MINUTES of TWELFTH DEFENSE CONFERENCE on NONDESTRUCTIVE TESTING

28-29-30 AUGUST 1961 NATICK, MASSACHUSE

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TWELFTH

DEFENSE CONFERENCE

ON

NONDESTRUCTIVE TESTING

28-29-30 AUGUST 1961

DEFENSE CONFERENCES ON NONDESTRUCTIVE TESTING

Scope.

The coordination of the development and the application of nondestructive methods for the testing and inspection of materials and assemblies for the Department of Defense.

Objectives.

1. To provide for an effective dissemination of information pertaining to nondestructive methods and applications among members and their respective establishments in the Department of Defense.

2. To provide for the utilization of the knowledge, skills, and experiences of specialists in the various branches of the Department of Defense for the analysis and solution of problems within the Military establishment.

3. To encourage (wherever applicable) uniform practices in the application of nondestructive testing methods.

Functions.

1. Development of a common report and information distribution list. (Emphasis on individuals rather than on organizations.)

2. Development and distribution of a current bibliography of defense establishment and/or contractor's reports, going back at least to 1946.

3. Development and distribution of abstracts of present and projected programs (Research, Development, and Engineering).

4. Compilation and distribution of useful information such as location, work specialities, staff, and facilities of:

a. Government laboaratories or other installations active in the development or utilization of nondestructive testing methods.

b. Commercial laboratories or installations active in the field of interest.

c. Consultants and Contractors active in the field of interest.

5. Evaluation of problems and proposed solutions in conference. The steps which are likely to be taken are:

a. Analysis of the problem in terms of design, engineering, production, inspection, and testing history. Such an analysis may be partial before presentation, but should be as complete as possible before specific solutions are detailed.

b. Presentation of problem to conference as a whole or conference panel qualified in nondestructive testing inspection.

c. Liaison between the group in which the problem arises and those qualified, as indicated by conference discussion, to provide assistance in the solution. (Once liaison is effected, the two groups proceed without channeling through the conference).

d. Report to conference (for the record) the degree of success or failure of the approach taken. If a solution has been reached, the formulation of requirements should be presented.

Organization.

1. Members - Employees of the Defense Establishment concerned with nondestructive testing, inspection, or evaluation, having confidential security clearance.

2. Officers - A secretarial board of not less than three, or not more than seven, who shall be elected yearly by a majority of the conference. This group will serve to receive and transmit information, to act as a steering group, and will elect one of their number as executive secretary.

3. Meetings - The conference shall meet at least once a year at various establishments, as agreed upon between representatives of the establishment and member whose establishment is acting as host.

HEADQUARTERS
QUARTERMASTER RESEARCH & ENGINEERING COMMAND, US ARMY
Quartermaster Research & Engineering Center
Natick, Massachusetts

SUBJECT: Twelfth Defense Conference on Nondestructive Testing

TO: Member Organization, Department of Defense

1. The attached document is the summary of the Twelfth Defense Conference on Nondestructive Testing which was held at the Quartermaster Research and Engineering Center, Natick, Mass. on 28-30 August 1961.

2. The papers and problems have been reported as they are written in the copies furnished. Report of business meeting has been condensed for editorial reasons. If changes or corrections are to be made, it is requested that these be addressed to:

QMR&E Command
Attn: Air Delivery Equipment Division/Budnick
QMR&E Center
Natick, Massachusetts

