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ARPA Project

"High Performance Composites"

FIBERS FOR HIGH PERFORMANCE COMPOSITES:

A Literature Survey

Charlotte M. Bower, Editor

W. C. Peterson

John D. Calfee, Program Manager

Each transmittal of this document outside the Department of Defense must have prior approval of the Director, Material Sciences Division, Office of Naval Research

August 31, 1965

Monsanto Company/Washington University St. Louis, Missouri

Joint program sponsored by the Advanced Research Projects Agency, Department of Defense, through a contract with the Office of Naval Research, N00014-66-C-0045, order number 1001/58(C-65-006).

FOREWORD

The research reported herein was performed under the sponsorship of the Advanced Research Projects Agency, Department of Defense, through a contract with the Office of Naval Research, N00014-66-C-0045, order number 1001/58(C-65-006).

The prime contractor is Monsanto Research Corporation. The Program Manager is Dr. John D. Calfee (Phone: 314-WY 3-1000, station 3754). The work is done by Washington University, St. Louis, Missouri with Dr. James M. McKelvey (Phone: 314-VO 3-0100, station 4464) as Project Director, and Monsanto Company, Central Research Department, St. Louis, Missouri, with Dr. John D. Calfee as Project Director.

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ABSTRACT

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The present state-of-the-art and the future promise of fibers for use in high performance composites are discussed. The literature survey, which covers the period from 1957 to August 1965, inclusive, contains 235 references with abstracts.

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I. INTRODUCTION

There is a widespread recognition of the potential of fiber reinforced materials. The recent trend of rapid growth of the technical literature and the varied use of such materials support this conclusion. There has been particular interest in an area known as high performance composite structural materials. Experience and theoretical studies have shown that fiber will always be stronger than bulk materials and engineers need to find a way to use this property - hence, composites. The trends to develop stronger, stiffer, more refractory fibrous materials has arisen largely from an urgent need for reinforcing substances having very high strengths and low weights. Filaments, in addition to having desirable strength to weight ratios, can be combined with other materials to form systems with particular properties for specific purposes. An example is the glass filament wound plastic rocket motor cases that are finding use and great promise in materials for aerospace applications with their great strength to weight ratio being their main asset.

The field of composite materials is a relatively new one and until recently, little of a scientific nature was done. The area of fiber reinforced materials advanced slowly with almost all the work being empirical and progress coming with improved technology. The first significant work in the area of high performance reinforced materials appeared in the literature as random, unorganized, and unreliable presentations of experiments conducted using glass fibers as a reinforcing agent in plastic resins. Since then, the sophistication of the materials, methods, approaches, and end use have changed greatly; now the area of composite materials and especially fibrous composite materials represent the source of the strongest and most adaptable of all known materials. With light and strong materials as the goal in the materials field, the fibers for reinforcement naturally became of interest and investigations were begun to match fiber and matrix in a composite form to the task at hand. In the area of fibrous reinforced high performance composite materials, the fibers of main interest have been divided into various groups for discussion:

- A) Special glasses and silicon or vitreous fibers
- B) Metallic fibers
- C) Ceramic fibers
- D) Carbon and Graphite fibers
- E) Other asbestos, organic, composite fibers

II. DISCUSSION OF FIBER FOR HIGH PERFORMANCE COMPOSITES

. GLASS FIBERS

Glass fiber, as a reinforcement, had admirable properties as early workers found. High strength and low weight were the primary attributes. For a while , glass fibers were the only reinforcing agent under consideration and as a result, the technology of their production and usage was refined. Experimentation with glass composition, treatment, drawing and all other production variables was carried out along with work in the areas of composite fabrication and testing procedures.

The properties of glass fibers, particularly the strength properties, are subject to many factors which must be controlled in order to obtain predictable and reproducible results. Composition and temperature of the melt, drawing and cooling speed, environment after drawing, coating, and other factors are some of the variables which account for the variation of properties attainable for glass fibers. Compositional factors determine the theoretical or potential strength of the fiber. This is determined by calculation of the force necessary to actually break interatomic bonds and does not account for surface flaws and cracks. Theoretical calculations estimate tensile strengths of one to two million psi for common glasses, depending on composition. The chemical constituents of glass also govern its hardness, resistance to corrosion, thermal properties, density and electrical properties among others. "A" glass is a high soda lime or "alkali" glass and is susceptible to a weight loss in aqueous environments of 10-15%. Electrical or "E" glass, a borosilicate glass, with Be₂0₃ added and less SiO₂ and little alkali content, has a higher softening point than "A" glass and has good resistance to water and alkali but poor acid resistance. "E" glass represents the composition in largest usage in reinforced plastics. Chemical glasses, "G" glasses, are a series of glasses which show improved chemical resistance and can be used in environments of adverse nature. High modulus glasses are highly desirable in providing increased stiffness to laminate structures particularly and various composition experiments to this end have been conducted with results ending in the production of glasses designated as YM31A, Imperial N672, and House 29A. High tensile strength glasses have been produced, House 29A and S-994. Along with the high modulus and strength goes an increase in density so some strength to weight advantage is lost. The glasses are expensive, difficult to fiberize and where BeO is used, possibly toxic. Environment, coating and handling are all major factors in the difference between the theoretical and observed values of the strength of glass fibers. Bare glass fibers rarely have strength exceeding 200,000 psi. due to the relatively poor abrasion and corrosion resistance of the filament surface. Flaws occur quite readily upon contact with other fibers and failure will occur at a crack long before the intrinsic strength is taxed. As a result, efforts have been made to protect the surface of the fiber by applying a protective coating at the time of production. The sizing material applied

to the fiber is usually one of two types; a starch oil emulsion or a surfaceactive agent referred to often as a coupling agnet. Filaments with a coupling agent size are expressly for the purpose of reinforcing plastic resins.

Common coupling agents for use in epoxy, phenolic, and amine resins are Y-4087 or chemically, γ -glycidoxypropyltriethoxysilane and A-1100 or γ -aminopropyltriethoxysilane. In polyester and other common resins not mentioned above, A-150,A-151, A-172, all vinyl silanes and A-174 or γ -methacryloxypropyltrimethoxysilane, are used to improve the resin-glass bond. These finishes improve the wet-strength of laminates made with glass fiber by 50% to 500% over bare glass fibers fabricated into similar laminates.

Another variable which causes a weakening of glass fibers is heat treatment. A glass fiber compared to bulk glass of the same composition will usually be of higher tensile strength, lower modulus of elasticity, lower density, and other differences. These properties result from being chilled from forming temperature of about 2300°F to room temperature in less than .1 sec. and are the result of the fact that the glass structure does not stabilize because of the high room temperature viscosity. Therefore as a fiber is annealed at an intermediate temperature, its properties return to more like the bulk state. Heat cleaning of woven glass cloth to remove the starch-oil size applied before weaving will, therefore, weaken the laminate produced from it.

Another variable to be considered is the variation of strength with the length the filament. It is a statistical problem with the continuous filament having the greater chance of having a surface flaw and failing more prematurely than a short fiber.

With the knowledge of the many variables controlling the strength of glass fibers, technology has been able to produce commercially, fibers with ultimate tensile strengths ranging from 300,000 - 700,000 psi and moduli up to about 17,000,000 psi in spite of the many handicaps presented. Also experimental glasses and fused silica fibers with strengths over 1,000,000 psi are being produced in laboratories. There is still room for improvement in the strength of commercial glass fibers. The factors mentioned affecting their strength emphasizes the critical problem areas in which further research and development are needed.

Glass and fused silica fiber have been used in a multitude of matrices as reinforcing agents and in many fabricated forms. Filament wound structures take advantage of the full tensile properties of the fiber as the mechanical properties in any one direction are proportional to the volume percent of the fibers oriented in that direction. A filament wound structure is mechanically a unidirectional composite. Glass cloth and mat laminates lend strength in a plane, and in many cases, increased elastic modulus and compressive properties are sought in these sheet forms. As fiber orientation becomes more random, the mechanical properties in any particular direction decrease. Short chopped fibers can be used to

reinforce in three dimensions.

Almost all glass fibers, continuous or short, have solid and circular cross sections and are easy to produce and handle. However, other cross sections such as hollow circular, hexagonal, square and irregular have been produced and have potential for improvement of mechanical properties of composites in some ways. Hollow fibers have high stiffness to weight and compressive strength. Square and hexagonal shapes have been shown to pack almost perfectly to more than 90% glass for high strength filament wound structures. These fibers are difficult to handle and incorporate into a composite in other than a laboratory situation as well as being costly at present.

The theory of fiber reinforcement in composites has been studied by many workers. The basic idea is that when a load or stress is applied at a point, it can be distributed along a continuous load path in a shear transfer process at the matrix-fiber interface. The plastic flow of the matrix can be absorbed in a tensile stress on a fiber usually of greater modulus and strength than the matrix material. Also, the materials are usually such that the full tensile properties of the fiber are realized before matrix failure due to elongation. However, the theory cannot account for fibers stressed in fabrication and nonuniform tensile properties and the effects of many other factors. As a result, theory and practice do not coincide but very often a good estimate can be made with a reasonable degree of accuracy.

Fiber	Maximum Tensile Strength	E Modulus of	S.P.G.
	psi	Elasticity	gms/cc
		× 10 ⁶	
E glass	300-500,000	10.5	2.55
YM31A	400-600,000	16	2.89
Imperial			
N-672 glass	520,000	17.3	2.94
S-994	650-750,000	12.5	2.49
29-A	700-900,000	14.5	2.66
Si0 ₂	800-1,000,000 +	10.5	2.2
Steel	600,000	30	7.87
Tungsten	580,000-620,000	58	19.3
Molybdenum	320,000	52	10.2

Glass and silica diameters = 0.00038 in.

Metal wire 0.006 in.

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Glass fiber and all its virtues cannot fulfill all the requirements demanded of a reinforcing agent in the field of high performance composites. The strength properties of glass and silica fibers fall off fairly rapidly as soon as the temperature rises above room temperature and become virtually useless long before they reach their melting point. Also vitreous fibers are subject to static fatigue, i.e. delayed failure. Often less critical handling is required and different electrical, thermal and mechanical properties are desired and other materials must be used.

B. METALLIC FIBERS

Metal fibers or wires are another source of good reinforcing agents for high performance composites. The research on metal fibers as reinforcements was not as haphazard as the first experiments with fiberglass. For the most part, the application of metal fibers in composites is a relatively new and premeditated endeavor. The objectives are to produce high performance materials of specific design, properties and use.

Almost all the high strength metals have been tried as a reinforcing agent in plastic, metal or ceramic matrices. As would be expected, metal fibers change the properties of the matrix material greatly. By incorporating short, random metal fibers in plastic resins, systems can be made with impact strength, wear resistance, thermal conductivities and heat distortion points greatly exceeding those of glass fiber reinforced materials. Besides being mechanically strong and tough materials, they can be designed to be easy to machine and be good conductors of electricity. Continuous metal wires have also been investigated as a reinforcing agent for plastics in filament wound structures particularly. Here, the strength to weight ratio must be considered if it is to be in contention with glass fibers. Because of this fact, light metals are of main interest with boron and beryllium leading the way. Where weight is no factor, steel and tungsten, etc., can be used. Fatigue resistance, high strength, chemical resistance and ease of handling compared to glass fibers are their main attributes. Similar to glass fibers, metal filament can be used in a variety of forms. Mats, wools, woven fabrics are some examples plus felts and curled shavings which set them apart from glass.

In a metal matrix such as aluminum, copper, or cobalt, metal fibers change the mechanical strength properties significantly. In a ceramic matrix, they have effect on the thermal, electrical, strength and ductile properties of the composite.

The fibers of greatest interest have been those produced from the refractory metals, tungsten, nickel and its alloys, steels, molybdenum, boron and others. These refractory fibers exhibit greater strength at elevated temperature than do glass fibers and as a result, the composites made from these materials have higher operating temperatures.

The production of metal wires of reproducible properties is usually

done by drawing or extrusion but there are limits on diameter. Smaller diameters can be obtained through bundle drawing to 0.0005 inch. Like glass, fine metal wires appear to increase in strength with decrease in diameter with the exact cause not known. "Taylor process" wires must be mentioned as a method of producing wires of very small diameter, on the order of 10^{-5} cms. A molten metal encased in a glass or quartz tube is drawn down to a fine filament and the glass removed by etching. The glass apparently lends dimensional stability during drawing and prevents necking which limits standard procedures.

Fiber	Maximum Tensile Strength	E Modulus	S.P.G.
	psi	Elasticity x 10 ⁶	gms/cc
Aluminum	90,000	10.6	2.70
Beryllium	200-250,000	44	1.84
Boron	400,-850,000	64	2.59
Molybdenum	200-320,000	52	10.2
Rene' 41	300-600,000		
Stee1	600,000	30	7.87
Ti-13V-11Cr-3A1	280-320,000	16.7	4.7
Tungsten	580-620,000	58	19.3

All wire except Be .006 in.

Be .007 in.

To the ends desired, metal fibers are effective but not without problems. At elevated temperature, metal fibers are subject to oxidation and recrystallization, both of which reduce their strength. In metal-metal combination composites, brittle interface bimetallic compounds can form, which are undesirable. In ceramic materials, wetting of the metal surface for maximum bonding is a problem and as in all other matrices, the differences in thermal expansion rates create stress problems. These problems and the fact that strength is reduced at higher temperatures, though not to the extent of glass, suggests that metal fibers are limited in their scope of application and that other materials are necessary to remedy this situation.





This graph shows an application of metal fibers as a reinforcement in metals.

C. CERAMIC FIBERS

Ceramic fibers present a source of high performance materials relatively new in nature. Ceramics offer potentially higher strengths than most other materials. In bulk form, however, they are usually weak and brittle due to internal and surface defects. Most commercially available fibers and materials have already become inadequate with the taxing environment of aerospace applications. The problems are mainly in strength at elevated temperatures. An increasing effort in the area of ceramic fiber technology with special regard to high temperature structural materials is now being put forth on development, testing and evaluation of materials.

Methods of production and testing in this field are not as well developed as in the area of glass fibers and sometimes a critical eye is needed to interpret the data reported to obtain meaningful information.

A variety of ceramic fiber compositions for high performance use have been developed. These fibers are used primarily as reinforcements in composites, but other applications include use as insulators, fillers and filters. They are produced, with varying degrees of difficulty, as either long, continuous filaments or short fibers, and formed into yarns, rovings, felts, etc. Fabrication and handling of ceramic fibers are similar to the methods used for glass and metal fibers though modification or further development of present techniques is sometimes necessary.

With specific reference to reinforcement in composites, ceramic fibers have certain properties which set them apart from the previously mentioned classes of materials. They have higher melting points, higher elastic moduli, great hardness, good chemical resistance, and have high tensile strengths when made carefully. Therefore, economy must be considered when designing with these high modulus fibers, and thought has been given to combinations of glass fibers for tensile strength and ceramic filaments for stiffness in composites. At present the high cost of ceramic fibers for reinforcement limits their use commercially to proprietary projects and research.

Metals and plastic resins are of main interest as matrix materials for ceramic fibers. In plastics longer fibers are desirable due to the low shear strength of the polymers and more length is needed for stress transfer. The higher shear strength of metals permits the use of short fibers, provided they are well bonded to the matrix. As insulators, fibrous wools of ceramic materials are becoming more available and have been found to have admirable properties. The useful temperature limit of a given insulator is determined by the composition of the fiber.

The strength of a ceramic fiber, as in all materials, depends to a large extent on its microstructure. Crystalline size, inclusions, voids, cracks, etc. all affect the properties of a material. The microstructure in turn is directly related to the nature of the material, the production process,



J. H. Ross, Air Force Material Symposium (Tech. Papers) June 1965

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and treatment of fibers. Ceramic-polycrystalline fibers of interest can be produced in several ways: colloidal evaporation process, exvrusion, rayon spinnerette method and vapor deposition of filament substrate.

Zirconia fibers have been produced by rapid evaporation of stabilized suspension and heat treatment of the ribbon-like fiber that results. Other materials being studied for this process are Al_2O_3 , ThO_2 SiO₂ and some mixtures. The tensile strengths are low but the fibers are good for insulation.

Fibers of materials such as zircon, zirconia, alumina, and magnesia have been formed by extrusion and heat treatment of fine particles. Fine grain filaments of strengths of 200-340,000 psi are common. Careful control of thermal treatment must be observed to prevent excessive grain growth.

The rayon spinnerette method has been used to produce continuous polycrystalline fibers of alumina, zirconium silicate, aluminum silicate, zorconia and rutile.

Vapor deposition is a technique for producing filaments by reducing or decomposing a volatile compound of the desired material onto a fine substrate wire or other filament. The volatile compound must vaporize at a temperature well below the melting point of the substrate and be stable enough to resist decomposition until it contacts the substrate.

$$BC1_3 + CH_4 \xrightarrow{\text{at the substrate}} B_4C + HC1 + H_2$$

Although composite polycrystalline filaments made by this method have varying properties depending upon substrate and other variables, close process control has resulted in some strong filaments. Recently, workers have used this method and reported filaments with strengths several orders of magnitude greater than presently produced fiber. Fibers of boron, boron carbide, silicon carbide, titanium boride, titanium carbide, alumina, beryllia, beryllium, beryllium carbide, and tungsten carbide have been produced by this method and show great promise for the future. Though a large number of materials which could not otherwise be produced in fibrous form can be made in this way, the present state of development of this method is very young and as a result, expensive. Many factors must be understood and manipulated to produce high quality materials in quantity and at

D. CARBON AND GRAPHITE FIBERS

Elemental carbon base fibers are a small but important class of fibers due to the special nature of their properties. Though chemically identical, the crystallographic forms are varied. Carbon fibers have an amorphous structure while graphite fibers represent the hexagonal allotrope. This difference in crystal structure arises from the method of production.

Synthetic filaments or fabrics can be thermally treated in a reducing atmosphere to produce partially carbonized, carbonized, or graphitized materials. The difference in these closely related materials is generally defined as follows: Fibers prepared at a temperature of $1300-1700^{\circ}$ F are referred to as partially carbonized or carbonized. The degree of carbonization depends upon the length of time at the process temperature. Partially carbonized materials are up to 90% carbon and carbonized indicates 90-98% carbon. Graphite fibers are produced from organic filaments or fabrics at $4900-5400^{\circ}$ F and are 98% + in carbon content. These definitions are not alwasy strictly adhered to and as a result the literature is sometimes misleading.

Another major process of producing elemental carbon filaments not previously mentioned is the deposition of pyrolytic graphite on a filamentary tungsten substrate by pyrolysis of hydrocarbon vapor in contact with the tungsten. The resulting fiber has a core of tungsten carbide.

Carbon and graphite have many desirable properties and characteristics such as high thermal resistance, light weight, chemical inertness, low thermal expansion, and thermal shock resistance. As a result carbon and graphite fibers have become very important engineering and specialty purpose materials and have been used in industrial, military and scientific applications. There are limitations on certain properties which restrict their use in some cases. These include oxidation susceptibility, high thermal conductivity, and poor abrasion resistance. The strength of carbon and graphite fibers is another property which has not been optimized. The potential strength of these materials is extremely high but current usage is mostly in reinforcements for ablating thermal protection systems rather than in high strength applications. Commerically available carbon and graphite fibers have tensile strengths up to 250,000 psi and moduli up to 7,000,000 psi depending on the method of preparation. Considerably higher values have been reported but they are only on a laboratory scale as yet.

The primary use of carbon and graphite fibers as mentioned before is in ablation applications. This use was initiated when it was realized that this fibrous material did not melt but vaporized slowly at a very high temperature. (6600° F) Their use was further enhanced by the increase in strength with temperature to twice room temperature value at 3000° F . The creation of textile forms of these materials considerably broadened the sphere of usage. Cloth, fibers, felts, battings are now available and provide missile-parts designers with materials for reinforcement of high temperature plastics such as some phenolics. These composites have superior ablation properties for short duration exposure to very high temperature.

Most metals and ceramics do not wet the surface of graphite and carbon fibers or fabrics unless specially treated, though they are readily wet by a wide range of organic resins. Other uses for these materials include filters for corrosive liquids and vapors, fire protective curtains, conveyor belts, and, in the case of "Pluton", 1. a carbonaceous material which is electrically non-conducting, as electrical and thermal insulators.

Graphite and carbon fiber technology is new but is progressing rapidly due to the excellent properties of the materials and the interest shown by the workers in the field of high performance composites.

E. MISCELLANEOUS FIBERS

Among the fibrous materials that cannot be properly classed in the proceeding sections of high performance materials are the natural inorganic fibers such as asbestos, composite fibers such as core-sheath filaments, and synthetic organic fibers such as nylon and rayon.

