now National Materials Advisory Board)
McDAC	McDonnell-Douglas Aircraft Corporation
MDF	mild detonating fuze
MERADCOM	Mobility Equipment Research and Development Command
MICOM	Army Missile Command
MIRV	Multiple Independently Targetable Reentry Vehicle
MSAT	Missile System Advanced Technology
MSTE	Missile System Technology Evaluation
MT	Manufacturing Technology
MX	Missile X (a term for Advanced ICBM)
NASA	National Aeronautics and Space Administration
NAVAIR	Naval Air Systems Command
NAVSEA	Naval Sea Systems Command (also NSSC)
NAVSEC	Naval Ship Engineering Center
NDT	nondestructive testing
NMAB	National Materials Advisory Board
NOL	Naval Ordnance Laboratory (now Naval Surface Weapons Center)
NOTS	Naval Ordnance Test Station (now Naval Weapons Center)
NRC	Nuclear Regulatory Commission
NRL	Naval Research Laboratory
NSF	National Science Foundation
NSSC	Naval Sea Systems Command (formerly Navy Ship Systems Engineering Center)
NSWC	Naval Surface Weapons Center (formerly NOL)
NUSC	Naval Underwater Systems Center
ODS	oxide dispersion strengthened
ONR/NRL	Office of Naval Research/Naval Research Laboratory
OSD	Office of Secretary of Defense
OSRD	Office of Scientific Research and Development

PBCS	Post-Boost Control System
RDT&E	Research, Development, Test and Evaluation
REI	research-engineering interaction
REVMAT	Re-Entry Vehicle Materials Technology
RFI/EMI	radio frequency interference/electromagnetic interference
RFP	request for proposal
RSR	rapid solidification rate
SCC	stress corrosion cracking
SMS	Synchronized Meteorological Satellite
SPFDB	superplastic forming and diffusion banding
SSPO	Strategic Systems Project Office
SST	supersonic transport
STS	special treatment steel
TACOM	Army Tank Automotive Command
TCP	Technology Coordinating Paper
TTL	Transistor-Transistor Logic
TVC	Thrust Vector Control
ULMS	Undersea Long-Range Missile System
UTC	United Technology Center
UTTAS	Utility Tactical Transport Aircraft System
VLF	very low frequency
VSTOL	Vertical and Short Take-Off and Landing