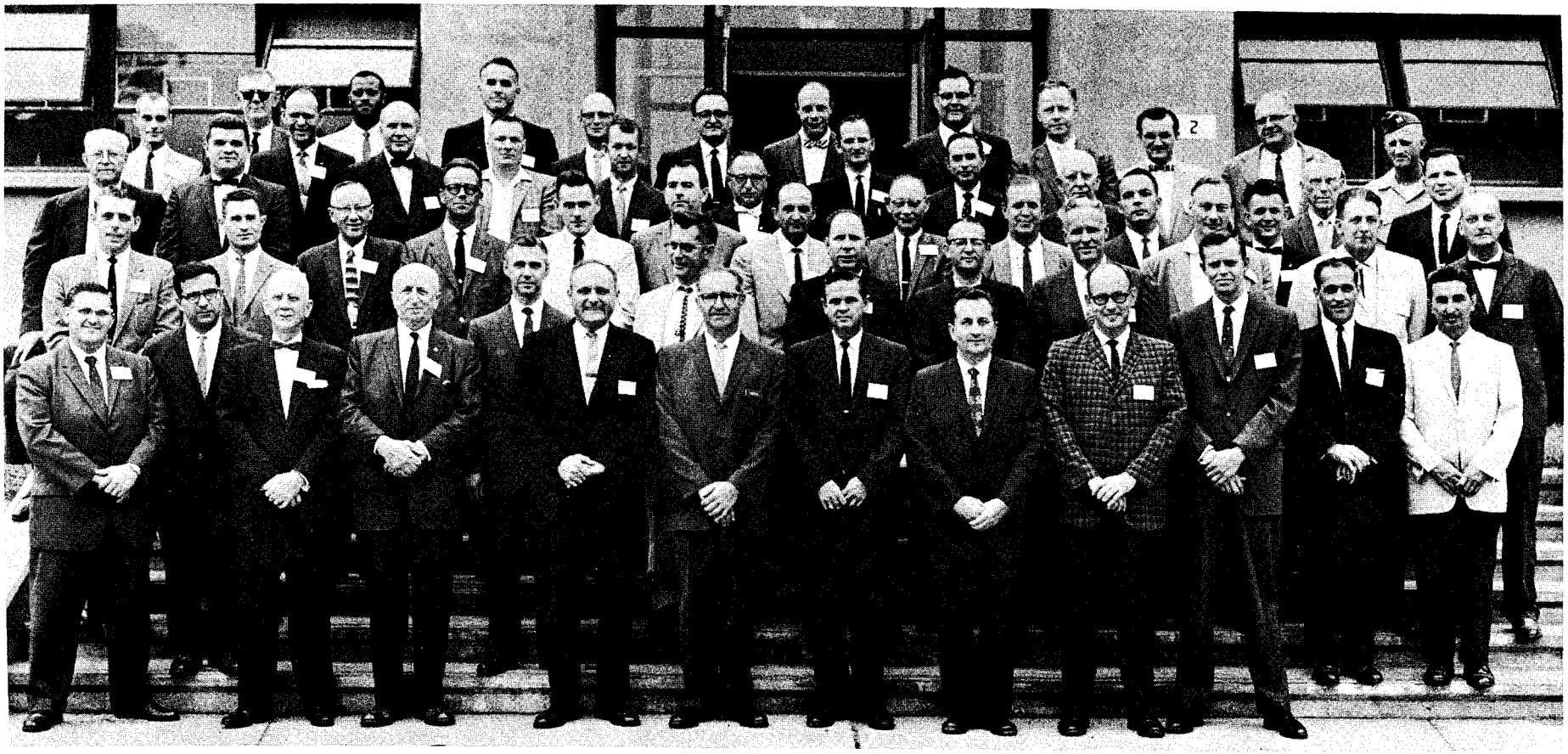
3. The editor wishes to thank all participants for a very successful conference and hopes that future conferences will be of continuing benefit to the Department of Defense.