Asbestos has long been a major raw material for a great variety of essential products. Asbestos is the name given to a group of fibrous minerals that occur naturally throughout the world. Crysotile and crocidolite asbestos are the main types around which most interest in the high performance composite field is centered. Crysotile is a hydrous magnesium silicate of a very fine fibrous structure, on the order of millionths of an inch in diameter, possessing high strength and can be fabricated into many textile forms. Owing to their small diameter, the testing of individual fibers is difficult and as a result these hollow filaments have been reported with varying properties. A common strength value obtained is 350,000 psi. Crocidolite asbestos, commonly known as blue asbestos is a complex silicate of iron and sodium. Its property of particular interest is its high strength and retention of this strength at elevated temperatures and through rough handling. Values of about 500,000 psi are common and expected.

The characteristics of these fibers which contribute the most to their usefulness as reinforcing agents for plastic compositions are tensile strength, small diameter, and elevated temperature service. The tensile properties of asbestos are excellent and range above most other fibers presently in textile usage. The tensile characteristics under conditions of elevated temperature are also high.

Fiber dimension is important in reinforcing plastics as a high surface area promotes superior bonding and results in high strength and durability characteristics.

The uses of asbestos as a reinforcing agent for high performance composites has been mainly in an application where strength at high temperature was desired. The main industry employing asbestos in this manner has been the missile and aircraft industry. Asbestos in the form of mats, fabrics, felts, papers, yarns and textiles have been used in combination with resin systems such as phenolic, phenyl-silane, silicone, melamine, epoxy and

¹.) Minnesota Mining and Manufacturing Company

some polyesters and fluorocarbons. Also asbestos fibers have been used in combination with other fibrous materials in various metal and ceramic matrices though these are not as common.

Asbestos reinforced plastics offer high erosion and ablation resistance, retention of strength at high temperatures, relatively low cost, chemical resistance and high modulus of elasticity. Excellent wettability provides strong bonding between fibers and resin. Complex parts can be easily made by standard fabrication techniques. These and other more specialized uses and properties qualify asbestos as a good reinforcing agent for high performance composites.

Another unclassified type of fiber in the high performance category is the composite fiber. Some have already been mentioned as ceramic and graphite fibers produced by vapor deposition on a substrate filament. Metal-coated glass and silica fibers produced by core-sheath methods are the other filaments of interest. These materials are primarily special purpose fibers designed to maximize a particular property in a composite or in the fiber itself.

Aluminum-coated fused silica and in general, metal-coated glasses have been produced with varying degrees of success. Metal coatings on glass are sometimes used to aid in wetting another metal matrix. The metal coatings often reduce the strength of the glass fibers but their handling properties in this state are improved. Aluminum coatings on fused silica fiber have been successful and high strength has been retained. Values of 850,000 psi have been reported. The core-sheath method combines the formability of glass fibers with the high modulus of polycrystalline ceramic fibers. Fused silica sheath with cores of alumina, zirconia, and other materials have been produced by drawing with substantial improvement in modulus after heat treatment.

Some organic polymer fibers are very strong. Their strength to weight ratio is good when compared to most metals, but in most applications in composites, they are considered marginal due to the rapid decrease in strength above room temperature. Also their low modulus is a drawback.

III. CONCLUSION

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The science of fiber reinforcement has presently reached a state that it can predict that high strength, toughness and other properties can be produced in composite materials over a wide temperature range.

The potentialities of the different types of fibers described in the preceding discussion suggest that to consider properties such as specific strength and thermal stability is enough data to design structural materials efficiently. In practice, production in large quantities, cost of materials and other factors must always be considered. From the properties of known materials, it is clear that fiber reinforcement may be capable of increasing the strength of engineering materials greatly at elevated temperatures, but for low cost and efficiency, it is clear that further research in the area of high strength, high temperature fibers is required.

IV. BIBLIOGRAPHY WITH ABSTRACTS

A. GENERAL INTEREST

Carroll-Porczynski, <u>Inorganic fibers</u>. Academic Press, Inc., New York, (1958) 353 pp., 157 illus.

This is an encyclopedic work covering both the scientific and technological aspects of the subject. The author has collected technical data from a wide range of sources, chiefly in America and Great Britian, and his list of acknowledgments runs to three closely printed pages. Various chapters have been checked by specialists in particular fields. The references given at the ends of the chapters are copious and well presented; journal references are listed first, the titles of articles given, followed by a list of more general references for further reading. Many chapters also include valuable lists of U.S. and British patents and occasionally of German and Japanese patents, and these are subdivided according to topics. At the end of the volume are subject, mineral, and name indexes. The book is comprehensive in regard to subject matter, covering glass fibers and textiles; glass, slag, and rock wools; refractory fibers; asbestos and its numerous applications; and finally metal and metallized fibers and fabrics.

Carroll-Porczynski, C. Z., <u>Advanced materials, refractory fibers, fibrous</u> <u>metals, and composites</u>. Chemical Publishing Co. (1962) N.Y. 276 pages.

The various chapters of this book present data on high temperature polymers, reinforced materials, fibers of all kinds, fused silica, plastics, metals, asbestos in old and new compositions, coated fibers, metallic fibers. In some cases the only information available is supplied by the firm who developed the product discussed. Glass fibers were not discussed except at objects of comparison as they were not in the high performance range of the other fibers. The chapter headings are: 1) High Temperature Problems 2) Fused Silica Fibers 3) High Silica Content Fibers 4) Aluminum Silicate Fibers 5) Potassium Titanate Fibers 6) Super Refractory Fibers 7) Asbestos-Composite Materials 9) Metallic Fibers 10) Coated Fibers 11) Misc. Fibers. <u>Comprehensive bibliographies given.</u>

Carroll-Porczynski, C. Z. "Progress with refractory fibres." Eng. Materials and Des. (1961) <u>4</u>: 418-424 No. 7

The production and properties of a number of refractory fibres, including those of silica, potassium titanate, graphite, zirconium oxide and other metal oxides, are given and their properties are tabulated. Some of the reinforced plastics are described and their properties are tabulated. Particularly, phenolic resins and these fibers can be produced for high temperature applications.

Coplan, M. J. and Johnson, D. E., "Ultra high temperature textiles," <u>Materials in Design Engineering</u>, Vol. 54 No. 6, (November 1961) pp. 117-123 A. D. Little, Inc.

> Summarizes the present status of high temperature textiles. Included are information on filaments of refractory metals, super alloys, ceramics, and organics.

Cratchley, D. "Experimental aspects of fibre-reinforced metals" <u>Metallurgical Reviews</u> Vol. 10 No. 37, Page 79 (1965)

> This review covers the practical work that has been carried out both on a laboratory and a semi-pilot scale on fiber-reinforced metals and summarizes the results obtained.

Dow, N. F. and Rosen, B. W. "Evaluation of filaments-reinforced composites for aerospace structural applications" NASA CR-207. (April 1965)

> Studies of the influence of constituent properties upon the performance of structural composites for aerospace applications are described. Previous elastic constant and tensile strength evaluations are extended to broaden their range of applicability. An analysis of compressive strength is presented. Properties of fibrous and matrix constituents given in tables.

Goodwin, P. M. "Composite materials," Machine Design 35, 190 (July 18, 1963)

Short <u>review</u> article on design capabilities of present and potential types of fibers.

Hausner, H. H. <u>Modern Materials</u> Academic Press New York (1958) Vol. 1 pp. 143-185, Chapter 3. "Fiber materials."

> This chapter discusses the developments in fiber technology in the areas of organic and inorganic fibers. Under inorganic fibers are included glass, ceramic, asbestos and metallic. Glass and asbestos fibers are discussed in detail.

Hosford, Robert C., "A guide to fillers and reinforcements for thermosets," <u>Plastics Tech. 11</u>, 34-44 No. 2(Feb. 1965)

> A state of the art review article. Tables of selected fibrous reinforcing agents given with physical properties and <u>cost</u>. Characteristics of the plastics these are used in are given, before and after reinforcement, for both thermosetting and thermoplastic materials. Also qualitative discussion of application suggestions for the materials.

Kelly, A. and Davies, G. J., "The principles of the fiber reinforcement of metals." Metallurgical Reviews, Vol. 10 No. <u>37</u> page 1, (1965)

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The present review is divided into six parts. Section I deals very briefly with the essential difference between the fibre reinforcement of metals and conventional methods of strengthening. Section II discusses the strong fibres available today, and some of their properties.Section III <u>develops in detail</u> the theory of fibre reinforcement, while Section IV is concerned with the principles and the practice of methods for fabricating fibre-reinforced metals. This is followed by a summary (Section V) of the conclusions to be derived from these principles, which allows a suitable combination of matrix and fibre for the ideal microstructure to be formulated. A tentative account of the circumstances in which fibre reinforcement can be used to the best technical advantage is also included in this section.

Machlin, E. S., "Non-metallic fibrous reinforced metal composites," AD-265 943 (September 1961).

> The literature on fibers and fiber reinforced composites was reviewed and evaluated. It was concluded that, in principle, non-metallic fiber reinforced metals can have the largest strength/weight and modulus/weight ratios of all known materials, exceeding the values of those presently available by many factors, particularly at elevated temperatures. In practice, realization of the potential has been frustrated by two factors (1) thermal degradation of fiber strengths; and (2) inadequate fabrication procedures. The inadequacies of fabrication techniques employed to date were considered in detail and the requirements for an adequate fabrication technique are listed. (Author) Extensive tables give information on properties of fiber-reinforced composites and properties of inorganic, nonmetallic fibers.

Metals, American Society of, <u>Fiber composite materials</u>, Metals Park, Ohio (1965)

A review of fiber composite materials. Design criterion and data on the properties of a variety of potential high strength fibers. Theoretical analysis is supported with experiment. Discussion of progress of fiber strengthening presented.

S.A.M.P.E. Filament winding conference, March 28-30, 1961, Pasadena, California, Symposium Papers. (1961) <u>AD 254 650</u>

> Contents: Applications Design Filaments Engineering Manufacturing techniques Matrices Reliability State of the art

Materials selector issue, <u>Materials in Design Eng</u>., Annual publication about Mid-October.

> Review of years developments in materials. Data Section-Comparison of materials, nonmetallics, plastic and rubber, composite materials, others. Supplier's Literature - after each section of above headings with source of data and further literature on subject. Literature can be ordered with cards in back of recent issues only. Data presented on materials - physical, mechanical, chemical, electrical, thermal, and fabricating properties.

Oleesky, S. S. and Mohr, J. G., <u>Handbook of reinforced plastics of the S.P.I</u>. Reinhold, New York (1964).

Chapter 3, "Reinforcing materials and fillers," covers many commercial glass fibers; A, E, 475, 295, C and high modulus glasses with physical properties given. The section on asbestos gives the physical properties of several types of asbestos and the properties of various mat laminates with common resins. Miscellaneous fibers are covered with limited data. Such fibers were inorganic polymers, organic synthetics, metals, ceramics, and alloys.

Price and Wagner, "Preparation and properties of fiber-reinforced structure materials," <u>DMIC Memo 176</u>, Battelle Memorial Institute, Columbus, Ohio (1963).

> A sumary of the work of the 17 references with abbreviated data on metals reinforced by ceramic fiber, ceramics reinforced by metal fibers, and other non-metallics reinforced by metal fibers.

Rice, R. G., "Composite materials," Armed Services Technical Information Agency, Arlington, Va., Bibliography for 1952-December 1962. (December 1962). <u>AD 295 500</u>

Bibliography of 1,000 references covering the period from 1952-December 1962. No index or subdivisions given.

Riley, Malcom W., "Reinforced plastics get stronger," <u>Materials in Design Eng</u>. <u>56</u>, 99-103 (August 1962).

Report on New fibers for winding and preoriented laminates. Materials tested: Glass fiber - E glass, YM-31-A, HTS, X-994; Boron, steel. Data for composites and reinforcing agents presented.

Robbins, David L. and Epstein, George., "Thermal Erosion of ablative materials," Final summary report, (July 1961). <u>AD 274 138</u>.

An investigation was conducted to determine the erosion resistance of ablative materials. The materials were examined in the form of rocket nozzle specimens using a gaseous H-gaseous O rocket motor as the exposure facility. Environmental exposure was accomplished at a combustion-chamber pressure of approximately 500 psia and a flame temperature of 5500 F. Measurement was made of the time required for the chamber pressure to decay to 200 psia. This time period depends on the nozzle materials and construction and is a measure of the thermal-erosion resistance of the specimen. A total of 55 nozzles were evaluated; the chamber-pressure decay time varied from 0.9 to 14.9 sec. The majority of the specimens were composed of a continuous-phase resin, usually reinforced with fibers of glass, asbestos, nylon, alumino-silicate glass, high-silica glass, or graphite, and sometimes filled with ceramic or graphite particles. (Author)

Rolston, J. A., "Literature survey on filament-wound composite structures," (September 1961,) AD 268 949.

Filament winding is the newest phase in the state-of-the-art of structural reinforced plastics. This report brings together abstracts of existing literature relating to filament winding.

Rosato, D. V., "Nonwoven fibers in reinforced plastics," <u>Ind. and Eng. Chem.</u> <u>54</u>, 30 (August 1962).

Physical property data on 23 different fibers given. Metals and refractory, Synthetic organic, Natural organic, Natural inorganic, Synthetic inorganic. Also some calculations of laminate properties as related to nonwoven fiber orientation were made. Rosato, D. V. and Grove, C. S., Jr., <u>Filament Winding</u>, Interscience Publishers, New York (1964).

> Chapter 3 deals with reinforcements in the form of fibers. Description, composition and porperties of many new glass fibers given: "E", X37B,"S" YM31A, 556, 488, "C". Also strength, physical, thermal data given for metal, ceramic, inorganic, and organic fiber in common usage as reinforcements.

Ross, Jack H., "Symposium on fibrous materials," Final report, (January 1963). AD 299 030

Review of recent progress of research programs sponsored by Fibrous Materials Branch of the Nonmetallic Materials Laboratory in the area of new fiber forming substances, protective chemical finishes and coatings, and translation of fibers into flexible, high strength materials. Data supplied on many metal alloy, organic, and glass fibers.

Schmidt, D. L., "Ablative materials," Air Force Materials Symposium (June 1965) AD 463 572.

> A section on fibrous materials given limited data on a variety of possible refractory fibers used in ablative applications. Zirconia and graphite are among those discussed with some data given. Discusses many metals and ceramics as fibrous reinforcements in many possible forms; cloth, etc. Future goals outlined in the field of ablative materials.

Schulman, Stanley, "Elevated temperature behavior of fibers," (August 1961), AD 267 360

> A study was conducted to determine the mechanical behavior of fibers at high temperatures for varied periods of time. These fibers are candidate materials for decelerators, and expandable and rigid structures for satellites or space vehicles. Besides condition of extremes of temperature, these fibers will have to be capable of operation under shock, vibration, solar and cosmic radiation and related aerospace environments. These high temperature fibers were evaluated in filament form at temperatures ranging from 70 F (65% relative humidity) to 2000 F. Since fibers normally were of diameters as low as 0.0003 in., special equipment and procedures were designed to obtain their basic properties. (Author).

Schulman, Stanley, "Elevated temperature behavior of fibers," (April 1963), AD 407 103

An investigation was conducted on the behavior of fine filaments at

ambient and elevated temperatures.Polymeric and inorganic filaments of diameters ranging from 0.2 mil to 1.8 mil in diameter were included. The ambient temperature tensile strengths ranged from 68,000 psi to 501,000 psi. The effects of temperatures up to 1700°F on the properties were determined. (Author)

Schwartz, H. S., "Nonmetallic composites", Air Force Materials Symposium, (June 9, 10, 11, 1965). <u>AD 463 572</u>

A review of the more structurally useful non-metallic-inorganic reinforcements in the form of fibers developed in the past few years.

Thibault, Charles D., "Materials research abstracts," A Review of the Air Force Materials Research and Development, (1962). <u>AD 400 703</u>

> Descriptors: Abstracts, Materials, Air force research, Chemistry, Biochemistry, Acoustics, Ceramic materials, Elastomers, Electricity, Electronics, Fibers, Fuels, Antiseize compounds, Graphite, Hydraulic fluids, Coolants, Lubricants, Films, Metallurgy, Mechanical properties, Metals, Brazing, Alloys, Adhesives, Plastics, Polymers, Crystal growth, Coatings, Textiles, Fur, Leather.

B. GLASS

Baer, Eric, ed. <u>Engineering Design for Plastics</u> S.P.E. Polymer Science and Eng. Series. Reinhold Pub. Corp., N. Y. (1964) "Reinforced plastics," by Corten, H. T.

The role of fiber reinforcement and resin in controlling strength and failure of composites was studied. The strength of single filaments and bundles of filaments was analyzed using Weibull Statistics. The influence of some flaws associated primarily with fabrication was reviewed. Evidence of the influence of size effect on strength was examined. The influence of mascroscopic surface flaws was reviewed. This article presents a more sophisticated approach to reinforced plastics that will define more precisely the required properties and characteristics of composite materials.

Bastian, R. R. and Ottoson, A., "Development work on improved high modulus structural glass fiber," Final Report (June 30, 1957). <u>AD 207 884</u>

This investigation demonstrated that glasses of 16,000,000 psi and higher for Young's modulus could be composed with as much as 50% silica. Beryllium oxide gave the greatest increase in Young's modulus of any oxide introduced into the compositions shown. Extensive tables of compositions of glasses, specific gravity, Flow pt, and Elastic modulus.

Bell, J. E., "Effect of glass fiber geometry on composite material strength," ARS Journal, P. 1260-1265, (September 1961).

> Reports the results of a study to determine the effect of glass fiber geometry on composite material strength. Equations are derived for the load distribution in a composite material and also for the stress distribution. To determine the effect of fiber geometry, epoxy resin composites were analyzed for composite efficiency. The highest composite efficiency determined was 58% for filament wound fibers, and the highest fiber efficiency was 68% for cross laminated fibers. Potential fiber materials discussed with properties of Mo, W, Ta, Nb and Steel given as examples.

Bevis, R. E. and Thomas, G. L., "Glass fiber bundles, theoretical vs. actual tensile strengths," Proceedings of the 19th Annual Tech. and Management Conf. of the R. P. Div. of the S.P.I.

> The strength distribution of fiber drawn from a 36 hole laboratory bushing was determined. The strength of a bundle of fibers was calculated from this distribution. The actual strength of the coated bundle was higher than the calculated strength.

Brossy, J. F., and Provance, "Development of high modulus fibers from heat resistant materials," (March 1960,) <u>AD 236 991</u>.

> Hand-drawn fibers from 184 high-temperature compositions were tested for tensile strength, diameter size, specific gravity, ultimate tensile strength, and modulus of elasticity. Ring burners were used primarily to accomplish hand drawing of fibers.

Butler, J. B. and Senyk, B. M. "Effect of mechanical damage on glass yarn strength," <u>Materials Research and Standards</u>, (August 1965), P. 406.

The variables of glass yarn handling were studied as related to composite fabrication. The tensile tests are represented graphically with reference to variable.

Burgman, J. A. "Basic study on hollow fibers," (November 13, 1964,) AD 458 825

> Tensile strengths are presented for solid and hollow filaments formed from bushing No. 3 at temperatures from 2280 to 2440 F and drawing speeds from 1,000 to 10,000 feet/minute. Initial evaluation of a

bushing that will allow the formation of a twelve filament strand is discussed. (Author).

Burgman, J. A. "Hollow glass fibers," Proceedings of the 20th Technical Conference S.P.I, reinforced plastics division.

> Hollow glass fiber is described as a material that will fulfill the need of strength and stiffness when used as a reinforcement in filament wound structures. A theory is developed to show the advantages of hollow fibers, and experimental data is given to substantiate the theory. The physical properties and applications of this material are discussed.

Cameron, N. M. "An investigation into the effect of environmental treatments on the strength of E-glass fibres," (January 1965,) <u>AD 456 850</u>

Various experiments were carried out to determine the effects of certain environmental treatments on the load carrying capacity of virgin glass fibers. The state of the fiber surface, during and after environmental treatments, with the tensile strength used as a characteristic measure of state was also examined. The effect of strain rate on strength was also studied in the range 0.01 in/in/min. Fiber strength in vacuum was found to increase with time. Subsequent envelopment in dry nitrogen gas at atmospheric pressure and at room temperature did not influence the observed strength, but at nitrogen gas temperature below room temperature a strength resulted which was a function of both the temperature and the time of prior Maximum value was reached at liquid nitrogen temperature. evacuation. The standing of fiber samples in air decreased their strength; heating of stressed and stress-free fiber specimens led to large reductions in strength as the heating temperature was raised. G.G.