FOR THE STEERING COMMITTEE:

M. L. Budnick

1 Incl
Summary

M. L. BUDNICK
Host Member



TWELFTH DEFENSE CONFERENCE
ON
NONDESTRUCTIVE TESTING

Quartermaster Research & Engineering Center
Natick, Massachusetts

28, 29, 30 August 1961

Host: Quartermaster Research &
Engineering Command

CONFERENCE AGENDA

Time	Event	Location
	Sunday, 27 August 1961	
1700	Registration of conferees	Hotel Madison
	Monday, 28 August 1961	
0745	Buses leave for QMR&E Center	Hotel Madison
0845	Final registration of Conferees	Lobby, Administration Building
0910	Host Chairman Welcoming remarks by Commanding General, QM R&E Command Technical Chairman - William Sheppard Warner Robins Air Materiel Area Robins AFB, Georgia	Hunter Auditorium Administration Building
0930	Formal Problem No. 1. - "Loss of Strength of Nylon Parachute Material due to Ultra Violet Light Degradation" - Middletown Air Materiel Area. 28439 10969-02 Non-Destructive Tests for Cable	
1000	Formal Problem No. 2.- "Nondestructive Inspection of Potted Electrical Multipin Connectors for Flight Environment Adequacy" - Picatinny Arsenal 10969-02	
1030	Formal Problem No. 3. - "Nondestructive Examination of Ag-Zn and Ag-Mg Type of Dry Charge Batteries for Degradation of Active Materials after Extended Storage" - U. S. Naval Underwater Ordnance Station. 10969-03 28440	

Monday, 28 August 1961 (continued)

Time	Event	Location
1100	Coffee Break	Cafeteria
1115	Formal Problem No. 4.- "Nondestructive Inspection of Bolt of 7.62mm M14 Rifle" - Springfield Armory	Hunter Auditorium
1145	Formal Problem No. 5.- "Nondestructive Inspection of Thickness and Quality of Bond of Zirconium Oxide of XLR-99-RM-1 Rocket Engine Thrust Chamber" - Air Force Flight Test Center	
1215	Formal Problem No. 6.- "Nondestructive Inspection of Cracks in Ferrous Boiler Tubes" - Norfolk Naval Shipyard	
1245	Announcements	
1300	Lunch	Cafeteria
1400	Informal Problem No. 7.- "Evaluation of Bond and Homogeneity of Brazed Joints of Mine, Land, 2-Gallon, M23" - U.S. Army Chemical Corps	Hunter Auditorium
1415	Informal Problem No. 8.- "Nondestructive Inspection for Adherence of Babbitt in Poured Bearing Shells" - Norfolk Naval Shipyard	
1430	Informal Problem No. 9.- "Nondestructive Inspection of Turbine Blades for Cracks at Mechanical Joints with Securing Members" - Norfolk Naval Shipyard	
1445	Coffee Break	Cafeteria
1500	Informal Problem No. 10.- "Nondestructive Inspection for Presence of Hydrogen Embrittlement in Metals Induced by Various Plating Procedures" - Kelley Air Force Base	Hunter Auditorium
1515	Informal Problem No. 11. - "Nondestructive Method for Determining Impact Stresses and Water Entry Angles of Small Air Drop Projectiles" - U. S. Naval Underwater Ordnance Station	

10969-05

28442

28441

10969-04

Time	Event	Location
1530	Informal Problem No. 12.- "Nondestructive Evaluation of Teflon Bladder Due to Degradation Caused by Long Period Contact with High Strength Hydrogen Peroxide" - U. S. Naval Underwater Ordnance Station	Hunter Auditorium
1545	Informal Problem No. 13. - "Nondestructive Determination of Static Load Required to Separate Steel Ball from Aluminum Housing" - Picatinny Arsenal	
1600	Informal Problem No. 14. - "Nondestructive Evaluation of Aircraft Cargo Tie Down Chains for Homogeneity of Links and Welds, Cracks, and Voids" - Middletown Air Materiel Area	
1645	Buses leave for Hotel Madison	Administration Building

Tuesday, 29 August 1961

0800	Buses leave for QM R&E Center	Hotel Madison
0900	Panel Discussions on Formal Problems	Hunter Auditorium
1045	Coffee Break	Cafeteria
1100	Panel Discussions on Formal Problems	Hunter Auditorium
1300	Lunch	Cafeteria
1400	Demonstration of "Tester of Insulation Value of Insulated Footwear" - Textile, Clothing & Footwear Division, QMR&E Command	Hunter Auditorium
1430	Technical Paper No. 1.- "New Concepts in Non-destructive Testing of Explosive Systems" - U. S. Naval Ordnance Laboratory	
1500	Technical Paper No. 2.- "Inspection of Solid Propellants of Missiles by Graphless X-Radiation Detectors" - U. S. Naval Ordnance Test Station	
1530	Coffee Break	Cafeteria

Tuesday, 29 August 1961 (continued)

Time	Event	Location
1545	Technical Paper No. 3.- "Nondestructive Testing Applications of X-Ray Analysis" - U. S. Army Ordnance Tank Automotive Command	Hunter Auditorium
1615	Technical Paper No. 4.- "The Use of Magnetic Resonance for Nondestructive Testing of Solid Propellants" - Picatinny Arsenal	
1645	Technical Paper No. 5. - "X-Ray Diffraction as a Functional Tool" - Frankford Arsenal	
1715	Announcements	
1730	Social Hour	Officers' Open Mess
1900	Dinner	Cafeteria
2030	Speaker	Hunter Auditorium
2200	Buses to Hotel Madison	Administration Building

Wednesday, 30 August 1961

0800	Buses leave for QM R&E Center	Hotel Madison
0900	Panel Discussions Summarization	Hunter Auditorium
1030	Coffee Break	Cafeteria
1045	Business Meeting	Hunter Auditorium
1130	Feedback Information	
1300	Lunch	Cafeteria
1400	Tour of QMR&E Center Buses to airport or Hotel Madison	Administration Building

"Report on Nondestructive Testing Activities at Research + Development Group FRANFORD ARSENAL" 10969-08

"Integrity of Missile-Motor-Liner Bond utilizing Ultrasonics" - E. D. Besser 10969-09

LOSS OF STRENGTH OF NYLON PARACHUTE MATERIAL DUE TO ULTRA-VIOLET LIGHT DEGRADATION

10969-01

OTTO H. LIZUT
USAF LOGISTICS COMMAND
MIDDLETOWN AIR MATERIEL AREA
OLMSTED AFB, PENNSYLVANIA

The problem of ultra-violet light degradation of nylon fabrics is not a new one. It has been the subject of continual investigation by all services and producers of nylon yarn.

Nylon parachute fabrics are of interest to all services. Parachutes have saved countless lives, materiel and aircraft. The biggest drawback of nylon has been its loss of strength in canopy materials. This comes about through exposure to the sun's rays and atmospheric chemical degradation during usage. The loss in strength of nylon canopy materials cannot readily be determined without destructive testing in the field or laboratory. This, in a nutshell, is the problem. Finding a non-destructive test method which can be readily used in the field as well as in the laboratory.

The magnitude of this problem was previously presented at the "Ninth Annual Defense Conference on Nondestructive Testing" by Dr. E. C. Yelland of the QMR&E Command. The problem, as presented by Dr. Yelland, only concerned itself with U. S. Army responsibility. To this, we must add the vital interest which the U. S. Navy, U. S. Marine Corps and the U. S. Air Force also have in extending the service life of its parachutes and components.

The primary consideration is the absolute necessity for maintaining the safety of personnel using parachutes. With the advent of the successful Mercury space program, it behooves us more than ever to be able to safeguard the landing of re-entered vehicles unto the earth's atmosphere using today's decelerators. Thus, we will be safeguarding an investment in terms of millions of dollars.

The problem then as now is the same. Since the original presentation by Dr. Yelland, E. I. DuPont de Neumors & Co have developed a Type 330 nylon which does not lose its strength as readily when exposed to ultra-violet light.

Another approach to preserving nylon's strength has been through the use of dyestuff. A highly successful dyestuff has been a premetalized Capracyl Yellow N. W. developed by E. I. DuPont de Neumors & Co.