Capps, Webster and Blackburn, Douglas H., "The development of glass fibers having high Young's moduli of elasticity," (April 1, 1957,) AD 222 924

A survey was made to find glass compositions with high values of Young;s modulus of elasticity. Glasses were discovered which had moduli ranging from 17 to 21 million psi. Glasses containing little or no silica were made with extraordinarily high moduli but fiberization would probably be impossible on a commercial scale. Additions of silica made possible the fiberization of many glasses otherwise too unstable. Oxides of Ca, Mg, Be, Al and Zr were most effective in raising the Young;s modulus of elasticity; Be oxide the best. Fiber-forming equipment was especially designed to handle unusually fluid and unstable glasses. Some of the glasses had higher temperature limits of performance than commercial fibers. Glass fibers were coated with Al and 24ST Al alloy on emerging from the fiberforming process by drawing them through molten Al or alloy without physical damage from abrasion. No composites were made from metal coated fibers. An instrument was devised which makes possible easy, rapid, accurate and precise diameter measurements on fibers or wires up to about one thousandth of an inch. (Author)

Carter, J. S. and Moorefield, S. A., "New glass fibers," <u>Plastics Tech.</u> <u>9</u>, 42-4 (Sept.1963.)

Comparison of S(994) HTS, D-556, E, and Hollow glass fibers for tensile strength and strength/wt. ratio.

Cornish, R. H. and Chaney, R. M., "Glass fiber strength enhancement through bundle drawing operations," (June 1963,) <u>AD 416 134</u>

Work toward the development of a production technique for bundle drawing fibers with greatly enhanced tensile strength is described. This work includes studies on size, dependence of strength, wettingdewetting of glass fibers by metals, metallizing techniques, bundle drawing studies, bundle separation studies, and specimen fabrication. Fibers in the size range from 1 to 3 microns were drawn in bundles, separated from the bundles, and shown to exhibit tensile strength well in excess of 1,000,000 psi. Super-strength fibers produced by this method can be made in continuous lengths as opposed to "whiskers" which are limited to discrete lengths. (Author)

Cornish, R. H. and Chaney, R. M., "Glass fiber strength enhancement through bundle drawing operations," Final report, (August 17, 1964.) <u>AD 450 285</u>

> Work toward the development of a production technique for the bundle drawing of fibers with greatly enhanced tensile strength is described. This work has included studies on size dependence of strength, wetting-dewetting of metal from glass fibers, metallizing techniques, bundle drawing studies, bundle separation studies and specimen fabrication. As an outgrowth of this program, E-glass fibers in the range of from 1.5 to 5 microns have been drawn in bundles using a glassy matrix, separated from the bundles, and shown to have apparent hoop strengths in excess of 1,000,000 psi as compared to the 500,000 psi virgin strengths normally observed for conventional sized E-glass fibers. Fibers produced by this method can be made in continuous lengths as opposed to "whiskers" which are limited to discrete lengths.

Cratchley, D., Met, M., Baker, A. A., "The tensile strength of a silica fibre reinforced aluminum alloy," <u>Metallurgia</u> (April 1964.) p. 153

Tensile strengths of silica fiber and aluminum alloy composite given

as a function of Temp., fabrication method, and time.

Dunn, Stanley A. and Roth, William P. "High viscosity refractory fibers," Final summary technical report. (February 1962,) <u>AD 272 788</u>

> It was demonstrated in C glass, E glass, and silica that the viscosity of vitreous materials was increased by admixture of fine dispersions of insoluble, nonreactive, refractory materials. The effect was too large to be accounted for by volume immobilization and appeared to be due mainly to fractional forces between additive particles. Evidence was obtained that insoluble nonreactive additives, which were molten instead of solid, would also increase viscosity, provided the particle size was sufficiently fine. With vitreous reinforcing fibers aligned with the flow of heat, color in the fibers was shown to improve the thermal shielding afforded by the composite layer. Marked increases in the ablation resistance if silica through the additions of various additives appeared to be the result of cooling effects associated with the generally increased emissivities of the silica-additive mixtures. (Author)

Eakins, W. J., and Humphrey, R. A., "Feasibility study on hexagonal glass filaments,". Final report, (June 21, 1962,) <u>AD 409 538</u>

> It was demonstrated that hexagonal filaments can be made from E-glass by the preform attenuation technique. A procedure was developed for grinding flats on round rods in order to produce hexagonal preforms. Strength tests on single filaments proved the desirability of acid polishing the ground preforms prior to forming filaments. Photomicrographs on a sample of hexagonal filaments forced together in an embedding resin show perfect packing.

Edmunds, W. M., "High temp. properties of vitreous fibers and coatings," Final report (February 1960,) <u>AD 256 208</u>

> Tensile strength-temperature relations were determined for E Glass monofilaments coated with various resins and coupling agents. X-815 experimental glass fibers were also tested. Heat treatment effects were determined.

Edmunds, W. M., "Research on protection of high temperature glass fibers against flexing and abrasion," Final report, (November 1962,) <u>AD 293 828</u>

This investigation involved the utilization of suitable known chemical systems to protect glass fibers from the effects of

flexing and abrading with improved properties of strength, flexibility, abrasion resistance, and temperature resistance. X37B glass strand, the base material for this study, was evaluated as received with 640 size, with the forming size removed by heat cleaning and with the various chemical systems applied over the forming size and over the heat-cleaned glass. The strands were tested for tensile strength retention after heat treating for five hours at 500, 750, and 1000 F and for flex-abrasion resistance after a 500 F heat soak. Modulus of elasticity and relative stiffness were determined for each coated condition. Comparative measurements were made on E glass and X994 glass strand with selected finishes or sizes. (Author)

Fechek, F., and Tomeshot, R., "Reinforcements - Air Force approach to planned composite properties," Proceedings of the 18th Annual Tech. and Management Conference of the R. P. Div. of the S.P.I. (1963.)

> Data is presented for AF-815, AF-994 and Boron filaments in tabular form. Also comparative graphs of YM31A and "E" glass reinforced plastics are shown. Strength vs. temperature is given for "E", AF-815 and AF-994 glass fibers.

Frickert, Philip J., Tiede, RalphL., and others, "High modulus, high temperature glass fibers for reinforced plastics," (March 1960,) AD 251 7831

> A glass was developed which had a forty-five percent higher modulus of elasticity than the commercial glass used in the fibrous glass industry. Report describes composition research, production testing, fabrication and comparisons of YM31A glass fiber and composites produced from this material.

Gates, L. E., and Lent, W. E., "Studies on refractory fiber research," (October 30, 1960,) Contract DA-04-495-ORD-1723)

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The greatest portion of the work in this program was devoted to the development of a carbon-arc fiberizing apparatus and glass fiberizing techniques. The carbon arc provided a high-temperature source capable of melting all the refractory materials investigated. Refractory glasses were screened by evaluating the fiberizing characteristics of their compositions, formulated according to the hypotheses of glass formation. Four oxide compositions which produced high fiber yields were selected for comparison with E-glass fibers produced by the same method. Tensile-strength measurements of the fibers revealed no significant statistical difference in the strengths of the four compositions and E-glass. One refractory fiber composition was not visually affected when subjected to a temperature of 1250C for 1/2 hours. Sufficient quantities of fibers were produced from the

four selected refractory compositions for the fabrication of phenolic resin composite test specimens. Measurements of flexural strength, flexural modulus of elasticity, block compressive strength, and punch shear strength indicated that the refractory fiber-resin composites were slightly better than the E-glass-resin composites and compare favorably with chopped roving glass-phenolic resin composites, according to data reported by their manufacturers.

Gruntfest, I. J., and Dow, N. F., "New shapes for glass fibers," Astronautics (April 1961,) Vol. 6, No. 4, p. 34

Preliminary report on Hollow glass fibers used in plastic reinforcement.

Hashin, Z., and Rosen, B. W., and others, "Hollow glass fiber reinforced laminates," Final report, (August 1964,) AD 451 684

> This report presents the results of the first phase of a study to evaluate the properties of hollow glass fiber reinforced plastics for potential application in thermal and electrical as well as various mechanical functions. The overall program includes analytical and experimental studies of various composite properties. The initial phase, reported herein, consists of an exploratory treatment of pertinent material properties. The results indicate certain areas in which theory and experiment are in agreement and others in which presently unexplainable incompatibilities between theory and experiment exist. It appears that further study of fabrication techniques for the hollow fibers and the hollow fiber composite is required, as well as a more detailed investigation of composite properties. The test program was performed with three major objectives: to compare the properties of hollow fiber composites with those of similar solid fiber composites; to compare theoretical and experimental results; and to provide initial property data for use in other evaluations.

Hollinger, D. L., Kanetzky, W. G., and Plant, H. T. "Influence of stress corrosion on strength of glass fibers," <u>AD 609 985</u>

> Static fatigue tests at liquid nitrogen temperature have been completed on virgin E-glass single filaments. In these tests, fibers approximately 0.0005-inch diameter were dead-loaded in tension while at -196C and maintained at that temperature for at least 48 hours. Loads were varied within the high stress/region from 400,000 to 650,000 psi. No static fatigue failures were observed under these conditions, even though the stress range was high enough to cause immediate failure of some fibers upon load application. This is in distinct contrast to the behavior observed at room temperature in normal humidity where delayed failures occurred over several decades of time with stress level ranging from 200,000 to 400,000 psi.(Author)

Hollinger, D. L., Plant, H. T., and Mulvey, R. F., "High strength glass fibers development program," Final report, (May 20, 1963,) <u>AD 405 897</u>

> The influence of moisture on the effective strength of E-glass fibers, both as single monofilaments and when incorporated with epoxy resin into filament-wound ring structures, has been investigated. Split ring tensile tests on the composites showed a definite advantage for the maintenance of dry surroundings throughout all processing steps. Furthermore, these indicated that the presence of moisture at the time of application of high stress is of much greater significance in determining strength than mere exposure to moisture during other periods. Work with single bare fibers of E-glass has served to verify the importance of this stress corrosive reaction. By conducting tensile tests at liquid nitrogen temperature (-196°C), the reaction rate was reduced essentially to zero with a resulting change in average fiber strength from 507,000 psi at room temperature to 814,000 psi at -196°C. At the low temperature, 25% of the fibers exceeded 900,000 psi and values as high as 974,000 psi were recorded. In order to separate the effect of moisture from any possible effect of temperature alone, bare fibers were tested submerged in a very powerful desiccant solution, lithium aluminum hydride in ether, at room temperature. Strength values were obtained significantly above the normal room temperature range, although not equal to the results at low temperature.

Hood, J. C., "A quality assurance test for tensile strength of glass fiber strands, yarns and rovings," Proceedings of 18th Annual Tech. and Manag. Conf. of the R. P. Div. of the S.P.I.

Data on 801-E, HTS-E, and HTS-S glass rovings are presented. The general method of testing is discussed, and a detailed up-to-date procedure is presented.

Howard, J. S., "When to design for filament winding," <u>Product Engineering</u> 35, 102 (October 26, 1964.)

> The physical properties of glass and synthetic fibres and of the resins employed are tabulated, and the specific strength of epoxy composites is compared with those of aluminum.

Jobaris, J., "Optimum filament diameter," (January 16, 1964,) AD 428 586

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Material properties of 5-mil fiber unidirectional composites with three different resin contents have been established. As expected, the compressive modulus, parallel and perpendicular to the fiber direction, as well as the shear modulus, increases as the volume percentage of glass increases. (Author) Ketler, Albert E., Jr., "Some limitations on hollow glass fiber reinforced plastics," (1963) Presented at AIAA Launch and Space Vehicle Shell Structures Conference, April 1-3 1963, Palm Springs, Calif. <u>AD 406 654</u>

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A discussion is presented of some of the peculiar factors which place limitations on the application of hollow fiber reinforcements to composite structure. Some of these limitations are associated directly with the hollow filament and composite manufacturing technologies and can be relieved with proper quality control and shrewd process development. Other limitations are related directly to the hollow nature of the fibers and consequently, must be reflected in the design optimization of a structure. (Author)

Kies, J. A. "Composite strength as related to the properties of glass filaments and resins," Paper presented at the meeting of the Plastic Institute, London, England (March 24, 1965.)

> Theoretical analysis of composite strength as related to the reinforcing agent and matrix is discussed. Experimental data are present to substantiate theory.

Kies, J. A., "The strength of glass fibers and the failure of filament wound pressure vessels," (February 28, 1964,) <u>AD 439 217</u>

In glass-reinforced-plastic rockets such as Polaris A3 and third stage Minuteman, the current strength/weight advantage over metals has been achieved in a major degree by utilizing a new glass designated S or S-994. Examination of what is meant by the strength of glass filaments has led to a new method of characterizing the strength, especially for the purpose of exploring new glasses. Since the tensile strength is influenced by flaws and by environmental effects, the strength is therefore strongly dependent on the gage length tested and the time under load. In considering the statistical distribution of fiber strengths more than one population of flaws has been detected. The amount of damage to the fibers in such handling as the making of roving is rather severe, judged by the size or length effect on the strength of filaments. On the other hand with good design this mechanical damage is not reflected in a correspondingly large size effect in the strength of pressure vessels. (Author)

King, R. B., "The effect of heat and moisture on the tensile strength of quartz fibres," 7 p., (May, 1960,) <u>AD 241 136</u>

The tensile strength of quartz fibres of two different diameters has

been measured in the "as received" conditions and after periods of heat treatment at 100 C and 200 C under conditions of high and low humidity. The effect of varying the rate of strain was also examined. All fibres showed a loss of up to 26 percent on exposure to 100 percent R. H. but there was no marked effect of heat. On doubling the rate of strain the fibres showed an increase of nearly 40 percent in tensile strength. The smaller diameter fibres had a tensile strength of approximately three times that of the larger fibres of 2.5 times the diameter. Some suggestions are proposed to account for this. (Author)

Kinna, M. A. and Prosen, S. P., "The development of a quality control test method for glass fiber rovings and yarns," Proceedings of the 19th Annual Tech. and Management Conf. of the R.P. Div. of the S.P.I.

> Information, data, and tensile test results leading to the selection of the most suitable strand test method are discussed.

Kiselev, Boris Abramovich, "Glass fiber reinforced plastics," (May 24, 1963.) AD 410 633.

> Contents: Fibrous glass fillers Binders used in the glass plastic industry Method of manufacturing glass plastics and glass plastic articles Properties of fiberglass Primary areas for use of glass fibre reinforced plastics.

Kroenke, William J., "Flexible glass fibers," (March 1964,) AD 448 617

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This research was to develop glass compositions which, in fibrous form, would exhibit increased elongation and flexibility. Emphasis was placed on developing glass compositions patterned after the naturally occurring silicate minerals which are characterized by linear chains of SiOh tetrahedra. However, compositions based on other natural and synthetic silicates built up from other silicate structural units were also investigated. The relative flex-fatigue resistance of selected, representative glass fibers synthesized during the first year's research are reported. Additional new glasses were synthesized and tested during the second year in order to obtain even greater flex-fatigue resistances. Glass fibers were developed which had flex-fatigue resistance approximately 2-1/2 times that of E glass fibers. In addition, most of these glass fibers had increased tensile strengths and elastic moduli. (Author)

Lasday, A. H., "Development of high modulus fibers from heat resistant materials," (October 1958,) <u>AD 202 500</u>
The report deals with the development of new glass fiber forming compositions in order to produce high temperature continuous fiber monofilaments having high modulus of elasticity and strength. These properties need to be maintained at least to 1000 F. The most important objective is to produce a glass fiber whose elastic moduluus divided by specific gravity is at least 9 million psi. Extensive information given on chemical compositions of a variety of glasses or glass like minerals. Combinations of these materials were tested, to obtain high modulus glasses.

Laue, E. W., "Glasfaserverstarkte ungesättigte polyester," <u>Plastverarbeiter</u> (1961) <u>12</u>, No. 4 ,136-40; No. 5 ,202-206; No. 6, 233-38.

Reinforcements are listed with their properties. The mechanical properties, thermal properties, electrical properties and chemical resistance properties are given for the composite materials.

Layton, P. "Review of various glass filaments," (1961,) AD 254 650

Properties of various glass filaments including newly developed high modulus, high strength, and high temperature glasses presented.

Levenetz, B. "Compressive applications of large diameter fiber reinforced plastics," Proceedings of the 19th Annual Tech. and Management Conf., R. P. Div. of the S.P.I.

Standard E glass fibers with diameters between .001 and .01 inches were used in winding NOL ring specimens and tested in a compression fixture. These compressive composite stresses were higher than any reported to date on Standard E glass fiber and prove the potential of large diameter fibers for compressive applications.

Levenetz, Boris, and Holland, Herman, "Optimum filament diameters," (October 30, 1963,) <u>AD 424 113</u>

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Research on E-glass fibers with diameters between 0.001 in. and 0.010 in. was conducted to determine both their physical and mechanical properties, and their potential as structural elements in filament-wound circular specimens subjected to external pressure. It was found that 0.005-in. diameter fiber combined with a high-strength epoxy resin produced in a ring specimen an ultimate composite compressive stress over 300,000 psi. A special process was developed to protect the large diameter fibers from mechanical damage and to control the resin content. Cylindrical unidirectionally and bidirectionally

wound specimens demonstrated the strength potential of the large fiber composite by improving the buoyancy efficiency in comparison to standard E-glass roving specimens. Problem areas for further optimization of the large diameter fiber composite are outlined. (Author)

Lewis, A., and Robbins, D. L., "High-strength, high-modulus glass filaments," Part I., <u>AD 464 944</u>

> Statistical and classical composition experiments were performed to develop high-tensile-strength, high-tensile-modulus glass filaments. Nine glass compositions were developed that had tensile strengths of more than 700,000 psi and tensile-modulus values greater than 16,000,000 psi. The variability in mechanical properties of glass fibers due to fiberization variables and to sampling and testing procedures was examined. Fiberizing parameters most responsible for variation in the properties of one glass were determined. A procedure was developed for reducing sampling and testing variability. Glass fibers were examined micrographically. Development of a very high modulus filament composed of a composite of elemental boron in glass was found to be feasible. (Author)

Lewis, A., Provance, J., and Kelley, L., "Research to obtain high-strength continuous filaments from type 29A glass formulations," (December 1963,) <u>AD 437 187</u>

> The highest average tensile strength obtained on the 29-A fibers was 860,000 psi, with occasional test results in excess of 1,000,000 psi. Fibers of this quality have been drawn continuously for 21 min. Modifications of the glass composition have increased the continuous fiberizing time to more than 1 hour; however, a reduction in filament tensile strength, to 732,000 psi, has resulted. Filament modulus-of-elasticity (14,500,000 psi) was not affected by modifications to either the glass or bushing. (Author)

Lindsay, Edwin M., Hood, James C., Augenstein, D. Brent, and Sakowicz, A. "Glass reinforcements for filament wound composites," (April 1963) AD 406 701

> This is a continuation of an investigation of the process for manufacturing glass filament reinforcements for filament winding plastic composite structures. On the basis of average single filament tensile strengths of 500,000 psi compared with average glass strengths in wound composites of less than 400,000 psi, it appears that the fibers are either being degraded in the production process, or for some reason are incapable of delivering their full tensile strength in the composite. This investigation is directed

toward determining if this strength loss can be attributed to the manufacturing processes and minimizing that part which is attributable to the manufacturing process. All the data obtained to date show that strand and cylinder tensile strengths for roving are equal to those for forming stock (material that has not been through the roving process). As a result, the following major possibilities are being explored; (1) the average virgin single filament tensile strength may not be as high, or the frequency distribution curve as normal, as has been previously assumed; (2) strand and cylinder tensile strengths of material taken between the sizing applicator and the traverse may be higher than those for dried forming stock; (3) strand and cylinder tensile strengths of material taken between the collet and the drying oven may be higher than those for dried forming stock; (4) higher temperature forming may produce higher and more consistent strand and cylinder strengths. (Author)

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Lindsay, Edwin M., Hood, James C., Humphrey, Ted and Parks, C. Dean, "Glass reinforcements for filament wound composites," (August 1963) <u>AD 416 125</u>

> An investigation of the process for manufacturing glass filament reinforcements for filament winding plastic composite structures continues. On the basis of average single filament tensile strengths of 500,000 psi compared with average glass strengths in wound composites of less than 400,000 psi, it appears that the fibers are either being degraded in the production process, or fibers are either being degraded in the production process, or for some reason are incapable of delivering their full tensile strength in the composite. This investigation is directed toward determining if this strength loss can be attributable to the manufacturing process. All the data obtained to date show that strand and cylinder tensile strengths for roving are equal to those for forming stock (material that has not been through the roving process). (Author)

Lockwood, P. A., "Investigation of glass-metal composite materials," (January 1960), <u>AD 402</u> 330

> Studies of the tensile strengths of single fibers both bare and aluminum coated are reported. Behavior of glass aluminum composites are given. Research directed toward development of high temperature composites.

Lockwood, P. A., "Investigations of glass fiber-metal composite materials," (November 30, 1960), Final report., <u>AD 274 530L</u>

Metal-glass fiber composites containing 0 to 50 vol.-% glass fibers were produced of low melting metals Pb, Zn and Al metals and alloys) and high melting stainless steels. The glass fibers used with the low melting metals were similar to commercial textile glass fibers. Fibers used with the stainless steels were experimental high-temperature-resistant glass and crystalline fibers. The low melting metal composites were produced by casting molten metal around both bare and metal-coated fibers and by powder metallurgy pressing the metal-coated fibers. The casting was accomplished both by pressure casting and by contrifugal casting. The high temperature metal composites were amde by the powder metallurgical techniques of hot pressing or slip casting followed by sintering in a reducing atmosphere. The properties of greatest interest were stress-strain properties in tension and stress-rupture at room temperature and at elevated temperatures. (Author)

Lorsch, H. G. and Ketler, A. E., Jr. "Hollow glass fiber reinforced plastics," Conference on Structural Plastic Adhesives and Filament Wound Composites. Tech. Doc. Rep. No. ASD-TDR-63-396 (April 1963).