Laboratory tests on DuPont Type 330 nylon have been conducted by Wright Air Development Division. The results of these tests have been published as WADD Technical Note 60-253.

An outline of the elements which make up the tensile properties of nylon are its molecular structure and arrangement, which, in turn, are made up of (1) chain length of the polymer (2) extent of cross-linking (3) the relative proportions of oriented (giving tensile strength) and amorphous (giving elongation) areas. These areas are modified, with a resultant loss of strength,

by ultra-violet radiation, chemical agents and heat.

The United States Department of Defense spends approximately 15 million dollars in the procurement and maintenance of deceleration devices. Therefore, it is highly desirable that an effective method of nondestructive testing be developed, through a joint cooperative effort of all services concerned. This method should be readily applied in the field as well as in the laboratory. From the data which would be gathered a set of guide lines could be established for better prediction of service life. Today the prediction of service life is at best a guesstimate. By developing adequate guidelines through accurate testing procedure we would enable the Department of Defense to save millions of dollars annually in replacement costs.

In conclusion, I know of no valid method for nondestructive testing of nylon materials for tensile strength. Therefore, I submit the above problem for your deliberation. Thank you.

10969-02

NON-DESTRUCTIVE TESTS FOR CABLE BY CONNECTORS AND CABLE ASSEMBLIES

By
JACK HOTTINGER
Picatinny Arsenal
Dover, New Jersey

Just short of 20 years ago the United States became involved in World War II and in this same number of years the designs of Ordnance ammunition has changed from rather simple types of fuzes, shell, grenades, bombs and demolitions to include exotic types such as Polaris, Davey Crockett, Minutemen, and Nike Zeus. Not only has the ammunition itself become extremely complex, but test sets and fire control systems become even more complex. To keep pace with this fast moving change of Ordnance materials, the life of the Quality Assurance Groups has become equally complex. Evaluation procedures must be developed to assure performance but to do this inspection equipment must be designed, developed, and evaluated. We find ourselves in the same vicious circle that the armor plate and armor piercing shot designers find themselves, that is, that a better plate must be available to test shot and then a better shot must be available in order to test the plates and so on. The HUAP shot and the introduction of shaped charges now have the shot designers ahead.

The Ordnance Inspector always has had the impossible problem of never being able to run a complete test on any Ordnance item by testing it to the end point because if he did, his efforts would only result in a big boom and no end item would be left to send to the field. This leads us to our current discussion. The particular problem is the non-destructive testing of electronic circuits in a black box, circuits of a group of black boxes and the non-destructive testing of cable assemblies with their connectors that make that system of black boxes the most reliable Ordnance item possible. The most reliable electronic circuits are no better than the cable assembly that connects that black box to its function.

The typical process in potting a connector is:

a. Wires are soldered to back end of pins.

b. Soldered pins are inspected.

c. Connector and wires are placed in jig.

d. Potting compound is prepared.

e. Curing of plastic - oven, air, accelerators.

f. Inspection of potted cable - (visual, X-ray continuity)

Typical of the conditions that a cable must withstand in its life cycle are:

a. High frequency vibration.

b. Relatively high acceleration forces.

c. High and low temperatures -65 to 160°C.

d. High and low humidity 0 to 100%.

e. Corrosion and fungus.

f. Extremes in atmospheric pressures from sea level to 500,000 feet and above.

g. Extended stockpile storage with a minimum of protection.

h. Certain abuses during transportation and surveillances for 5-10 year periods.

In development, cables that are exposed to all simulated tests of the above are not thereafter suitable for use in the field. Sampling plans could be used if one can assure a high quality control in the step-by-step process used to prepare and pot the connectors and wires. Our question is, "Can a non-destructive simple method be used to assure that the end items provided are all essentially alike?". Sampling incorrect test plans on an end item inspection is more economical than the more expensive in-process with inspection where there is more opportunity for human errors.