The ratios of compressive strength and stiffness to density of reinforced plastics can be increased by changing the geometry of the reinforcing fibers from solid rods to hollow tubes. Theoretical considerations, based on the assumption that compressive failure of the composite occurs by buckling of the individual fibers, predicted this result, and experimental verification has been obtained. A series of theoretical performance curves is presented showing the effects of fiber geometry and resin content on various structural efficiencies. Experimental results of hollow glass fibers reinforced epoxy resin composites are reported.

McMarlin, Robert M., Tiede, Ralph L., and Veazie, F. Munro, "High strengthhigh modulus glass fibers," (March 1965) AD 464 159

Glass fibers drawn from compositions located in the Si02-Al203-ZnO system possess tensile strengths in excess of 800,000 psi. Glass fibers drawn from compositions in the Si02-Al2-O3-MgO system have given similar results, particularly at fiber diameters in the 0.00015 to 0.00020 inch range. The modulus of elasticity of glass fibers obtained from compositions located in the Si02-Al203-MgO system can be increased to 16.0 x 10 to the 6th power psi. This is accomplished by lowering the fiber diameter to the 0.00015 to 0.00020 in. range. In the Si02-Al203-Na20 system, the fiber forming temperature, the tensile strength and the modulus of elasticity change with composition in a manner which gives a break in the composition vs. property curves at the point where the Al to Na ratio is 1. This is related to the change in the coordination number of the aluminum at this ratio, proposed in the literature. The properties measured for glasses in the $SiO_2-TiO_2-K_2O$ system do not appear to lead to as clear a concept of glass structure. Properties vary gradually with composition, with no evidence of a sharp break at any point. (Author)

Marshall, D. W., "Glass fibers for reinforced plastics," Survey of Technical Literature, Soc. Plastics Industry - Annual Tech. and Mgmt. Conference, 1961, 16th sec. 20-C, 4 p.

> Review of published literature through February 1960 including some more recent pertinent articles; articles concern characteristic properties of fibers common to all forms of reinforcements. 79 refs.

Morley, J. G., Andrews, P. A. and Whitney, I., "Strength of fused silica", <u>Physics and Chem. of Glasses</u>, Vol. 5, No. 1, (February 1964).

> Silica fibers about 30 μ in diameter were produced under controlled_conditions and had an average tensile strength of 850,000 lbs./in when tested in air at room temp. The fibers were time, temp, stress, and atmosphere-dependent. The tensile range was from 250,000 - 1,100,000 psi. No data presented on any composite work.

Morrison, A. R., Wong, Robert, and Brady, W. C. "High modulus, high strength heat resistant reinforcements, (February 1963), <u>AD 405 437</u>

A program to optimize the properties of and to develop fiberization techniques for X-994 glass resulted in the formation of fibers having an average tensile strength of 650,000 psi at room temperature and 123,000 psi at 1500 F. Fiberization techniques were developed to the extent that X-994 fiber can now be produced on a semi-commercial basis. A program to optimize the high temperature properties of X-815 glass was discontinued after considering the problems involved and the erratic data obtained from the fibers produced. Inorganic surface treatments were found to improve the tensile strength and abrasive resistance of X-994 and E glass fibers at elevated temperatures. These treatments also gave some protection to fibers from the corrosive attack of inorganic matrices. The effect of a series of proprietary treatments on the properties of YM31A, X-994, and E glass fibers were determined. (Author)

"Improvement of reinforced plastics," (October 31, 1962) AD 290 279

Work during this period has included; transverse and axial compression

tests, and bending tests of solid and hollow fiber composites; axial tension tests of single solid and hollow fibers; computation of transverse moduli and uniaxially stiffened composites as a function of plastic volume fraction and inner to outer fiber radii ratio. (Author)

"Improvement of reinforced plastics," (October 25, 1961), AD 266 277

Development of hollow-glass and quartz-fiber reinforced plastic specimens, and the evaluation of their mechanical, thermal, and electrical properties was initiated. Investigation showed that (1) manufacture of small diameter E-glass hollow fibers feasible; (2) simultaneous drawing of 35 E-glass fibers produces a uniform outside diameter but with variations in wall thickness; (3) E-glass, because of impurities, has a tendency to boil, quartz does not; (4) drawing of single, hollow filaments of quartz results in reasonably consistent geometrical dimensions.

"Improvement of reinforced plastics," (January 19, 1962), AD 272 303

Significant improvements in manufacturing of hollow glass fibers and specimen fabrication produced extremely encouraging results. Tests on improved hollow glass fiber composites showed compressive strengths as high as 110,000 psi for a strength to density ratio of over 2 million inches. Similar solid fiber specimens demonstrated strength to density ratios only as high as 1.3 million inches. These results demonstrated increased structural efficiency through the reduction of material density. Continued development of hollow fiber manufacturing processes produced excellent configuration and uniformity of single fibers. Improved E-glass composition and impurity control eliminated the boiling difficulties observed earlier, negating the need for conversion from glass to quartz.

"No title," (February 12, 1964) AD 447 950

The relative flex-fatigue resistances of 39 new glass fibers patterned after the composition of the naturally occurring pyroxene and amphibole silicates are given. Some of the new virgin glass fibers exhibited flex-fatigue resistances greater than 2-1/2 times those obtained for virgin E glass fibers. In addition, the formulation, synthesis, and testing of other new glasses, based on the initial results of the flex-testing, are reported. Single filament date, including spinning temperatures, fiber diameters, fiber densities, tensile strengths, elastic moduli, and fiber elongations, are presented for all of the new glasses. (Author)

No title, (October 31, 1963), AD 449 613

The relative flex-fatigue resistance of virgin E glass and 17 new glasses patterned after the composition of the naturally occurring pyroxene and amphibole silicates are given. An analysis of the flex-fatigue sample failure distribution is made which indicates that failure is occurring by means of two separate mechanisms. In addition 81 new pyroxene and amphibole glass compositions are formulated. Tensile strength data obtained on fibers drawn from the new glasses and the results of a screening flex-fatigue resistance evaluation are reported. (Author)

"Composite and reinforced materials," Final report(June 30, 1961) AD 441 755

Hollow glass fiber reinforced plastics are a structural composite of highest promise for use in missile and space applications. Based on strength/weight and stiffness/weight criteria, this composite is the most efficient structural material in existence. The achieved compressive strength of hollow glass fiber reinforced plastics is in excess of 100,000 psi for a density of 1.6 gr./cu cm; the compressive strength/density ratio is 1,790,000 psi, an outstanding value. These values represent a significant improvement over currently used reinforced composites. This excellent structural material also possesses a very low thermal conductivity. For many applications like cryogenic pressurized storage, therefore, the structure itself can also act as the insulation, thus producing a significant saving in weight. The low dielectric constant and the possibility of using small amounts of admixtures having desirable electrical properties give additional potential to material from the point of view of reduced radar detectability. High uniaxial strength and low weight can be achieved by diffusion and subsequent thermal expansion of carbon dioxide in polystyrene fibers, thus forming foamed strands. (Author)

"Linear structured glass fibers," (September 10, 1964) AD 461 018

A new approach to the problem of synthesizing continuous glass fibers with an oriented linear chain structure is described. Glasses have been formulated following the new approach and some of their properties are discussed. Procudures described for investigating strength and structural anisotropy in glass fibers. It was decided that E glass fibers are i^S otropic because the average strength in simple tension was determined to be equal to the average strength in simple torsion. The results of aging in a controlled air atmosphere on the flex-fatigue resistance of selected glass fibers is reported. It was found that the flex-fatigue resistance was insensitive to the length of time the fibers were aged prior to testing. Glass fibers of the recently developed A-150 glass continued to exhibit superior flex-fatigue resistance properties. (Author) "Bibliography on glass fiber and plastic laminates," (March 1961), 48 pages, 280 references <u>AD 257 58</u>7

> This bibliography contains references to unpublished reports and papers on glass-fiber and plastic laminates. It covers the period 1950 - 1960, (inclusive) the references being presented in chronological order.

"What quartz fiber can do for reinforced plastics," M/DE 52, 11-12 (November 1960).

> Short resume of electrical and mechanical properties of some silicone laminates reinforced with quartz fiber woven into a fabric. Tensile strength of fiber and flexural, compressive, and tensile strengths of composite given.

Ogden, G., "Glass fibre," Fibres and Plastics, 22, 37-41, No. 2 (1961).

The properties of glass fibre which make it particularly useful for use in plastics are given and some properties of the reinforced materials are considered briefly.

Otto, W. H., "Properties of glass fibers at elevated temperatures," (December 29, 1958), AD 271 936

> Tensile strength, inelastic yield stress, modulus of elasticity and static fatigue were measured on fibers of a high temperature candidate glass (Fiberglas X-37B) at temperatures up to 1300 F. Fiber tensile strength of Imperial Glass Corporation N-672 composition was measured up to 930 F. The effect of temperature on the modulus of silica fibers was measured at temperatures up to 1800 F. (Author)

Otto, W. H., "Relationship of tensile strength of glass fibers to diameter," Journal of the American Ceramic Society, Vol. 38, No. 3, pp. 122-124, (March 1955).

The measured strength of a glass fiber is associated with the special circumstances attending its formation. Experimental evidence is presented which shows that, contrary to generally accepted belief, the strength of a fiber does not depend on fiber diameter. When fibers of different diameters are formed under controlled, nearly identical conditions, the breaking strengths are identical within the experimental limits and there is no significant effect of diameter as such.

Otto, W. H., "Properties of glass fibers at elevated temperatures," Final report, (September 15, 1959.) AD 228 851

> The tensile strength, modulus of elasticity, and stress rupture, at 75 F and at elevated temperatures were determined for single fibers of (1) Imperial Glass Company N-672 (2) Fiberglas E, (3) Fiberglass X-37B, and General Electric Company silica. The tensile strengths became 25 percent of the values at 75F as follows: (1) N-672 at 1050 F, (2) E-glass at 1240 F, (3) X-37B at 1360 F, and silica at 2000 F. Heat treatment of the fibers caused changes in strength, yield stress, and modulus of elasticity which were both time and temperature dependent and resulted from thermal compaction or densification of the glass and surface effects. Heat treatment caused a decrease in strength at room temperature, but an increase in strength at high temperatures.

Patrick, A. J., Jr. and Hood, J. H., "Glass reinforcements for filament wound composites," Proceedings of the 20th Technical Conference S.P.I. reinforced plastics division.

> The paper discusses the experimental investigation of the fiberglass forming process to determine which processing steps degrade the fiberglass strength through mechanical or chemical damage. The strength measurements before and after each of the processing steps are reported.

Peterson, G. P., "Evaluation of a variation in glass composition of glass fibers for plastic laminates," (October 1957), AD 142 065

A variation of "E" type glass, designated 1145 "E" glass was developed by Owens-Corning Fiberglas Corporation to replace the standard "E" type glass designated 621 "E" type glass designated 621 "E" glass for use in glass fiber reinforced plastic laminates. The evaluation program included the fabrication and evaluation of laminates with: (1) 181 style glass cloth woven from both types of glass fiber, (2) Owens-Corning 136 and 139 finishes on each type of glass fiber, (3) typical polyester (Paraplex P-43) and heat resistant polyester (Vibrin X-1068) resins. Room temperature, elevated temperature, chemical immersion, water immersion, fatigue and electrical tests were conducted to provide a comparison of the properties of laminates made the two glasses. Electrical tests were also made on bulk glass samples of the two glasses. The 1145 "E" glass fiber was satisfactory and equivalent to the standard 621 "E" type glass fiber in all respects except for dielectric constant properties. At room temperature the dielectric constant at 8.5 KMC of the 1145 "E" glass was some what higher than that of 621 "E" glass, which is undesirable, but within acceptable limits. However, the dielectric constant at 8.5 KMC of the 1145 "E" glass increased far more rapidly with increasing temperatures than that of 621 "E" glass, and to an unacceptable extent. Consequently the 1145 "E" glass would not be satisfactory for radomes for radar

guidance control purposes. Since the proposed alternate composition is not acceptably equivalent to the 621 "E" glass, the 1145 "E" glass is not considered satisfactory for Air Force reinforced plastic applications. (Author)

Peterson, George P., "Engineering properties of high modulus reinforced plastics," (May 1962), <u>AD 292 311</u>

A review of data obtained on the performance and capabilities of high modulus YM31A glass for use in structural composites is presented. The data obtained include mechanical and thermal properties in woven fabric flat laminate form, including stress-rupture and fatigue data. Initial data are also presented and discussed on the properties of YM31A high modulus glass in roving and yarn form for utilization in filament wound composites. The significance and critical nature of the finish for YM31A, in both woven and non-woven form, in establishing optimum laminate mechanical properties, is also discussed. (Author)

Peterson, George P., "High modulus glass fibers for reinforced plastics," (September 1961), <u>AD 268 902</u>

> Contents: Properties of high-modulus reinforced plastics, by George P. Peterson High-modulus glass fibers for structural plastics, by Ralph L. Tiede. Processing and fabrication techniques for reinforcing plastics with YM-31A fibrous glass, by Allan B. Isham Structural efficiency of sandwich as affected by elastic modulus and weight of facing, by E. W. Kuenzi Safety precautions for handling and fabrication of high-modulus glass fibers for structural plastics, by L. J. Schafer and L. H. Miller Potential uses of high-modulus glass fibers, by P. Layton Marketing and Availability of high-modulus glass fibers, by W. M. Keller.

Plummer, Jesse, H., Lindsay, Edwin M. and others, "Glass reinforcements for filament wound composites," (November 1962), <u>AD 296-028</u>

> An investigation of the process for manufacturing glass filament reinforcements for filament winding plastic composite structures has been initiated. The investigation is directed toward determining what strength losses can be attributed to the manufacturing process and minimizing them. Equipment and test procedures for measuring single filament tensile strength, strand tensile strength, and for

filament winding and hydroburst testing three-inch-diameter, open-end cylinders were set up and checked out. Material for the initial screening experiments to determine where strength losses occur was collected and testing started, but no significant conclusions were reached. A bibliography on the strength of glass with particular reference to fibers was compiled. A review of these articles indicates that minor glass composition changes have no effect on glass fiber strength. (Author)

Provance, J. V., "Development of ultra high strength vitreous fibers and studies leading to maximum translation of fiber properties to reinforced plastic laminates," Final technical report (October 1960), <u>AD 252 190</u>

A new glass composition has been developed which, when drawn into glass fibers and combined with conventional resins, produces a reinforced plastic laminate of unusual properties. The research and development program conducted to develop this glass composition was divided as follows: (1) Development of glass compositions from which continuous glass fibers with high tensile strength, high modulus, and high temperature resistance can be drawn. (2) Development of experimental furnace to permit fiberizing the glass composition. (3) Application of a maximum percentage of the high strength fiber properties to plastic structure. The glass fiber, identified as Houze 29-A glass fiber, has a specific gravity of 2.70, a high tensile strength and a high modulus of elasticity, and will offer designers a material with greatly improved strength to weight ratio. (Author)

Provance, Jason, and Brossy, J. Frees, "Development of ultra high strength vitreous fibers and studies leading to maximum translation of fiber properties to reinforced plastic laminates," (December 1959) <u>AD 233 696</u>

Fibers were drawn during this period from thirty-four compositions. Seventy-seven fiber drawing trials were accomplished, and 248 single fiber tests were conducted. Modification of the fiber drawing take-up wheel made possible improved fiber orientation. This, in turn, resulted in the first reasonably good laminates for test purpose. MOdulus of 12.2 x 10^6 and flexural strengths up to 243 x 10^3 were recovered in the testing of thirty-nine laminates. Individual fiber tests exhibited improved tensile strengths and moduli.

Provance, J. D., "Stronger glass fibers lighten aerospace vehicles," <u>Cer. Industry</u> v. 82 n. 4, (April 1964) p. 108-11, 132, 142.

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Optimization of methods of fiberizing glass system Mg0-Al203-Si02,

which had shown desirable properties for reinforcing plastics; report covers design of orifice for fiberizing glass, method of determining optimum orifice design, and composition modifications; fibers with average strength of 686,000 psi and modulus of elasticity of 14.5-million psi were obtained; glass is designated as 29-A.

Phoenix, M. S., and Provance, J., "Development of ultra high strength vitreous fibers and studies leading to maximum translation of fiber properties to reinforced plastic laminates," (June 30, 1960) Available only at DPC offices <u>AD 241 570</u>

Forty-two fiber drawing trials were conducted to determine the capabilities of various crucibles, evaluate glass batches as to working range, and obtain fibers for testing and laminate fabrication. An improvement in the quality of the fibers was noted as a result of increasing the refinement time of the glass. Tensile strength and Young's modulus were determined by means of the Instron tensile tester. An ultimate tensile_strength of 5.50 x 10^3 psi and an ultimate modulus of 36.1 x 10^6 psi were recorded, the highest modulus obtained in this program from a continuously drawn fiber. In fiber tests, indications were that fiber strength increased with a decrease in fiber diameter. Many of the higher strengths were obtained from fibers with large diameters, ranging from 0.0007 to 0.001 inch. An increase of consistency in high flexural strengths and moduli was observed in laminate tests. The previous high flexural ultimate of 243 x 10³ psi was approached and surpassed. An average flexural strength of 235 x 10^3 psi and a modulus of 9.9 x 10⁶ psi were obtained for all laminates constructed from batch 20-A fibers and a modified epoxy resin. A high flexural ultimate of 302 x 10^3 psi was recorded for laminate No. 83 while an ultimate flexural modulus of 10.9 x 10^6 psi was noted for laminate.

Rosen, B. W. and Ketler, A. E., Jr., "Hollow glass fiber reinforced plastics," Final report (November 30, 1962) <u>AD 400 237</u>

A program to evaluate the improvement in bulk properties obtained by utilizing hollow glass fibers as the reinforcing material for glass-plastic composites was conducted. Controlled fabrication of fibers and uniaxially stiffened composites was demonstrated. Mechanical performance was defined analytically. The hollow glass fiber composites were shown to have imporved structural efficiency for applications where stiffness or compressive strength is the governing structural criterion. Extensive physical and mechanical data on the hollow glass fibers and composites made from them. Also fabrication of fibers and composites presented. Theoretical strength calculations made and experimental data to support them given. Rosen, B. W. and Dow. N. F., "Influence of constituent properties upon the structural efficiency of fibrous composite shells," NASA Contract NASW-1144, General Electric Co., Missile and Space Division, 1965.

Parametric studies of the influence of geometry and properties of fiber and matrix upon the structural efficiency. Analytical and experimental studies of elastic constants and tensile strengths of modified matrix materials are also included.

Rosen, B. Walter, "Effect of glass form on strength," (February 1964), AD 433 806

> The effect of glass fiber from on the strength of reinforced plastic composites was studied analytically. Results indicate that hollow glass fiber composites produce lower weight structures than equivalent solid glass fiber composites for applications where compressive strength or stability is the governing design criterion. Compressive and tensile applications were considered and the results of an approximate solution for the internal stresses in the vicinity of a discontinuous fiber are presented. (Author)

Rowe, E. and Thomas, G., "New high strength glasses", B. F. Goodrich Research Center, Brecksville, Ohio 1963

> A program report on an effort to develop glasses with fiber tensile strengths higher than commercial "E" glass. Composition studies were made. Methods of testing and mechanical properties were given.

Runck, R. J. and King, B. W. "Glass fiber for solid-propellant rocket motor cases," OTS PB 171629 DMIC memo 110, (June 6, 1961).

Manufacturing Processes, compositions, properties of E type glass. Properties for various high modulus glasses given: Imperial N 672, YM31A, X-37B, Bjorksten Silica. Discussion of performances in various composites presented.

Schmitz, G. K. and Metcalfe, A. G., "Exploration and evaluation of new glasses in fiber form," Solar, Division of International Harvester Company, San Diego Progress reports as follows: Final report (first contract year), (April 12, 1963,) Contract NOnr 3654(00)(X)

The length effect on fiber strength was studied for E-glass and 994 glass fibers. These fibers were primarily from strands. Tensile test results confirmed the linear log strength-log length relationship previously established and that fiber strength increases with

decreasing length. At short fiber lengths (below 0.5 cm), the strength-length curve showed a change in slope due to the presence of mixed flaw propulations, which were indicated in bi-modal failure distributions in the vicinity of the slope change. This change in slope was not anticipated in an earlier concept developed for failure prediction on the basis of the length effect. A tentative revised model is suggested but must await confirmation by experiments with controlled fiber damage. Some comparative tests on strand strength showed a strength reversal with decreasing test length. The reversal could be related to the effect of fiber collimation. Comparison of fiber properties of the different glasses on the basis of strength, weight, Young's modulus, surface damage, and fiber length showed that 994 fibers are superior to E-glass by a factor of approximately 1.7 for longer fibers.

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Schmitz, G. K. "Exploration and evaluation of new glasses in fiber form,"
Final report (Second contract year), Solar, Division of International
Harvester Company, San Diego, (March 25, 1964), Contract NOnr 3654
(00)(X)

The resulting strength-length curves are similar in shape to the strand fiber curves indicating that mixed flaw populations were present also on freshly drawn fibers, but to varying degrees depending on drawing conditions. Analysis of the corresponding failure probability plots provided detailed information on the characteristics of the different types of flaws as well as on the proportional amounts present at the various fiber lengths. The existence of mixed flaw populations limits the application of single exponent failure probability functions, such as the Weibull, to certain gage lengths. The work performed during the second contract year was to establish the first year's findings on a firmer basis. This goal has been achieved by tensile strength measurements on virgin E- and S-glass at approximately the same gage lengths previously used for fibers from E- and S- glass strands. Resistance to mechanical damage was found to be similar for the two glasses and it was concluded that surface defects due to stranding were similar with respect to both severity and density.

Schmitz, Gunther K., "Exploration and evaluation of new glasses in fiber form," Final summary report, (May 20, 1965), AD 464 261L

> Descriptors: (*Glass textiles, Fibers), (*Ceramic fibers, Glass), Fibers (Synthetic), Mechanical Properties, Fatigue (Mechanics), Humidity, Moisture, Failure(Mechanics), Stresses, Strain (Mechanics), Corrosion, Tensile properties, Test equipment, Statistical analysis.

Schwartz, H. S. "Non metallic composites," Air Force Materials Symposium, (June 1965), AD 463 572 「「「「「」」」」」」

A short review article on recent developments in fibers for reinforcements. Data given for some glasses and Zirconia fibers and test methods are discussed for composites.

Shand, E. B., "Fibrous glass," <u>Glass Engineering Handbook</u>, Section 4, pp. 375-426, 2nd ed. McGraw-Hill, New York.

Properties of glass fiber and reinforced plastics given in tables and graphs. Parameters affecting strength of finished composite discussed. Tests on reinforced plastics included.

Siefert, R. F., "Hollow glass fibers," il. Glass Ind., 44, 321-24

A short introductory report on the conception, production and properties of hollow glass fibers with come preliminary data and suggestions for applications.

Smith, Ronald Henry, "Glass-fiber-reinforced plastic structures," (1964), N65-18533

> A new, high-strength glass fiber with HTS finish, when used as filament-wound or fabric reinforcement with an epoxy-resin system, results in a glass-fiber-reinforced plastic (GFRP) material with superior strength characteristics. This glass fiber, S(994), when used in a new loom-weaving process that fabricates truss-core sandwich construction and when cured with an epoxy-resin system, results in a new lightweight, low-cost sandwich that promises extensive use in light aircraft structures, powerboat hulls, deepsubmersible structures, and other application. Some of the most noted advancements in the state of the art of GFRP materials and construction that can be attributed to space-age development programs are the development of filament-wound pressure vessels, advanced orthotropic-material optimum-design techniques, loomwoven-fabric sandwich structure, and development of S(994) glass D.E.W. with an HTS finish.

Soltis, P. J., "Evaluation of glass fibers produced by bundle drawing operations," (November 19, 1963), <u>AD 424 211</u>

Two samples of glass fibers produced by bundle drawing operations were evaluated and found to have fracture strength in the range 0.07 to 0.20 X to the 6th power psi corresponding to fibers in the size range 1.5 - 4 microns. The relatively low strength of bundle drawn glass fibers, 0.07 to 0.20×10 to the 6th power psi as compared to commercially available glass fibers having strengths approaching 0.50 x 10 to the 6th power psi, is probably due to the presence of small transverse cracks or surface imperfections introduced either during processing or during handling. (Author)

Stevens, D. W., Otto, W. N., and Chia, C. Y., "Potential of filament wound composites," Final summary report (May 1963), <u>AD 405 536</u>

> Studies of single-fiber strength retention were pursued with the aims of preserving the intrinsic strength of glass in the composite and determine the causes of strength loss in the composite. Date presented on fiber with variation of treatment. Data on composites with variation of parameters given also. Mathematical analysis developed for composites.

Stevens, D.W., Otto, W. H., and Chia, C. Y., Final summary report, (May 1963), AD 405 536

> Studies of single-fiber strength retention were pursued with the aims of (a) preserving the intrinsic strength of glass in the composite and (b) determining the causes of strength loss in the composite. These investigations included the effects of coating methods, coupling agents, amine and anhydride hardeners, cure schedules, tension during resin cure, stress-rupture, fiber collimation, long-term humidity cycling, dual resin coatings and measurements of glass fiber-resin interfacial shear strength. Indications are that a coupling agent provides better adhesion of the resin to the glass and, therefore, should improve composite strengths. Experimental techniques for evaluating interface shear were studied. Futher development is required. A dual resin system was evaluated. It appears to reduce stresses around discontinuities but does not improve interfacial shear strength. (Author)

Tomashot, R. C., "AF-994-A superior glass fiber reinforcement for structural composites," Directorate Materials and Processes, Wright-Patterson Air Force Base, Ohio, 1963, Tech. Doc. Rept. ASD-TDR-63-81.

Report on properties of AF-994 glass fiber given and also properties of laminates reinforced with both woven and non woven reinforcement.

Vondracek, C. H., Moberly, L. E., and Berg, D., "Fiber reinforced boron phosphate structural composites," (January 1964), <u>AD 447 823</u>

New formulations for making boron phosphate were studied but no reduction in porosity of asbestos-reinforced laminates was realized. The compatibility of BPO₄ with a number of fiber materials or potential fiber materials was investigated. Incompatibility

did not appear to be the limiting factor in the application of BPO₄ as a binder for these materials. Surface coatings for glass were investigated and found to improve the shear strength between glass disks bonded with boron phosphate. Improvement in the strength of boron phosphate bonded X-994 glass fiber composites has been achieved by low pressure molding. The heat cleaning of both silica and X-994 glass fibers at 500° C results in a high loss of tensile strength of the fiber. An improvement in strength and a major reduction in moisture sensitivity of BPO₄ asbestos laminates resulted from post impregnations with certain metal phosphates. Mechanical property data for both BPO₄-glass and BPO₄-asbestos composites aged at room temperature and elevated temperatures are presented. A number of other reinforcements were evaluated and had good compatibility iwth boron phosphate but did not produce high strength laminates.

Vondracek, C. H., Moberly, L. E., and Berg, D., "Synthesis and formulation or inorganic bonded-inorganic fiber reinforced non-metallic structural Materials," (January 1963), <u>AD 412 707</u>

> An examination of the physical and chemical reactions and properties of boron phosphate and the boron phosphate-asbestos composite was made. Processing parameters involved in making boron phosphate bonded asbestos composites were determined. Flexural strength of these composites were determined both after aging and at temperatures up to 800 C. A study was made of the effects of boron phosphate on other reinforcements, boron fibers and glass fibers. Composites were constructed of boron phosphate and glass fibers and tested for flexural strength. An investigation was initiated on the use of coupling agents to improve the boron phosphate bond to glass fibers. (Author)

Welter, J. T., Wagner, H. C. and Broda, S. J., No title, (January 15, 1954) AD 222 918

> This report includes: Owen-Corning Fiberglas Corp., Newark, Ohio. Strength of Glass Fiber, by F. O. Anderegg. Oct. 14, 1938, 9 p. incl. illus. table, refs. Reprint from Industrial and Engineering Chemistry, <u>31</u>, 290-298, March 1939.

Glass 153 was further refined to Glass 153E. The glass was melted at 2600 F. in clay cricibles and drawn at speeds up to 3 mi/min. The glass was easier to make and as easy to draw as Glass E, with the melting, forming equipment, and procedures required being the same or similar. The first glass made and tested gave tensile strengths up to 706,000 psi with the average being 443,040 psi. Glass E similarly tested gave strengths up to 445,000 psi, with the average being 376,000 psi. Efforts to bond Al powders through superficial oxide surfaces failed at temperatures below the actual melting temperature of Al. Above the melting temperature, some bonding did occur. Reducible oxides such as TiO₂, SiO₂, etc., formed Al2O3 in situ in a desirable form and apparently with some degree of oxide-metal bond. (Author)

Whitehurst, H. B., Michner, J. W. and Lockwood, P. A., "Investigation of glass-metal composite materials," 6th Sagamore Conference (1959), <u>AD 233 158</u>

> Investigation as to the strength reduction in coating glass fibers with aluminum was conducted and results tabulated.

Whitehurst, H. B., "Investigation of glass-metal composite materials," AD 141 173

> Glass and metal composite materials have demonstrated tensile strengths from 16,700 to above 37,500 psi at room temperature without excessive decrease in strength as the testing temperature is increased. Three general methods for forming the composite materials were employed: (1) precoated glass fibers and flakes were compacted by the application of heat and pressure; (2) glass fibers or flakes were mixed directly onto molten metals; and (3) glass fibers or flakes were introduced into a matrix of a metal having a relatively high melting point, and further attentuation of the glass was accomplished by hot working the composite above the softening point of the glass by rolling, forging, and other similar methods. Al, Zn, Pb, Cn, and Wood's metal were used in exploratory experiments with a commercial borosilicate (E) glass and somehigh modulus glasses furnished by NBS, but the major effort was directed toward combining glass fibers with Al since a low density material with good tensile strengths at temperatures up to 1000 F was desired. Auxilliary studies of the tensile strengths of individual glass fibers coated with Al established that fibers which were coated during the forming process were about one-fifth as strong as bare glass fibers formed under similar conditions. (Author)

Young, R. E., "The fiberglass filament wound structure, a correlation of continuing studies," P. 174-189 (April 1963), <u>AD 408 781</u>

Qualitative comparison of E-HTS, X-994, YM31A glasses in the application of filament winding is presented. The effect of finishes on tensile strength is given in tabular form and data on pressure cylinders appears to be reproducible.

Yurenka, S. B., Duft, B. L., and Chia, C. Y., "Potential of filament wound composites," Narmco Research and Development Corp., San Diego, Calif., NARM 623 FR, Final Report, (March 1962), Contract NOw 61-0623-c.

A study was made on the effect of coupling agents and resin coatings on the tensile strength of single E-glass fibers. It was found that Volan E and A-1100, when properly applied to the virgin fiber, do not cause any strength loss in the fiber. The resin coating alone, without any coupling agent, appears to protect the fiber in water immersion as well as the combination of a coupling agent and the resin. The A-1100 appears to improve the fiber strength of 500F and to be more heat resistant than the chrome complex Volan E. Mathematical expressions were derived to predict the probable behavior of composites under loads of tension, compression, and bending. Experimental verification on photoelastic study models showed good correlation between theory and experiment.

Zak, A. F., "Physicochemical properties of glass fiber," 288 p. refs. transl. into english of the book <u>Fiziko-Khimicheskiye svoystva</u> <u>steklyannogo volokna</u>, (1962) p. 1-224, May 1963. N64-17465.

> This book gives concise information on the existing methods of producing glass fiber for various purposes. The most important properties of glass fiber and the objects produced from it, and the dependence of these properties on factors of various natures are presented in detail. The existing theories on the structure of glass fiber are critically discussed. Author

C. METALLIC

Achbach, W. P. and Runck, R. J., "An evaluation of materials for rocketmotor cases based on minimum weight concepts," <u>AD 273 297</u>

> Comparisons are made between the properties of different materials, including steel, titanium, and glass, as potential materials of construction for rocket-motor cases. These comparisons are useful in two situations in which the highest strength-to-weight ratio is wanted; where buckling is not critical; and where buckling is critical. High-strength steel wire-resin composites may have the potential to exceed the properties of monolithic steel plate in some rocket-motor cases. As compared with monolithic titanium plates, however, high-strength titanium wire does not have sufficiently greater strength to indicate any advantages in rocket-motor cases. Comparative data given for filaments.

Baskey, R. H., "Final report on fiber reinforcement of metallic and nonmetallic composites," (July 1963), <u>AD 417 390</u>

> Manufacturing techniques were developed and successfully applied to produce improved strength-to-weight values in metallic composite materials for structural applications in mission-oriented systems. Hot-pressed composites with matrices of cobalt, cobalt alloy L605, nichrome, and stainless steel, reinforced with 10 to 30 volume percent of 10, 5, or 2 mil tungsten wire, produced tensile strength values up to 2 times greater at room temperature and up to 17 times greater at 2000° F than obtained for the unreinforced matrix material. At 2000° F the tensile strengths of cobalt, cobalt alloy L605, and nichrome sheets containing aligned tungsten wires were approximately 6 times, 1.6 times, and 2 times greater, respectively, than for pure cobalt, L605, and nichrome sheets. Random wire patterns strengthened the metal matrices at 2000° F, but to a lesser degree than aligned wires. The modulus of elasticity of sheets containing random wires was about the same as pure matrix material at room temperature. The modulus of elasticity of cobalt alloy L605 or nichrome sheets containing aligned wires was approximately proportional to the wire content. The moduli of elasticity decreased as the temperature increased.

Baskey, R. H., "Fiber reinforcement of metallic and nonmetallic composites," (August 10, 1962), AD 283 506

> Parameter studies have indicated that powder metal bars of cobalt were reinforced by 8.6 volume percent 10 mil diameter continuous tungsten wires. The room temperature strength was increased from 48,500 psi to 85,000 psi by adding tungsten wires. The elevated temperature (2000F in vacumn) short time tensile strength was increased from 2,700 psi for cobalt to 23,600 psi by adding 18 volume percent continuous 10 mil diameter tungsten wires to a cobalt matrix.

Baskey, R. H., "Fiber reinforcement of metallic and nonmetallic composites," (May 1962), AD 275 565

> Initial parameter studies have indicated that powder metal bars of stainless steel, type 316, were reinforced by 14 volume percent 10 mil diameter continuous tungsten wires. The room temperature tensile strength of the stainless steel was increased from 40,000 psi to 48,000 psi by adding tengsten wires. Nichrome V (80 Cr - 20 Ni) matrices were not reinforced by either tungsten or molybdenum wires. Tensile properties of unalloyed tungsten wire at elevated temperatures given.

Baskey, R. H., "Fiber reinforcement of metallic and nonmetallic composites," (December 1962), <u>AD 297 043</u>

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The elevated temperature (2000F) short time tensile strength of cobalt was increased from 2,700 psi to 23,700 psi by reinforcing the cobalt with 18 v/o of 5 mil continuous tungsten wires. This strengthening by 5 mil wires was equivalent to 89 percent of theoretical strengthening and was comparable to that attained by using the same quantity of 10 mil tungsten wire.

Baskey, R. H., "Fiber reinforcement of metallic and nonmetallic composites," (February 1962) 293 abstracts <u>AD 274 379</u>

> This survey summarizes the progress in fiber metallurgy. Whiskers possess the maximum strength but are not available commercially. The survey confirms that of the three types of fibers available, i.e., glass, ceramic, or metal, only glass fibers are used extensively to reinforce material. Metal fibers have a limited commercial application as a reinforcing agent. In the instances where metal fibers are used to reinforce metals, the processes are still laboratory or pilot plant size. Data on these fibers given.

Baskey, R. H., "Fiber-reinforced metallic composite materials," (May 26 1965), AD 464 137

This report describes studies on the compatibility of various high strength wires with nickel, nickel alloys; tensile and stress rupture data on extruded titanium composites and tensile data on hot-pressed nickel and titanium composites. Metallographic and microhardness studies of nickel alloy composites after thermal treatments of 100 hours at 2000 F indicate that Waspaloy and Udimet 700 matrices exert the least influences on wire recrystallization. TZM and KW molybdenum wires embedded in titanium alloys exhibit less recrystallization than commercial grade molybdenum wire after thermal treatments at 800 and 1200 F for 100 hours. Tensile and stress rupture data on extruded titanium 6A1-4V alloy containing 19 vol-% of wire exhibit an improvement over the unfibered alloy. Tensile data are also presented on hotpressed nickel alloyand titanium alloy containing continuous wires. (Author)

Bohanek, E., and Kessler, H. D., "Development of fine diameter, high strength Ti-13V-11Cr-3Al wire," final report November 15, 1961, Contract Now 60-0188 (FBM).

The results of this program indicate that 0.004 inch diameter

Ti-13V-11Cr-3Al wire can be produced to ultimate strength levels of about 285 Ksi. Occasionally, the 300 Ksi ultimate tensile strength objective was met, but not predictably.

Coplan, M. J., Freeston, W. D., Jr., and Platt, M. M., (February 1964), AD_447_821

> The objective of this program is the development and evaluation of fibrous structural materials exhibiting good flexibility and high strength at temperatures from cryogenic to 2000 F. The weight, permeability, flexural rigidity, wrinkle recovery, tensile properties, tear strength, and fold endurance at 70 F of a fabric woven from a multifilament yarn composed of 0.0005-inch-diameter nickelchromium alloy wire are given. The high-speed impact properties of a metal yarn, an HT-1 yarn wrapped with metal yarn are also given. A preliminary investigation of the joining of panels of metal fabric by sewing is discussed. The properties of coreless cord braided from half-mil and one mil wire are given. Metal yarns textured by the various textile texturing processes are evaluated. A preliminary investigation of metal filaments blended with other fibers is discussed.

Cox, J. E. and Veltri, R. D. and Shulze, C. E., "Exploratory investigation of glass-metal composite fibers" Final report, (May 7, 1965), AD 615 098

> An investigation has been conducted to develop a process for the production of continuous metal filaments by casting of molten metal as a core material in a glass capillary as the capillary is continuously formed from glass tubing. Beryllium and copper have been given major consideration as the core metals; chromium, aluminum, iron and tin have been given minor attention. Limited data given on finished filaments.

Cox, John E., Veltri, R. D., and Schulze, Charles E., "Exploratory investigation of glass-metal composite fibers," (July 13, 1964), <u>AD 449 761</u>

> This report is a summary of preliminary investigations of a process for the production of continuous lengths of 1 micron diameter wire. The process involves the casting of molten metal as a core material in a glass capillary as the capillary is continuously formed from glass tubing. To date, beryllium and copper have been given major consideration as the core metals; chromium and aluminum have been given minor attention. Short lengths of berylliumin-glass composite fibers have been produced by a hand-drawing technique. Initial attempts to form such composites in a continuous process have been unsuccessful due to the reaction between beryllium and glass at high temperature. Limited experiments to reduce the

reaction time or to slow the rate of reaction by atmosphere control have not shown appreciable beneficial effects. Initial work with copper in glass has been chiefly concerned with studying the variables in the formation process. Copper core diameters below about 16-20 microns, continuity has not been obtained over any appreciable length. (Author)

Cratchley, D., "Factors affecting the UTS of a metal/metal-fiber reinforced system," <u>Powder Metallurgy</u>, (1963) No. 11 p. 59

> A composite of stainless-steel fibres in an aluminum matrix has been chosen as a model fibre-reinforced system, to assess the effect of salient parameters on the ultimate tensile strength of the composite. Data on fiber strength and composite strength given . Theoretical explanations of data made.

Dean, A. V., "The reinforcement of nickel-base alloys with high strength tungsten wires," (April 1965), <u>AD 464 771</u>

> The reinforcement of conventional Ni-base alloys with fine W wires to improve high temperature strength was investigated. Fabrication by a casting process in which molten Ni-base alloy infiltrates a bundle of W wires was preferred. Considerable shorttime tensile and creep strength data were obtained at temperatures up to 1100 C for composites containing up to 50 vol-% reinforcement using wires of diam ranging from 0.005 to 0.070 in. Substantial strengthening was achieved at temperatures above 900 C. The composites exhibited poor impact strength below 200/300 C due to the brittleness of the W wires at these temperatures. Fatigue testing up to 500 C showed that the addition of W wires was very beneficial. The oxidation and wire-matrix interdiffusion characteristics of W. wire reinforced Ni-base alloys were studied. Theoretical considerations on the mechanical properties of wire reinforced Ni-base alloys are discussed. (Author)

Dean, A. V., "Development of composite materials for service at medium and elevated temperatures," <u>Applied Matls. Research</u> V 3, n. 4, (October 1964) p. 195-202.

> Composite materials consisting of high strength fibers and wires to reinforce conventional alloys permit application at temperatures considerably above present levels; nickel, tungsten, columbium and molybdenum alloys are discussed; dispersion hardening is considered inadequate for exacting applications such as gas turbine rotor blades. 33 rfs.

Delmonte, J., Metal filled plastics, Reinhold (1961).

Applications of metal fibers and filaments to plastics is reviewed in a qualitative manner. Fabrication and uses discussed.