Picatinny Arsenal has been attempting to improve the reliability of cable connectors for warheads for some time. This problem was advertised as QDRI Problem #32 and all cable connector manufacturers were invited. This program is continuing under three phases: (1) to improve cable connectors as they are now designed; (2) improve pin and sockets of current designs; and (3) to design radically new designs.

Improved test procedures are also being sought with emphasis on non-destructive testing. Along with improved cable connectors, better methods of fixing wires, potting and jacketing are prime efforts.

To date the most promising non-destructive test of the completed cable assembly is X-ray or radiography using a cobalt 60 source in the field.

Other methods of end item quality assurance tests are being sought. The questions that we are out to answer are:

1. Is there good adhesion of plastics to all wires and connector materials? shrinkage?

2. Does the potting process create stresses?

3. What is the effect vaporous chemicals or air entrapped in plastic? What is the effect on electrical characteristics?

4. What is largest size of included air bubble permissible and how do they effect operation at high altitudes and cold temperatures?

5. What are long term storage effects?

Our search for better materials and manufacturing procedures is continuous and these efforts must include modern inspection methods and associated equipment. Some of you may have solved similar problems. If so, I ask for your recommendations and your solutions.

I thank you.

NON-DESTRUCTIVE EXAMINATION OF SILVER ZINC AND SILVER MAGNESIUM PRIMARY BATTERIES

1069-03

FRANCIS G. MURPHY
NAV UNDERWATER ORDSTA
NEWPORT, RHODE ISLAND

Ladies and Gentlemen:

Before I start, there are three things I want to emphasize:

1. I am not an expert.
2. I am presenting a problem to you.
3. I am looking to you, as experts on non-destructive testing, for a possible solution.

The modern torpedo weapon system employs a wire link for mid course guidance to improve hit probability. The position of the torpedo is dead-reckoned after it is launched. To minimize errors, the speed must be exactly as set. For the primary battery, this means a flat discharge curve that is accurately known. Besides the usual environmental hazards, the active materials in these batteries will deteriorate with age. Exposure to moisture and/or high temperature is especially harmfull. While an elaborate test program could establish statistical values for reasonable storage life, this is not of much interest to the sub-skipper. He would much prefer to know that the particular battery has just been checked and found to be good.

At present, there is no way to do this. Perhaps, after I give you some details on the batteries, you could suggest a method of non-destructive examination of these batteries.

Let's consider the Sea Water battery first. The choice of materials for a water activated battery is very limited.

Negative Electrodes

The difference in voltage between cells using magnesium and those using the other metals is so great that magnesium is the obvious choice except where the following properties are very disadvantageous.

Considerable local action occurs with magnesium so that:

(i) Gassing occurs - this may well be turned to advantage, as for example in the higher voltage water activated batteries, and for self-buoyant types.

(ii) Heating up occurs - this may be advantageous where operation at low temperatures is required, e.g. in freezing water or for balloon lights.

(iii) Considerable amounts of insoluble products may be formed which could, in some circumstances, lead to blockage and stifling of the cell. The amounts of insoluble products depend on the particular alloy used.

With zinc:

(i) No gassing occurs.

(ii) No heating up occurs, and

(iii) Hardly any insoluble products are formed, so zinc may have special application in units which are completely sealed during use such as reserve batteries for military applications.

These two metals are the ones in practical use, the others being unsuitable for either excessive reactivity with water or low voltage.

Positive Electrodes

Considering the properties of cuprous chloride and silver chloride as electrode materials, we may compare them as follows:

(i) Discharge characteristics

Cuprous chloride with magnesium normally produces 1.3 to 1.4 volts in a cell although this may be modified by the method of preparation. The discharge curve is usually rather hump-backed. Silver chloride with magnesium normally produces 1.5 volts or a little more and has a flat discharge curve for most of the life.

In consideration of electro-chemical equivalents cuprous chloride should deliver 50% more coulombs than the same weight of silver chloride; but, in practice, this does not hold due to direct reaction of water in the electrolyte with the cuprous chloride. Passivity of portions of cuprous chloride electrodes may sometimes occur with certain methods of manufacture.