Everling, W. O., "Super-high strength wire, a component of metallic composites," Proceedings of sixth Sagamore Ordnance Research Conference, p. 94-115 (August 1959), <u>AD 233 158</u>.

Presents physical property data on fine steel wires (0.005-inch diameter) from room temperature to 500 F. Commercial grade .004-inch diameter wire has a tensile strength of 450,000 psi. Experimental wire has been produced with strengths up to 700,000 psi.

Fechek, F. and Hennessey, M., "Boron-fiber-reinforced structural composites," (1963), Presented at the AIAA Launch and Space Vehicle Shell Structures Conference, 1-3 Apr 63, Palm Springs, Calif. <u>AD 406 660</u>

> Calculated strength properties and weight which a typical filament wound rocket motor case would possess if it were constructed with boron and AF-994 fibers have established the potential of this material. Additional research is being conducted to enhance this potential even further, since the maximum possible strength levels of these filaments have been achieved. The presently attained strength values are only approximately one-twelfth of the theoretical strength. The expected increases would permit thinner wall sections to be used which could result in additional weight decreases in the polar wrap in excess of 10%. Over-all, it is considered that the boron fiber used as a reinforcement in structural composites will make possible the use of these structures in applications which previously excluded materials of this general type. (Author)

Forester, R. H., and others, "Reinforcement of epoxy resins with metal fibers," <u>Mod. Plastics</u>, <u>40</u>, 117=18 (April 1963).

> The variables of fiber volume percent, fiber length, fiber cross section and two epoxy resin systems were examined for aluminum, steel, and copper fibers. Test for tensile strength, compressive strength, compressive modulus, and thermal conductivity were conducted.

Gorton, C. A., McMahon, C. C., and Rizzardi, J. A. "Ultra-fine high temperature, high strength metallic fibers," Final report (August 1962), <u>AD 287 443</u>

Eight superalloys of A-285, Elgiloy, Hastelloy B., M-252, Rene 41, Udimet 500, Udimet 700, and Waspaloy were processed to ultra-fine fibers of approximately 0.001-inch diameter or less and evaluated for drawability. The room-temperature mechanical properties of the annealed fine fibers are presented. The effect of cold reduction on the mechanical properties is also included. The Elgiloy and Hastelloy B alloys processed more readily with less die wear than the remaining alloys. The tensile strength at room temperature of each of the alloys, except A-285 was within the range of 160,000 to 220,000 psi as solution heat treated. Alloy A-286 tensile strength was approximately 100,000 psi. The alloys in order of decreasing strengths were U-700, Rene 41, Hastelloy B, Waspaloy, U-500, M-252, Elgiloy, and A-286. Multifilament yarns composed of 7, 19, and 37 filaments of Elgiloy and Rene 41 were successfully processed to less than 0.003-inch diameter when sheathed with alloy Chromel-C, but the sheath could not be removed without damaging the fibers. High-temperature tensile tests of the sheathed yarn at 1600° F, 1800°F, and 2000°F, in air and argon.

Gross, A. G., Jr. O'Rourke, R. G., and Beaver, W. W., "Fabrication of beryllium wire," Final report (November 29, 1959), AD 272 073.

> An investigation was made to develop techniques for the fabrication of beryllium wire and to produce 100 ft. of this wire. Standard wire-drawing equipment was altered to allow a working temperature of 850° F. Instrumentation was devised to allow measurement of draw force, temperature, and draw speed. The wire-drawing process was analyzed mathematically, and this analysis formed the basis of development for the drawing technique. True-stress-true-strain tensile microstructures quite similar to extruded structures. X-ray diffraction studies revealed a continuous development of (210) fiber axis as the wire diameter was decreased. Tensile testing of the drawn wire showed that relatively high strength was attainable but substantial ductility at room temperature was present only when recrystallization reactions occurred.

Gross, A. G., Jr. O'Rourke, R. G., and Beaver. W. W., "Fabrication of beryllium fine wire," TR200-228 Final report (April 1961), 62 p. NOas-60-6108-C

> Techniques were developed for the fabrication of beryllium (S-200B) fine wire and to establish optimum mechanical properties. Various lubricants and drawing temperatures were evaluated. Certain metal-sulfur compounds in conjunction with graphite were found to be the best sold-film combinations. An oil was found which would lubricate at temperatures up to 600°F in room atmosphere. Wire diameters as small as 0.00477 inch were produced with each lubrication system. Reductions in area as high as 99.41 percent

without intermediate stress relief were made at 800°F, using the solid-film lubrication. A pickling technique was found which enhanced the bending properties of beryllium wire. The bend testing showed beryllium wire of commercial chemical purity to be ductile so long as the applied stress was rotationally asymmetrical about the wire axis. Tensile testing revealed that strength levels in the vicinity of 200,000 psi were attainable and that tensile strength was a function of reduction ratio from the last anneal and of a drawn diameter but was not a function of pickled diameter. An appreciable amount of twinning, (1012), was produced as a result of drawing and these twins were not always destroyed by heat treatment. Little or no random recrystallization was produced by the annealing treatment employed.

Gross, A. J., "Work hardening of beryllium wire," <u>Trans. Amer. Soc.</u> <u>Metals</u> (1964) <u>57</u>, 355.

Tensile strengths of various diameter Beryllium wire determined.

Hodge, W., "Beryllium for structural applications, a review of the unclassified literature," 1958-1960, (May 1962), <u>AD 278 723</u>

Reports of government supported research on the physical and process metallurgy of beryllium that were received at DMIC during the period 1958-1960, inclusive, are summarized, together with some reports and publications of foreign authors. No proprietary or classified information is included. In addition, reference is made to some of the more important writings on health hazards and safety procedures that appeared during the period covered.

Holladay, J. W., "Titanium alloys for high-temperature use strengthened by fibers or dispersed particles," (August 31, 1959), AD 227 015

Available data are reviewed on heterogeneous structures of titanium in which the titanium matrix is reinforced by a dispersion of metal fibers or particles. Fiber reinforcement offers promise for improving the short-time strength and modulus of elasticity in the temperature range of 1000 to 1400 F. It has been demonstrated that titanium can be strengthened by dispersion hardening and by fiber reinforcement, although the best conditions have not yet been fully established.

Jech, R. W., Weber, E. P., and Schwope, A. D., "Fiber-reinforced titanium alloys," <u>Reactive Metals</u>, edited by W. R. Clough, Interscience Publishers, pp. 109-119,(1959) Clevite.

Reinforcement of titanium and titanium-6 A1-4V matrices with

20, 30, and 40 volume percent molybdenum fibers enhanced the elevated temperature properties of the matrix material. Tensile strength, strength-to-weight ratio, modulus of elasticity, and modulus-to-density ratio were found to be superior to alloyed titanium, up to 1400 F.

Jech, R. W., McDaniels, D. L., and Weeton, J. W., "Fiber reinforced metallic composites," (August 19, 1959,) AD 161 443

> It was possible to produce fiber reinforced composites by both powder metallurgy methods and liquid phase sintering. Furthermore, it has been shown that composites having higher room temperatures and elevated temperature tensile strength than the matrix material can be made using reinforcing fibers. Reinforcing fibers also had a marked effect on the stress-rupture life of the matrix. The studies of fracture mechanisms have shown that in both continuous and discontinuous fiber composites consisting of mutually insoluble materials, the tensile strength was directly proportional to the amount of reinforcement present for a wide range of fiber to matrix compositions. Of even more importance, discontinuous fiber composites can be produced which have tensile strengths equal to strengths observed in continuous composites.

Johnson, D. E., et al, "Metal filaments for high-temperature fabrics," Final report, (February 1962), <u>AD 276 292</u>

> This research was primarily on the oxidation and tensile properties of 0.5-to 5.0-mil filaments of three super-alloys (Elgiloy, Rene 41, and Inconel 702) and two refractory metals (tungsten and Molybdenum) in the 1500° to 2000° F temperature range. The oxidation data for the refractory metals are for ultra-short-time durations, up to 100 milliseconds. Preliminary creep rupture data are also given for the above three super-alloys, along with some data for Karma and Nichrome V. Attempts to develop very thin (0.5- to 0.1-mil oxidation-resistant coatings for molybdenum and tungsten by the use of various plating techniques are also reported. One-half-mil fibers of superalloy wire were woven into fabrics of various constructuion; the influence of fabric construction on permeability and aerodynamic drag (at Mach 4.8) is discussed. The literature pertaining to heat transfer to single cylinders in hypersonic flow is reviewed, and sample calculations of equilibrium temperature are made to point up areas where data are deficient. This report also presents the results of preliminary research on three new techniques for forming fine filaments without the use of a diamond die.

Kaplow, Roy, Peck, John F., Smith, Frank T. J. and Thomas, David A., "Substructure and mechanical properties of refractory metal wires," (May 1964) <u>AD 602 695</u>

> Polycrystalline columbium wires of three levels of interstitial impurity content show steady work hardening in wire drawing to the highest strain obtained, a true strain of 5.1. This behavior contrasts to fcc metals, which show saturation of work hardening at true strains greater than 2. During drawing of random texture columbium, a fibrous microstructure and the (110) wire texture continually develop. If the material has an initial (110) texture, however, the texture diminishes because of deformation band formation, and subsequent recrystallization produces near-random orientation. Swaged wire also shows continued work hardening and develops a cylindrical texture at high strains. Torsion stressstrain curves for both polycrystalline and single crystal columbium have been obtained, and the effect of strain reversal was studied. A region of work softening was observed in both as-drawn wire and single crystals after reversal of comparable forward strains in drawn wire annealed at 700° C or above prior to testing. Author

Kinna, Marlin A., "Some properties of beryllium wire reinforced NOLrings," Final report (May 24, 1965), AD 465 419

> Beryllium wire reinforced plastics composite NOL rings exhibited a tensile modulus of 22.6 x 10 to the 11th power dynes/sq cm (31,300,000 psi). This value represents a four-fold increase over typical modulus values for glass fiber reinforced rings. An ultimate tensile strength of 11.0 x 10 to the 9th power dynes/sq cm (159,700 psi) which is equivalent to the strength levels obtained for glass reinforced NOL rings, was also measured. Samples prepared with a combination glass fiber-beryllium wire reinforcement had significantly higher tensile and compressive moduli than all-glass fiber reinforced specimens. (Author)

Koppenaal, T. J. Parikh, N. M. "Fiber-reinforced metals and alloys," Final report (May 9, 1962), <u>AD 276 620</u>

> Mechanical properties of several different composites made from fibers of .0002-.001 in. diameter were investigated. Best results, aluminum matrix and tungsten fibers and silver matrix and type 430 stainless steel. Graphite fibers were found relatively poor reinforcing material.

Liu, Tien-Shih; Stowell, Elbridge Z., "Parametric studies of metal fiber reinforced ceramic composite material," Final report (January 26, 1961), <u>AD 252 916</u>. The purpose of this program was to conduct a theoretical study of the various parameters which affect the mechanical characteristics of metal fiber reinforced ceramic composite materials. In addition, a survey of properties of potential fiber and matrix materials was made so that promising combinations needing intensive investigation can be defined in specific terms. Certain room temperature mechanical behavior of metal fiber reinforced ceramics (MFRC) were predicted using an inclusion concept and from geometric, elastic, statistical distribution and plastic strangth considerations. Certain elevated temperature mechanical behaviors of MFRC were predicted using a universal visco-elastic concept. A survey of pertinent physical, chemical and mechanical properties was made on refractory metals, metal oxides, carbides, borides, nitrides, silicides, sulfides and single crystal whiskers. (Author)

Marshall, D. W., "Research on wire-wound composite materials," Massachusetta Instutute of Technology, Research report R 62-43, (November 30, 1962).

High tensile strength steel wires have tensile strength of the order of 600,000 psi at diameters approximately 0.004 inches and should serve to overcome several of the disadvantages of fiberglas. The material handles readily with essentially no strength loss and can economically be wound into a variety of container shapes. To investigate the usefulness of this material a threepart program of research was undertaken: study of the adhesion characteristics of single wires in epoxy and polyolefin resin systems; testing of flat sheet laminates using both glass and wire reinforcing in epoxy and poyolefin systems; and testing thin walled small bore tubes. The polyethylenes were included in these tests along with the epoxies because of their low cost and chemical inertness. Satisfactory adhesion utilizing both epoxy and polyethylene resins has been obtained. The wire has exhibited excellent strength and modulus when used in unidirectional flat laminates and appears to offer certain advantages over fiberglas. This is also true for the helically wound tubes where uniaxial burst tests have yielded results averaging 335,000 psi for maximum stress on the wire compared with 242,000 psi for HTS fiberglas roving.

Miller, D. G., Singleton, R. H., and Wallace, A. V., "Metal fiber reinforced ceramic composites," Allison Division GMC., Indianapolis, Ind.

A series of experiments were made with tungsten and molybdenum reinforced mullite matrices. The effects of fiber reinforcement are tabulated and also some properties of some candidate composite materials. Fibers - Be, Fe, Ni, Nb, Ta, Mo, W, Rh, SiC Matrices - $A1_20_3$, $Zr0_2$, $Th0_2$, $Si0_2 \cdot Zr0_2$, $2Si0_2 \cdot 3A1_20_3$, and Be0.

Theoretical calculations of Modulus of Rupture were done and compared with exp. Microphotographs of materials were made.

Morden, J.F.C., "Metallic fibers," <u>Metal Industries</u> (London) 96 (25), 495-99 (1960)

> Summarizes the methods (mechanica, from molten metals, chemical and others) of making metallic fibers, the properties of the fibers, fabrication techniques, etc. Ceramics may be reinforced by impregnating a felt of <10%vol.) with a slurry or by incorporating fibers in ceramic mixes. Increased strength and thermal conductivity result. 76 ref.

Murphy, E. A. and O'Rourke, R. G., "Development of very high strength beryllium wire," Final report, (December 7, 1964), <u>AD 610 316</u>

> Results of a program of development of a process to produce highstrength beryllium fine wire are described. The beryllium-iron alloys could not be deformed at temperatures used for the warm drawing technique. It was found that the 2% copper level gives improved mechanical properties to extruded forms over the 5% copper level. Both copper beryllium alloys showed very poor strain-hardening characteristics during the warm drawing operation. The draw-ability of the fine grained materials from the same starting material was found to decrease with decreased grain size with increased oxide content. A 15% increase in tensile strength with a substantial increase in wire ductility at 0.00477-inch diameter was shown for wire, as-drawn from 0.210-inch diameter, from starting material having -20 + 10 micron particle size. Similar strength levels with excellent ductility were obtained with high purity -15 + 1 electrorefined material at wire sizes of 0.001278-inch diameter. A 25% increase in tensile strength was obtained after picking. Author

Parikh, N. M., and Fisher, J. I., "An investigation of metal-ceramic composites for high-temperature applications," Final report, (May 18, 1960), <u>AD 238 137</u>

Fiber metal reinforced-ceramic materials were investigated to determine the nature and strength of the bond between metal and ceramics and the residual thermal stresses resulting from hot pressing. Conical interface shear tests showed that in vacuo, there is no chemical or diffusion bond formed between Al_2O_3 and any of the refractory metals (W, Ta, or Mo) at any temperatures. In the case of Ni-TiC system, there was no bond formation below the eutectic temperature. Above the eutectic temperature, the molten metal wets the carbide and the bond is stronger than the

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carbide at the density to which the carbide was consolidated. These conic interface shear tests indicate that reinforcement of the ceramic oxides by metal fibers is primarily due to mechanical interlocking. The most improved property of a ceramic occasioned by metal fiber reinforcement was thermal shock resistance. Reinforced specimens could not be harmed by severe thermal shocks (e.g., from 1500 C) regardless of the orientation of the specimen with respect to the pressing direction, although unreinforced specimens shattered under the same test conditions.

Roberts, D. A., "Physical and mechanical properties of some high strength wires," (January 20, 1961), <u>AD 250 088</u>

> This memorandum presents the available data on the mechanical and physical properties of some high-strength fine wires. The high strength fine wires covered in this memorandum are produced from four types of alloys:

- (1) High-carbon steels
- (2) Stainless steels 17 types
- (3) Nickel base alloys-9 types.
- (4) Tungsten and molybdenum

Robinson, R. K. and Farnsworth, P. L. "Tungsten fiber reinforced nickel composites," Paper presented at 10th meeting of the Refractory Composites Working Group, April 12-14, 1965, Atlanta, Ga.

> This report deals with some preliminary results of reinforcing unalloyed nickel matrices with tungsten fibers by pneumatic impaction. Tensile data was presented for the composite material and reinforcing agent at 2 temperatures for aligned and random fibers. Elongation and Vol % W fibers given. Details on fabrication and testing procedures presented.

Ross, Jack H., "Flexible fibrous materials and coatings for expandable re-entry systems," (March 1964) N64 23704.

It is shown that super alloy fibers of ultrafine diameter can be twisted into a textile-type yarn and woven into fabrics having almost any weave patterns. Utilization of standard textile-type equipment, slightly modified in the case of twisters, was proven feasible. Fabrics formed had superior mechanical characteristics as compared to monofilament fabrics. J.R.C.

Rumbles, W. E., Watanabe, S. F., Hayes, E. J., and Petrick, E. N., "Properties of fine wire for use in filament-wound vessels," Paper presented at the SAMPE Filament Winding Conference, Pasadena, California, March 28-30, 1961, <u>AD 254 650</u>

Steel wire exhibited the highest tensile strength (585,000 psi for a 0.004-inch diameter). The highest strength-to-weight ratio wires are steel and beta titanium, 2×10^6 in. and 1.95×10^6 in., respectively. Data included for physical properties of 0.006-in. diameter wire. These are high carbon, molybdenum, tungsten, beta titanium, copper, silver, platinum, nickel, aluminum, Rene' 41, stainless steel and steel.

Schuerch, H., "Compressive strength of Boron-metal composites," Washington April 1965 NASA-CR-202

> The compressive strength and failure mechanisms of an idealized composite material are analyzed. A method of predicting, theoretically, the compressive strength and failure modes of an idealized composite material is developed. The method is applied to a boron fiber-magnesium matrix composite and predicted strength values are compared to experimental data.

Schwatzbart, H., Rudy, John F., Bredzs, N., and Zoiss, M. H., "An investigation of metal reinforced lead," (January 28, 1963) <u>N63 12801</u>

This report summarizes the results of experiments on the development of two lead-base products haveing commercial applications: metal fiber reinforced soldering tape and metal fiber reinforced bearings. The soldering-tape investigations to determine the optimum density of the steel fibers impregnated with lead-tin alloys have been continued. Several fluxing procedures, including soldering in air without any external fluxes, were studied. The bearing test-program results thus far indicate that reinforcement by the fibrous steel network extends bearing life 50 to 75 percent. Author

Smith, F. M., "Reinforced structural composites," Interim report, (February 17, 1964) N64 19009

> A technique of fabricating tungsten bodies of increased strength was sought. Vapor deposition of unstressed tungsten on stressed tungsten wires was selected as the method for attaining the objectives of the program. Equipment was designed and constructed for the vapor deposition of tungsten metal from a gaseous mixture of tungsten hexafluoride and hydrogen onto a heated mandrel surface. A set of operating conditions was developed for the deposition of high-quality tungsten in the form of tubes of one inch outside diameter. While these tubes had good microstructures, taper existed in the wall thickness. Two tubes were fabricated that

that were reinforced longitudinally with stressed tungsten wires. Author.

Stowell, E. Z., and Liu, T. S., "Parametric studies of metal fiber reinforced ceramic composite materials," Final report, Contract NOas 60-6077-C, January 1960 through January 1961. Dated January 26, 1961.

> The purpose of this program was to conduct a theoretical study of the various parameters which affect the mechanical characteristics of metal fiber reinforced ceramic composite materials. In addition, a survey of properties of motential fiber and matrix materials was made so that promising combinations needing intensive investigation can be defined in specific terms.

Swica, J. J., Hoskyns, W. R., and others, "Metal-fiber-reinforced ceramics," (January 1960) <u>AD 233 453</u>

> The principal geometric variables involved in ceramic-refractory metal fiber composites were evaluated, using thermal shock resistance as the most important criteria. Several different ceramic-metal fiber composites were investigated. Using the alumina-molybdenum and alumina-mullite-molybdenum fiber systems, the comparative properties of the two basic types of composites were demonstrated. Composites were developed which had flexural strength exceeding 30,000 psi following four severe thermal shock cycles. (Author) (See also AD 207 079) Modulus of elasticity for molybdenum fiber given for variou diameter sizes.

Talley, Claude P., Clark, Wendall J., and others, "Boron reinforcements for structural composites," (March 15, 1962), <u>AD 296 575</u>

> B and B compounds are of interest as reinforcements for structural composites because of outstanding mechanical properties. B. filaments, prepared by chemical vapor plating have strengths of approx. 500,000 psi and moduli of approx. 55,000,000 psi. Some filaments gave strengths as high as 1.0 \times 10 to the 6th power psi. A laboratory scale continuous process was developed for producing B filaments of high strength and modulus. Continuous lengths of uniform B filaments, about 2-4 mils in dia. and over 700 ft. long, were produced and wound onto spools. Thus far, filaments from this continuous process have strengths of approx. 250,000 psi and moduli of 55,000,000 psi. A number of filaments had strengths over 400,000 psi. At temperatures as high as 1800 F the filaments retained about 60% of their room temperature strength. Slow etching in boiling nitric acid increased strengths up to 1.4 x 10 to the 5th power psi over original room temperature strength. Tests on small composite structures containing up to

64 B filaments bonded with epoxy resin indicated high conversion of filament to composite strength and modulus resulting in composite specific strength and modulus as high as 3.4 x 10 to the 6th power and 448 x 10 to the 6th power, respectively.

Talley, Claude P., Clark, Wendall J., Gunn, Kenneth M., Wawner, Franklin E., Jr. and Schultz, James E., "Boron reinforcements for structural composites, part II," (April 1963), <u>AD 421 568</u>

Exploratory research to obtain new and improved reinforcements for structural composites continued with emphasis on B filaments. A feasible laboratory-scale process for making continuous filaments by the H reduction of BC13 or BBr3 in a chemical-vapor-plating apparatus with W as a substrate material was developed. Over 15,000 ft. of filament was produced in lengths up to 1000 ft. Work was started on the use of diborane as the B source. Continuous B filaments from the BC13 route have shown room-temperature strengths of about 400,000 lb/sq. in. and moduli of 60,000,000 lb./sq. in. The currently utilized E glass reinforcement exhibits a usable specific strength of 3.78 x 10 to the 6th power in. in tension and a specific modulus of elasticity of 116 x 10 to the 6th power inches. Zone melting showed that a zone-melted crystalling B filament 26 mils in diameter gave strengths up to 5.94 x 10 to the 5th power lb/sq. in. in bending test. (Author)

Talley, C. P., and others, "Boron reinforcements for structural components," Texaco Experiment, Inc. (March 15, 1962), <u>AD 296 575</u>

> An exploratory research program has been initiated to obtain new and improved reinforcements for structural composites with outstanding properties. All high-strength, light-weight, and highmodulus materials are of potential interest; however, boron and boron compounds are of particular interest because of their outstanding properties. Batch-process boron filaments have been propared by chemical vapor plating which have strengths of approximately 500,000 lb/in² and moduli of approximately 55,000,000 1b/in.². This corresponds to a filament specific strength and a modulus of 5.3 x 10^6 and 580 x 10^6 in., respectively. Some filaments gave strengths as high as 1.0 x 10^6 $1b/in.^2$. A laboratoryscale continuous process has been developed for producing boron filaments of high strength and modulus. Mechanical tests on small composite structures containing up to 64 boron filaments bonded with an epoxy resin indicated a high conversion of filament strength and modulus to composite strength and modulus resulting in a composite specific strength and modulus as high as 3.4×10^6 and 448×10^6 in,, respectively.

Tinklebaugh, J. R., Goss, B.R., and others, "Metal-fiber-reinforced ceramics," (November 1960) <u>AD 251 929</u>

> The flexural properties of a ceramic-metal fiber system were studied and it was 'ound that the metal fiber does assume a part of the load which is to some degree in proportion to the relative elasticity moduli of the ceramic and metal. The ceramic fails when its strength is exceeded, but the composite does not fail until the metal fibers are broken or pulled out of the ceramic. The test data for the alumina-molybdenum and aluminamullite-molybdenum systems were extended to 3000 F. Hafnium oxide was found to have desirable characteristics for use in a composite system. (Author)

Walton, J. D., Jr. and Poulas, N. E., "Slip-cast metal fiber reinforced ceramics," <u>Am. Cer. Soc. Bull.</u> <u>41</u>, 778-80 (November 15, 1962).

This paper describes a preliminary effort to incorporate nichrome and stainless steel wool fibers in fused silica or alumina slips. Volume percent reinforcement, diameter, length, Modulus of rupture, elasticity given.

Witucki, R. M., "Boron filaments," (September 1964), NASA CR-96.

Results of the work to date show that filaments made by depositing elemental boron on tungsten wire are under considerable strain. This effect can arise in two ways. First, from expansion of the tungsten lattice as boron is introduced. Second, since the filaments are formed at a high temperature, a differential thermal expansion between the tungsten boride core and the boron sheath can cause significant stresses to develop on cooling. Such internal stresses can drastically modify the inherent strength of both the boron sheath and the tungsten boride core. Using a core material with characteristics different from the tungsten boride can be expected to have a great effect on the mechanical properties of the composite filament. The value of 220,000 to 270,000 psi found for the 500 turn test band, made with little development effort, is highly encouraging. The basic limitations of the present process must be considered. These principal limitations include the cost of the tungsten core, as well as the limited lengths that can be drawn, and the very slow production rates for the boron coating process.

D. CERAMIC

Adams, James U., and Sterry, J. Patrick, (December 1963) AD 439 841

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Zirconia fibrous insulations were prepared and characterized for high temperature air environments. The original zirconia-base batts were friable, underwent high temperature shrinkage, possessed a moderately high density-thermal conductivity product, and were subject to phase changes and grain growth. Some improvements were subsequently made by: water-felting with binders or carrier fibers to improve integrity; heat-treating to reduce shrinkage; use of additive fibers, finer diameter zirconia fibers and higher batt densities to reduce the predominant radiative heat transfer; stabilizing against phase change with various metal oxides. Investigation of several commercial fibrous insulations in hightemperature air aging experiments revealed that many silica and alumina-silicate materials were inadequate for practical use at temperatures much above 2400 F. Neodymia-stabilized zirconia and chromium-modified silica fibrous batts survived five hours at 300 F reasonably well. Chromium-modified silica did not appear suitable for longer exposure periods or higher temperatures due to either loss of the modifying agent or fusion. (Author)

Bortz, S. A., "A review of current refractory composite research in ceramics," IIT Research Institute, Chicago, Illinois.

Al₂O₃ fibers (continuous) are considered for use as a reinforcing agent and the production and properties are given. Data on Molybdenum fiber composites presented.

Carroll-Porcznski, C. Z., "Refractory fibres in modern industry," <u>Engineering Materials and Design</u>, <u>2</u>, 540 No. 11 (1959).

> Some commercial aluminia-silica fibers are discussed with reference to industrial uses. Fiberfrax, Kaowood and Cerafelt fibers were tested for various properties and results tabulated.

Chorne, J., Sutton, W. H., Gatti, A., Mehan, R., and Rauch, H., (May 1965), AD 615 662

> The Space Sciences Laboratory of the General Electric Company has been conducting investigations of the preparation and properties of fibers and fibrous composites for more than five years. Much of the original effort was directed towards the growth and properties of whiskers (short, single crystal fibers) and towards the development of high strength metals reinforced with these whiskers. While these investigations are continuing, additional programs have been undertaken which include the preparation of high modulus continuous filaments, a study of the wetting and bonding of metals to (oxide) whiskers, the development of effective composite fabrication procedures, and the evaluation
of composite properties in terms of their potential properties. This report briefly summarizes some of the current results of six programs, sponsored by government agencies, which are being conducted at the Metallurgy and Ceramics Research Operation of the Materials Sciences Section, Space Sciences Laboratory. (Author)

Davies, L. G., Withers, J. C. and Bazzarre, D. F., "A study of high modulus, high strength filament materials by deposition techniques," (January 1, 1965), <u>AD 611 757</u>

> A research program was initiated for the investigation of certain high modulus, high strength, low density materials in filament form. The primary objective of the investigation was to establish feasibility for depositing the desired materials by state-of-theart vapor deposition techniques upon a substrate in filament form. The following materials were selected for study on the basis of their high modulus-to-density ratio; boron carbide, silicon carbide, boron, titanium boride, titanium carbide, beryllium, beryllium oxide, and aluminum oxide. The filaments were characterized by their physical appearance and by the measurement of their tensile strength. The modulus of elasticity and density was measured on representative samples of each group. All candidate materials were successfully deposited. The boron carbide, silicon carbide, and boron deposition reactions produced high quality coatings at reasonable deposition rates. The other materials produced filaments of generally lower strengths, probably due, in part, to the high crystallinity of the coating. Beryllium, beryllium oxide, and aluminum oxide had very low deposition rates. The tensile strength of the boron carbide filaments ranged from 480,000 psi to 1,130,000 psi on 0.001" tungsten substrates. The silicon carbide had tensile strength to 2,000,000 spi, and boron had strengths to 1,900,000 psi. Silicon carbide had the highest modulus of elasticity (up to 800,000,000 psi) of the materials studied, while titanium carbide with measured modulus of less than 20,000,000 psi had the lowest. (Author)

Davies, L. G., and Withers, J. C. "A study of high modulus, high strength filament materials by deposition techniques," (July 15, 1964) <u>AD 603 403</u>

> Aluminum oxide filaments were successfully produced. Coatings up to 1.5 mils were obtained on .001 inch tungsten substrates. The resulting filaments had an average tensile strength of 240000 psi. The x-ray spectra of silicon carbide filaments made from SiC14, trichlorosilane, and trichloromethylsilane were compared. The filaments produced from the two silane compounds showed strong peaks of SiC whereas the spectra for the SiC14 produced filaments were inconclusive. Work began on the production

of beryllium filaments by the zinc reduction of BeCl2. No successful deposit has been obtained to date. (Author)

Davies, L. G., and Withers, J. C., "A study of high modulus, high strength filament materials by deposition techniques," (March 15, 1964), AD 433 501

Silicon carbide and titanium carbide filaments were deposited by vapor deposition techniques. An abbreviated study of the effect of varying deposition parameters upon tensile strength was made using boron filaments produced from diborane. The tensile strength was found to vary widely by adjusting the deposition parameters of temperature and gas ratios. The tensile strength of all materials deposited to date was measured. (Author)

Ellis, Richard B., "Investigation of techniques and materials for the formation of high temperature (1500 degrees F) inorganic fiber," (November 1961), AD 270 464

Production of a high temperature (1500 F) inorganic fiber by forming the fiber with an organic matrix at a low temperature and firing this form to remove the organic part is discussed. The feasibility of the technique was demonstrated on a laboratory scale by continuously firing an organically-bonded fiber to an entirely inorganic form. The copolymer Acrilan is employed as the low-temperature fiber former and matrix. The inorganic materials consist of powdered silica or kaolin and low-melting frits. The service temperature of Acrilan was extended to overlap the temperature at which a low-melting inorganic material softens enough to bond. An entirely inorganic fiber 75 microns in diameter was fired continuously from a composite fiber composed of 25% Acrilan and 75% inorganic materials. Information was developed for selection of compositions and conditions for the formation of fibers with diameters down to 10 microns. (Author)

Gates, L, E., Lent, W. E., and Teague, W. T. "Development of ceramic fibers for reinforcement in composite materials," (April 15, 1961), NASA CR 50793.

The purpose of this project is to conduct research directed toward the development of ceramic fibers and ceramic fiber composites for use in space technology. Results of fiber production studies of composites R-45, R-59, R-74, and R-76 indicated that fiber yield is lower per 50 blasts with a vertical fiberizing apparatus than was achieved with a horizontal apparatus. A re-evaluation study showed that composition, R-58, will produce sufficient fiber to merit further evaluation. Composition R-79, also produced sufficient fibers to merit futher study. Physical property tests were determined for a fiber resin composite of 40% CTL 91-LD resin and 60% of randomlyorientated zirconia flat fibers. Also, thermal expansion tests were completed on fiber-resin composites with fibers of compositions R-45, R-74, R-59, R-76, E-glass and zirconia. N.E.A.

Gullege, H. C. "Fibrous potassium titanate , a new high temperature insulating material," <u>Ind. Eng. Chem. 52</u>, 117-8, (February 1960).

Preliminary report of properties and uses of fibrous potassium titanate.

Kliman, Morton I., "Formation and properties of alumina fiber," (November 1962), <u>AD 291 825</u>

> The production of high strength aluminum oxide fiber has been achieved by means of plastic extrusion and solid state sintering of selected alumina powders at elevated temperatures. The processing variables for producing fibers possessing transverse rupture strengths of 100,000 psi are presented. The variation of elastic modulus and transverse rupture strength with temperature for alumina fiber is reported for temperatures up to 1300 C. (Author)

Lackman, W. L. and Sterry, J. P., "Ceramic fibers," il. <u>Chem. Eng. Prog.</u> 58, 37-41 (October 1962).

> A discussion of the types of ceramic fibers now available for use as fibrous reinforcements in composites presented in review form.

Lambertson, W. A., Aiken, D. B., and Girard, E. H., "Continuous filament ceramic fibers," (June 1960) <u>AD 243 566</u>

> Several refractory glasses in the baria-alumina-silica, calciaalumina-silica, and magnesia-alumina-silica systems were successfully drawn into continuous filaments. Tensile strengths of filaments drawn were determined at room and elevated temperatures. Values as high as 138,000 pounds per square inch at room temperature and 132,000 pounds per square inch at 1500 F (815 C) were obtained.

"Zirconia fibres - a new reinforcement for plastics," <u>Plastics Des.</u> <u>Processing</u>, <u>3</u>, No. 4, 36 (1963).

The development of zirconia fibres as a high temperature reinforcement for plastic materials considered briefly and some properties of phenolic resins containing these fibres are given.

"Synthesis of fiber reinforced inorganic laminates," Final report, (June 1962), <u>AD 293 930</u>

The use of inorganic materials as the matrix for reinforced composites is studied. The objectives of this research were to study the compositional and processing variables on matrix strength, the explanation of the observed deformation of matrix bodies, and research on protective fiber coatings. Type of bonding acid used, premilling time of raw materials, reacting temperature and milling time of reacted materials were considered variables affecting strength. Reactions occurring during drying, firing, and the mechanism of deformation were studied. Tin oxide, vapor deposited antimony oxide, molybdenum trioxide, organic-inorganic oxides, and liquid silver were considered as coatings for glass fibers. Strength and corrosive effects of matrices on Al_2O_3 , TiO₂, and ZrO₂ rods were examined. Strength of fibers sized with various coatings given.

Palmour, III, Hayne, "The preparation and mechanical properties of spinel," (July 1964,) <u>AD 446 682</u>

Magnesium aluminate spinel, under consideration as a possible high-performance structural ceramic because of its chemical and thermal stability, isotropic structure, and multiple slip systems at high temperatures, has been produced in fine-grained, essentially pore-free polycrystalline compacts by two different hot pressing procedures, using feed materials prepared by two alternative methods (i.e., direct solid state reaction of the parent oxides; coprecipitation and calcination). Variations in microstructural features, and in transverse and compressive strength and Young's modulus of elasticity as functions of the basic process parameters; as well as those introduced by deliberate second phases (graphite and/or pores), by oxidation state, and by feed material impurity and inhomogeniety are discussed. The marked dependence of high temperature compressive strength on certain details of starting materials and processing is also described. (Author)

Skrill, M. M., Machlin, E. S. and Weinig, S., "Basic investigations of ceramic fiber-alloy compacts," (March 8, 1960), <u>AD 610 54</u>1

Produced tensile specimens comprising 6 percent by volume of continuous nickel coated "Fiberfrax" fibers wound as a helix with a 45° angle relative to the tension axis in a tin matrix. An investigation was made to devise techniques of producing composite materials of non-metallic fibers and metals and to test such composite materials with the view of evaluating the factors that affect their strength. The technique finally achieved involves the infiltration of coated fibers by molten metals that cannot penetrate the fiber coating. The necessity of having a coating on the fiber that prevents attack by the molten metal but which promotes wetting of the fibers by the molten metal was shown by the results of tests. Two coatings are especially useful, namely nickel and molybdenum. Both these metals do not tend to attack silica or more refractory oxides. Hence, they are compatible with most non-metallic fibers including glass fibers. The metals can be deposited on the fibers from the gas phase by thermal decomposition of the metal carbonyls.

Spencer, S. B. and Jackson, W. O., "A new fiber for reinforced plastic aerospace applications," Paper given at regional technical conference sponsored by the N. Y. section of the S.P.E., Inc. May 13-14, 1964, Garden City Hotel, Garden City, L.I., New York.

Detailed discussion of the properties and uses of the "TX" fiber which is a Mg O fiber, in aerospace application.

Sterry, J. P., and Newman, D., "Polycrystalline ceramic fiber reinforcements for high-temperature structural composites," (January 1963), AD 455 865

> Investigations were performed on materials formulation and process techniques to develop high strength, high modulus, high temperature resistant polycrystalling ceramic fibers for use in reinforced composites. Polycrystalline oxide fibers having tensile strengths as high as 20 to 50 x 10,000 psi and elastic moduli in the 20 to 50 x 1,000,000 psi range were developed on a laboratory scale. Fibers are produced by passing an aqueous dispersion of ceramic powder with other additives through orifices and then heat treating to remove the non-ceramic material and create the desired microstructure. Various oxide compositions were studied with major emphasis on zirconia. The effectiveness of various additives for stabilizing the zirconia crystal structure and for minimizing grain growth at elevated temperature are discussed. Fiber evaluation consisted of tensile and modulus tests, x-ray diffraction analysis and electron microscope analysis of microstructures.

Sterry, J. P., Lackman, W. L., and Stites, J., "Ceramic fibers for refractory composites," (June 1962), AD 427 180

A brief paper discussing zirconia fibers as a ceramic fiber for high temperature composites. Various forms of the fibrous material along with its physical and mechanical properties.

Walworth, C. G., "Ceramic fiber--seven forms and how to use them," <u>Materials in Design Engineering</u>, vol. 46, p. 124-129 Carborundum Company (October 1957).

Traditional inorganic fibers such as glass, mineral wool and asbestos provide excellent service at temperatures up to 1000 F. Exposure above this temperature seriously degrades these materials. <u>Aluminum silicate</u> fibers are primarily intended to serve in similar types of applications at temperatures ranging from 1000 to 2300 F, i.e., in the range between previously available inorganic fibers and insulating refractories. A table compares properties of aluminum, silicate fibers with those of glass fibers, mineral wools, and asbestos.

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Beasley, W. C., McHenry, E. R., and Piper, E. L., "Research and development on advanced graphite materials, volume IX. Fabrication and properties of carbonized cloth composites," (May 1964), AD 602 735

> The development of fibrous, carbonaceous composites and laminates is described. Graphite, carbon, and heat-treated cloth and graphite and carbon felts are evaluated as fillers. Physical properties of the carbonized and graphitized forms of these materials are tabulated. Test results of these materials as potential hardware components are presented. (Author)

Beltaran, A. A., "Pyrolytic graphite, an annotated bibliography," (July 1961), <u>AD 263 604</u>

> The literature concerned with the production, properties and nondestructive testing of pyrolytic graphite, pyrocarbide, pyrofiber, pyrographalloys, and high density graphite is covered in this bibliography.

Bushong, R. M., "Pyrolytic coatings of carbon filaments," Final report (November 1964), AD 608 764 Tensile strength of, as received, heat treated, and pyrolyticallycoated carbon filaments given. Thermal and electrical properties given also. A continuous process for coating the individual filaments of graphite yarn with pyrolytic graphite discussed.

Dawn, Frederick S₄, and Ross, Jack H., "Investigation of the thermal behavior of graphite and carbon-based fibrous materials," (October 1962,) <u>AD 291 674</u>

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The elevated temperature properties of graphitized and carbonized polymeric fibrous materials were investigated as part of a research program on high temperature fibrous materials for use in parachutes and other decelerators, and in expandable structures. Experimental quantities of woven materials were evaluated after they were exposed to various temperatures for specific periods. One of these materials was partially graphitic (as shown by some order in X-Ray diffraction), the other two were amorphous carbon (no order shown in X-Ray diffraction). All three had been converted to carbon by controlled pyrolysis of polymers while in fabric form. Characteristics investigated as related to temperature and time at temperature were: breaking load to rupture, breaking elongation, energy absorption, and breaking strength after repeated stressing. The graphitized fibrous material was superior in tenacity initially and at 600 F to the carbonized materials investigated. Also, these materials were superior to nylon at temperatures above 350 F. Future investigations will be conducted to characterize the behavior of the carbonized and graphitized fibrous materials in the range of 600 to 2000 F. (Author)

Epremian, E., "New graphites," <u>Metals Eng. Quarterly</u>, v. 4, n. 3 (August 1964), p. 1-7

> Various types and forms of fibrous, foam and pyrolitic graphites are compared with conventional graphite regarding uniformity and reproducibility, strength, oxidation and erosion resistance, and thermal shock; problems and design considerations involved in use of new graphites in rocket nozzle inserts; stress systems and graphite behavior under varying temperature conditions.