(ii) Handling characteristics and storage

Both cuprous chloride and silver chloride have a corrosive effect on most common metals and care must be taken to keep tools clean and dry. Since silver chloride is ductile and cuprous chloride brittle, the former is easier to handle and less susceptible to damage on rough handling. Because silver chloride has much better storage properties than cuprous chloride, which will deteriorate rapidly even when exposed indoors in the summer, it requires less careful handling. Complete sealing of cuprous chloride batteries is essential if any length of storage is contemplated.

Manufacture of Electrodes

The problems associated with the manufacture of the electrodes will now be discussed.

Negative Electrodes

The simplest problem may be considered to be the preparation of negative electrodes. Here a wide range of forms is commercially available. Magnesium alloys or pure metal may be obtained in rod, sheet, drawn and extruded forms. The major problem is to choose the appropriate composition of the alloy and the choosing is a question of obtaining the best compromise between satisfactory corrosion resistance and a tendency to polarize or film during discharge. The corrosion resistance is of more or less importance according to the life of the battery and its particular application. For very short time operation, the corrosion resistance is perhaps of little importance, except as it affects the storage properties of the unactivated battery. For very long time operation, e.g. as batteries for use on certain life-rafts which may have to operate for several days or a week or more, the corrosion resistance is of prime importance. Excessive corrosion may have the following effects:

(i) A major proportion of the electrode may be consumed by the corrosion, thus requiring the electrode to have much more than the theoretical weight of material.

(ii) The corrosion products are largely insoluble and may result in choking up the battery.

(iii) The corrosion may be uneven, producing local perforation of the electrode and detachment of portions, reduction of active surface area and, if the detached metal can bridge the electrodes, resulting in short circuiting.

Silver Chloride

Pure silver chloride may be easily prepared. It can be cast into any shape desired, or may be rolled into sheets.

Problems of obtaining a rapid rise of voltage are much simpler than with cuprous chloride. Since the silver chloride is of salt consistency, wires may be embedded in, or welded to the surface to act as current collectors. The silver chloride may be perforated and backed by a silver sheet so that the discharge commences at the holes. A porous layer of silver can also be deposited on the surface.

Design Problems

Having obtained electrodes of various kinds, they can be applied to specific problems.

One common problem with water activated batteries is that they are frequently connected to the load prior to use. It is, therefore, necessary that the case shall be non-conducting and impermeable to moisture and the internal insulating materials used shall be very good. Moisture transmitted through the case could,

under some circumstances, activate the cell and this must be avoided, particularly since one product of discharge - the soluble chlorides, mentioned above, which are deliquescent, would tend to accelerate any discharge commenced by the admission of moisture by providing a lower resistance path over the surface of the separators, etc. The cases and separators are, therefore, usually made from good insulating material, such as polythene, polystyrene, polymethyl - methacrylate or cellulose acetate in that order of preference.

Now let's consider sea water batteries for torpedo propulsion.

The magnesium-sea water-silver chloride battery was originally developed by the Bell Telephone Laboratories for the Bureau of Ordnance during World War II. The General Electric Company completed the engineering development work and has manufactured finished battery sections for the Mk 35-2 and Mk 45 Torpedoes. These batteries have been evaluated by various Naval Ordnance Test Stations. The Mk 35-2 battery was found to deliver nominally 495 amperes at 120 volts for fifteen minutes when tested in a Mk 35 Torpedo. The Mk 67 Mod 1 battery for the Mk 45 torpedo delivers 560 amperes at 245 volts for 9 minutes.

Excellent engineering and quality control resulted in a very reliable battery when the 35 torpedo is fired in sea water of normal salinity and at temperatures around 80° F. However, the battery is not suited to operation in arctic regions or river estuaries due to its inherent sensitivity to temperature and salinity. The normal power output of this battery at 80°F. is decreased approximately 35 per cent when operated at 32°F. When the salinity is decreased to 50 percent of the normal value for sea water, the power output is decreased by 25 per cent and will approach zero output in fresh water. The Mk 67 Mod I battery compensated for this by a recirculating pump and flow control valve. Because of the simplicity of design, we will concentrate on the 35 battery.