Hough, R. L., "Continuous Pyrolytic graphite composite filaments," (December 1964) AD 460 724

Through in-house research by U. S. Air Force Materials Laboratory, continuous pyrolytic graphite filaments have been synthesized in diameters from $25 \pm 0.125\mu$ and individual lengths up to 2600 ft; they are composed of inner core of tungsten carbide with

overcoating of pyrolytic refractory-like highly "turbostatic" carbon; details of laboratory apparatus, designed, built, and successfully operated to obtain these filamentous materials; potential uses in space applications, ablative and reinforced plastic structures, filament-wound composites, thermal insulation inflatable re-entry structures, thermally protective fabrics, etc.

McCreight, L. R., Rauch, Sr., H. W., and Sutton, W. H., "A survey of the state of the art of ceramic and graphite fibers," Final report, (May 1965).

This survey was prepared for the purpose of compiling research, development, processing, and property information on ceramic and graphite fibers and whiskers. It attempts to compile a unified source of authoritative scientific and technological information on whiskers and fibers of use above 1200 F as fillers, flexible structures, insulations, and for reinforcements in composite materials. Ceramic and graphite fibers for such applications are the subject of a considerable and still expanding emount of research and development effort. It is therfore difficult to provide an exact measure and account of this effort at any one time, because it will always be somewhat out of date. The background and indeed much of the pertinent scientific literature have become available over the past 30 to 50 years. This report therefore summarizes that information and provides a means of following the field by indicating the types of materials, sources of information, and principal organizations involved in the field. This report is based on a review of over 500 references, over 200 patents and over 60 personal contacts, which were selected as being representative of the current trends in new fiber developments. (Author)

"Pyrolytic coating of carbon filaments," (November 30, 1963), AD 437 595

The pull strength of carbon yarn was increased up to three times by continuously depositing a coating of pyrolytic carbon on the yarn filaments. The yarn was heated to coating temperature both by electrical resistance heating and by radiation. A uniform coating on all filaments in the yarn cross section was achieved as determined by microscopic inspection. The significant process variables were defined as methane concentration in the coating gas and deposition temperature. The electrical resistance was reduced by a factor of 3 through application of the pyrolytic carbon coating. It was estimated that the thermal conductivity of the yarn was increased by the same amount. (Author) "National carbon yarn," Technical Information Bulletin No. 116HD, Rev. No. 4, Grade VYB, Union Carbide Corp., Carbon Products Div. 270 Park Ave., New York, N. Y.

> Description, nomenclature, production, physical, mechanical, electrical, thermal properties of Grade VYB carbonized synthetic filaments. Available in yarn form of various construction.

"Carbon-base fiber reinforced plastics," Aeronautical systems division, Dir / Materials and Processes, Nonmetallic Materials Laboratory, Wright-Patterson AFB, Ohio. Final report. Rpt. Nr ASD-TDR-62-635. (August 1962), 60 p.

Current research on high-temperature materials has lead to the development of a new class of nonmelting filamentous materials. These fibers are carbonaceous in composition, and hold great promise for use as reinforcing agents in both structural and ablative plastic composites. First generation developments in carbon-base fiber technology are reported. Techniques for synthesizing the fibered materials are discussed, together with the properties and characteristics of available materials. Unique plastic composites were prepared using carbon-base fibers and fabrics. Initial empirical results indicate that the composites are useful for both high-temperature structural and ablative purposes. Additional improvements will be required to realize the the inherent potential of carbon-base fibers. Several technical recommendations are given for eliminating current material deficiencies and improving fiber properties.

"Pyrolytic coating of carlon filaments," (February 29, 1964), AD 447 951

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Typical pyrolytic graphite coatings on individual carbon yarn filaments as produced in this study were found to increase the filament tensile strength from 92,000 to 130,000 lbs./ quare inch which corresponds to an improvement of approximately 40 percent. The same type of coatings increased the thermal conductivity of carbon yarn filaments from 0.064 to 0.145 cal./sec.-cm-C at room temperature, and from 0.016 to 0.019 cal./sec.-cm-C at temperatures slightly above 2000 C. Variations in the integral pull strength of uncoated and pyrolytically coated carbon yarn are shown to be random and to follow an essentially normal Gaussian pattern. Superposition of pertinent distribution curves is used to separate scatter from genuine effects of processing. In this fashion, the superiority of coated over uncoated carbon yarn is characterized in statistical terms. A sample of cloth tape has been woven from carbon yarn coated with pyrolytic graphite. Indications are that products of this type can be commercially manufactured in an size without difficulties. (Author)

OTani, Sugio, "On the carbon fiber from the molten pyrolysis products," <u>Carbon</u> (1965), Vol. 3, pp. 31-38

> A new carbon fiber was prepared from molten pyrolysis products such as polyninyl-chloride pitch which was prepared at 400° C under nitrogen. Properties of fiber given and method of formation.

Pallozzi, A. A., "Carbon fiber reinforcement of epoxy resins," In: <u>Society of Plastics Engineers</u>, Annual technical conference, 21st, Boston, Mass., March 1-4, 1965, Technical papers. Volume II. Conference sponsored by the Society of Plastics Engineers, Eastern New England Section. Stamford, Society of Plastic Engineers, Inc., 1965, P. I-VII-5 to 3-VII-5. 8 refs.

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Description of the preparation and testing of epoxy resin composites. Surface area, and the presence of water and surface complexes were altered on carbon yarns. NOL rings were formed to determine the relative importance of these variables on the properties of carbon-fiber epoxy-resin composites. It is concluded that the experiment implies that the most important practice in using carbon fibers is to be sure that the surface constituents are removed. Surface area and yarn strength are of relatively minor importance within the range of yarns used. Within the limits of this experiment, the following conclusions are said to be useful: (1) thermal treatment of carbon surfaces permits reproducible high strength bonding between fiber and resins. This factor appears more important than surface area or yarn strength; (2) carbon fiber surfaces can be thermally cleaned safely and economically be heating above 600° C in nitrogen; (3) greater surface area produced a small improvement in composite strength; and (4) dry yarn tensile strength variations have less effect than other system variables on the final strength of the composite. M.M.

Prosen, S. P. "Some properties of NOL rings of carbon-base fibers and epoxy resin," (Decmeber 4, 1964), <u>AD 454 635</u>

The recent improvement in the strength and the modulus of carbon-base fibers indicates that such fibers will soon compete with glass in composite structures where light weight is important. Carbon fibers, made by the pyrolysis of rayon yarn, were evaluated in the NOL ring form. A composite tensile strength of 5600 kg/cm² (80,000 psi), a compressive strength of over 7000 kg/cm² (100,000 psi) and shear strengths of over 350 kg/cm² (5000 psi) were measured. A compressive modulus of ever 0.28 x 10^6 kg/cm² (4 x 10^6 psi) was also determined. Data on carbon fiber also given.

Schmidt, D. L., and Hawkins, H. T., "Pyrolyzed rayon fiber reinforced plastics," (April 1964), <u>AD 438 892</u>

Constructive pyrolysis of fibered rayon materials in the absence of oxygen yields a corresponding carbon or graphite fibrous product which is flexible, thermally stable, nonmelting, strong, lightweight, and chemically inert. These novel fibers have been used as reinforcing agents in structural, ablative, and pyrolyzed plastics, and the composites thereof exhibit a combination of properties not previously obtainable. Recent developments in fiber property improvements, acquisition of property data on both fibers and plastic composites, and expanding uses for the pyrolyzed rayon fibers are summarized. (Author)

Schmidt, D. L. and Jones, W. C., "Carbon fiber plastic composites," S.P.E. Journal, (1964) 20, 162-9, No. 2

> The properties of the fibre obtained depend on the heat treatment given during pyrolysis and some of these are shown in tabular form. The properties of phenolic resins containing these reinforcements are discussed and tabulated.

Schmidt, D. L. and Hawkins, H. T., "Fibrous carbon reinforcements," Reinforced Plastics 1965, Regional tech. conf., Pacific Northwest section of S.P.E. (Tech papers), July 14-15, 1965.

> State of the art review of the history, and present uses, properties and preparation of carbon and graphite fibers. Material suppliers of pyrolized fabrics and engineering properties of carbon fabrics given. Oxidation resistance, ablative performance and mechanical properties of fibers given.

Schmidt, Donald L., and Jones, Warren C., "Carbon-base fiber reinforced plastics," Final report, (August 1962), <u>AD 288 289</u>

> Current research on high-temperature materials has led to the development of a new class of nonmelting filamentous materials. These fibers are carbonaceous in composition and hold great promise for use as reinforcing agents in both structural and ablative plastic composites. First generation developments in carbon-base fiber technology are reported. Techniques for synthesizing the fibered materials are discussed, together with the properties and characteristics of available materials. Unique plastic composites were prepared using carbonbase fibers and fabrics. Initial empirical results indicate that the composites are useful for both high-temperature structural and ablative purposes. (Author)

Sieron, J. K., "Pyrolyzed fiber; a high temperature reinforcing material for fluoroelastomers," (July 1963), <u>AD 417 391</u>

New high temperature reinforcing materials for fluoroelastomers were investigated. Pyrolyzed fiber, carbon in fibrous form, proved to be a very effective reinforcing material. The tensile strength of fluoroelastomers at 400 F was increased by a minimum of 50% over that previously attainable. Compounds reinforced with pyrolyzed fiber of 94-97% carbon content also had greatly improved resistance to 600 F heat aging. A compound featuring carbon fiber reinforcement retained 30% of original tensile strength and 60% of original elongation after aging 72 hours at 600 F. Conventional vulcanizates of fluoroelastomers were brittle after a similar evaluation. A description of the preparation and properties of carbon-base fiber is also given. (Author)

Slone, M. C. and Fox, R. V. "Graphite and carbon reinforced phenolics," <u>Technical Papers</u>, Vol X, Annual tech. conf., 20th S.P.E., Inc. 1964.

Use of carbon and graphite fabric in ablative applications discussed. Experimental data give for both fabric and composite given.

Zeitsch, K. J. and Higgs, P. H., "Pyrolytic coating of carbon filaments," (November 1964), AD 608 764

A continuous process has been developed to coat the individual filaments of graphite yarn with pyrolytic graphite. The pyrolytic coating increased the strength-to-weight ratio of the filaments by a factor of 1.3 and acted as a sizing on the yarn to make weaving easier. Tapes woven from the coated yarn showed a load distribution factor as high as 0.9. (Author)

F. MISCELLANEOUS

Badollet, M. S., "Asbestos, a mineral of unparalleled properties," Canadian Min. and Met. Bull. Vol. 54, P. 151-160, (1951)

The various properties of six varieties of asbestos were investigated and given in table form. Solubility, heat affect, ignition, composition and mechanical tests were made on the fibers. Electromicrographs of the fibers were made. Barrable, V. E., Bennett, A., Jerome, A.F., "Properties of crocidolite asbestos fibers for reinforcement of thermosetting resins," Soc. Plastic Industry, 18th Annual Tech. and Mgmt. Conference, 1963, Proc. Sec. 10-C 6 p.

Laboratory investigations reported show properties of Crocidolite fiber and of polyester, epoxy and phenolic laminates reinforced with Moramite asbestos felt; bend strength more than double that of glass, high tensile strength, long term flexural strength and excellent chemical resistance Crocidolite asbestos particularly suited for reinforcement of plastics to be used in industry and for construction of chemically resistant plastic pipes, ducts. tanks and covers.

Bennett, A., "Crocidolite asbestos fiber and its uses in reinforced plastics," <u>Plastics</u> (1964) <u>29</u>, 70-72 No. 319

The chemical composition of 3 types of asbestos are given and the strength properties of 2 of these are compared with those of glass fibre in tabular form. The properties of laminates and reinforced plastics containing the blue asbestos as reinforcement are considered and tabulated.

Carroll-Porczynski, C. Z., "Asbestos in composite materials, part II," Eng. Materials Des. (1962), <u>5</u>, 98-102, No. 5

Properties of such combinations as asbestos, phenolic, nylon resins are discussed in high temperature applications.

Carroll-Porczynski, C. Z., "Asbestos in composite materials, part I," <u>Eng. Materials and Des</u>. (1962), <u>5</u>, 30-33, No. 1.

The types of asbestos which are available are given with their differences in properties. These fibers are also used with fibers of other materials and laminated with epoxy resins. Detailed discussion of asbestos glass fibre phenolic resin composites.

Fahland, Frank R., "Survey on metal coatings of glass filaments," (June 15, 1963), <u>AD 421 008</u>

> The results of a literature search on metal coatings of glass filaments are presented. Areas surveyed include types of metals used to coat fibers, coating techniques used, thickness of coating, effects of metal coating upon glass filaments, and

glass compositions. It is concluded that the research work thus far conducted on metal coatings of glass filaments does not provide information sufficient to determine the protective or strength-enhancing capabilities of such coatings in a marine environment.

Little, Jr., C. O., "Thermal and gamma radiation behavior of a new high temperature organic fiber," (June 1961,) <u>AD 266 328</u>

> This research study involves a new temperature resistant experimental organic fiber known as HT-1, whose chemical structure is a departure from the conventional polyamide and polyesters. The behavior of this fiber during and after exposure to temperatures up to 650 F alone and in conjunction with gamma radiation indicates that a major breakthrough in organic fiber technology was achieved. Exposure to ionized radiation does not affect the tensile or elongation of HT-1 yarn. Tensile retention at 500 F is increased from 84.8 to 91.6% after combined thermalionized radiation exposure; at 600 F, tensile retention is increased from 52.2% to 78.2%. The superior behavior of HT-1 can be exploited to affect the greatest over-all advance in temperature resistant fiberology through utilization in missile and booster recovery systems; aircraft tires, ducting, fuel diaphrams and expulsion bags; reinforcement for ablating plastics for re-entry heat shields, and other hyperthermal applications. (Author)

Otto, W. H., and others, "Silica fiber forming and core-sheath composite fiber development," Final summary technical report, (January 31, 1964) <u>AD 438 145</u>

> This report describes a feasibility study of fused quartz fiber forming from a fluid melt contained in a bushing of molybdenum or tungsten in an argon atmosphere, and the development of a composite ceramic-glass fiber of core-sheath configuration. <u>Silica Fiber Forming</u>: Continuous fused quartz fiber forming was achieved from melts contained in both molybdenum and tungsten bushings operated in an argon atmosphere. Fiber forming temperatures ranged from approximately 2150° C to 2300° C. Tungsten was superior to molybdenum as a bushing material. Fiber forming in helium atmosphere was unsuccessful. Fiber tensile strengths were widely scattered with maximum values of 500,000 psi being achieved. An optimum tungsten bushing design had not yet been achieved and the useful bushing life was 4 hours at the end of the program. Core-Sheath Fibers: Using a redraw method of fused quartz, Vycor,

> and Pyrex tubing charged with candidate core materials, the feasibility of the core-sheath fiber was demonstrated with cores

of Al_2O_3 , ZrO_2 , and other high modulus polycrystalline materials. Modulus values higher than 20 x 10^6 psi and tensile strengths up to 200,000 psi were achieved. These studies showed that a dual melt system is required for continuous core-sheath fiber forming.

Plaskon, D., "Silica fiber forming and core sheath composite fiber development," Final summary technical report, (January 31, 1964), <u>AD 438 145</u>

> Silica Fiber Forming: Continuous fused quartz fiber forming was achieved from melts contained in both molybdenum and tungsten bushings operated in an argon atmosphere. Fiber forming temperatures ranged from approximately 2150 C to 2300 C. Tungsten was superior to molybdenum as a bushing material. Fiber forming in helium atmosphere was unsuccessful. Fiber tensile strengths were widely scattered with maximum values of 500,000 psi being achieved. An optimum tungsten bushing design had not yet been achieved and the useful bushing life was 4 hours at the end of the program. Core-Sheath Fibers: Using a redraw method of fused quartz, Vycor, and Pyrex tubing charged with candidate core materials, the feasibility of the core-sheath fiber was demonstrated with cores of A203, Zr02, and other high modulus polycrystalline materials. (Author)

Plaskon, D. and Bradford D., "Silica fiber/core-sheath fiber," Whittaker Corp., San Diego, California (November 30, 1964), <u>AD 452 306</u>

> Progress is reported on (1) apparatus revisions to improve conditions in the fiber attenuation zone; (2) argon flow redirection to reduce the amount of gas required for oxidation protection; (3) an experiment on indirect heating of a tungsten crucible-bushing; (4) evaluation of sintered silver-infiltrated tungsten as a bushing candidate material; (5) evaluation of five candidate core materials in the calcium-aluminate field for core sheath fibers, and (6) studies of means for improving the tensile strength of core-sheath fibers. (Author)

Plaskon, D., Bradford, D., and Otto, W. H., "Silica fiber/core-sheath fiber," (February 10, 1965), AD 457 810

> Progress is reported on (1) design of a cold-wall environmental chamber for improved atmosphere control, (2) silica fiber drawing experiments, (3) indirect heating of tungsten crucible for silica melts, (4) evaluation of Core Composition No. 10, (5) vacuum heating for core crystallization and acid-etching experiments for improvement of core-sheath fiber strength,

(6) crystallization of core during fiber forming, and (7) initial evaluation of 16 high-alumina glass compositions.

Taylor, W. "Asbestos reinforced plastics," Brit. Plastics, v. 37, n. 8, (August 1964) p. 435-9.

Review article covering following topics; properties of chrysotile, crocydolite and amosite fibers; general characteristics and data on physical, mechanical, electrical properties and chemical resistance of phenolics reinforced with asbestos flock for molding; nature of resin-asbestos bond; asbestos reinforcement for epoxy, melamine and silicone resins; examples of application.

Vidanoff, R., and Bradford, D., "Silica fiber/core-sheath fiber," (July 31, 1964), AD 446 339

> Effort was directed toward (1) development of a hightemperature, fiber-forming process from a silica melt contained in a tungsten bushing operated in an argon atmosphere; and (2) fiberizing feasibility and fiber evaluation of coresheath composite fibers formed from a dual-melt system provided by a double-compartmented, fiber-forming platinum bushing. Developments described in this report are (1) improvements in argon atmosphere control for the tungsten bushing silica fiber-fomring apparatus, (2) heat cycling tests on unsintered, hydrogen-washed tungsten bushings, (3) fiber drawing experiments using G. E. Type 201 fused quartz, and (4) a core-sheath fiber systematic study of the effect of core crystallization of a nucleating glass on modulus, density, and tensile strength of the composite fiber. (Author)

Vidanoff, R. and Bradford, D., "Silica fiber/core-sheath fiber," (May 30, 1964), <u>AD 443 225</u>

> This report describes work performed on a Navy Bureau of Weapons Contract which calls for: (1) development of a high-temperature, fiber-forming process from a silica melt contained in a tungsten bushing operated in an argon atmosphere; and (2) fiberizing feasibility and fiber evaluation of core-sheath composite fibers formed from a dual-melt system provided by a double-compartmented, fiber-forming platinum bushing. Developments described in this report are (1) redesign and assembly of the fiber-forming system for silica; and (2) core-sheath composite fiber studies including (a) evaluation of the dual-melt bushing, (b) fiberforming characteristics using three candidate core material, and (c) core-crystallization studies with accompanying measurements of tensile strength, density, and elastic modulus. (Author)

Vidanoff, R. B., "Silica fiber forming and core-sheath composite fiber development," (July 15, 1963) <u>AD 410 051</u>.

> It was shown that continuous quartz fibers can be drawn from melts contained in both molybdenum and tungsten bushings. Fiberizing temperatures ranged from about 2050 C to near 2400 C as sighted by a Pyro micro optical pyrometer. Tungsten proved to be superior to molybdenum: in the temperature ranges 2300 C to 2350 C all the molybdenum bushings and test strips ruptured. Drawing feasibility attempts were continued for core-sheath studies. Primarily, cores of sapphire rods, flame sprayed Al203 over quartz rod, flame sprayed ZrO2 over quartz rod, 89w/o ZrO2 with 11w/o SiO2, and several of Narmco's PYCEROX compositions using quartz tubes as a sheath were tried. (Author)

Vidanoff, R. B., "Silica fiber forming and core-sheath composite fiber development," (September 30, 1963) <u>AD 421 947</u>

> Improvements in the argon atmosphere for tungsten bushings resulted in higher tensile strength with individual fibers exhibiting strengths of 500,000 psi. Strength values of fibers sampled over a 2-hour continuous drawing period were scattered and inconclusive. Fiberization was completely successful in argon but not in helium under any condition of temperature and pulling speed. Studies of bushing orifice diameter showed the minimum size for successful fiber forming to be 0.060 in. Residual fused quartz remaining in the tungsten bushing caused rupture of the bushing upon cooling to room temperature. Fiber diameters for present forming conditions averaged close to 0.001 in. (Author)

<u>v.</u>	APPENDIX
	The Literature Covered in the Survey
1.)	Applied Science and Technology 1955-date
2.)	Chemical Abstracts 1957-date
3.)	Carbon 1964-date
4.)	Engineering Index 1960-date
5.)	International Aerospace Abstracts
6.)	Plastic Rapra Abstracts
7.)	S. P. E. Annual Technical Conferences 1955-date
8.)	S. P. E. Regional Technical Conferences 1955-date
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