The magnesium-sea water-silver chloride battery is a dynamic, primary reserve system employing bipolar electrodes. The system is activated by directing a flow of sea water through the cell assembly. A bipolar electrode (Figure 1) is constructed of magnesium (Dow Alloy J-1Z, 0.0105 in. thick), a barrier layer of silver foil (0.001 in. thick) and silver chloride (0.017 in. thick) whose surface has been activated by chemical reduction with a photographic type developer. The assembly is cemented together and the edges sealed by a stop-off lacquer. The electrode spacing (0.017 in.) is maintained by glass beads forced into perforations in the silver chloride by a coining operation.

Figure 1

Unit Cell Assembly

1. The .001 thick silver foil is glued to the .010 thick Mg alloy sheet.

2. Mg-Ag foil assembly is edge dipped in paint to reduce internal losses.

3. Beaded AgCl is glued to the silver foil.

(a) AgCl has been perforated and its surface reduced with Kodak D72 developer to decrease "come-up time."

Size:		
	Mg alloy sheet	11 1/8 x 11 7/8
	Ag foil	11 1/16 x 11 13/16
	AgCl	11 x 11 3/4

A unit cell pack (Figure 2), rated at 90 amperes at 135 volts for fifteen minutes, consists of 110 cells enclosed in a laminated plastic box. Supporting ribs at the bottom and top form ducts which permit the sea water electrolyte to pass up through the cells in hydraulic parallel. Five unit cell packs comprise the Mk 35-2 battery which is enclosed in an aluminum shell (Figure 3). The battery section is 46 inches long, of which 28 1/2 inches is taken up by the active portion of the battery.

The aft section contains a sliding water scoop which is forced outside the torpedo shell, after it leaves the firing tube, by a spring-powered release mechanism. The release mechanism is triggered by a small firing squib. The protruding scoop directs a continuous flow of sea water through the cell pack system and exhausts to the sea.

The output of this system is very dependent on the temperature and salinity of the ambient sea water. Since the temperature coefficient of the power output is approximately 3/4 per cent per °F., a battery operated at 32° F. will only deliver 65 per cent of its power rating at 80° F. A 50 per cent decrease in the normal salinity of sea water will lower the power output approximately 25 per cent and fresh water will cause the output to approach zero.

The Mark 67 Mod 1, by limiting the flow through the battery at low temperatures and by recirculating the electrolyte for low salinity water, can maintain almost constant voltage independent of environment. Figures 4, 5, and 6 illustrate the design of the Mk 67. In this battery, the positive silver zinc thickness is increased to 20.5 mils and there is a 25 mil plate separation. There are 2 parallel packs with 230 cells to give 245 volts and 560 amps. Plate size is approximately 12 x 15 inches.

Now, to consider the silver-zinc primaries:

For some 20 years prior to 1948, Andre of Paris had carried on development work, not only on the silver zinc couple, but general researches into the characteristics of silver and its compounds. In 1947 the world rights on the Andre patents were acquired by Mr. Yardney of New York who appointed licensee manufacturers in France, Germany, Sweden, Gr. Britain, Canada, and the United States. In 1949 the Electric Storage Battery Co. was given a contract to develop a silver zinc primary for torpedo application.

Development work led to the introduction in 1953 of thin sintered positive plate material with a constant thickness of 0.015 inches. This thin plate material has a density of 4.0 to 4.5 grammes per cubic centimetre.

A process of forge welding is used to attach the lead. Positive plate material for very high rate cells has silver expanded mesh introduced into the powder before pressing and sintering to provide better current distribution. This type of positive plate construction gives a return often equivalent to 2.5 to 3.5 grammes per ampere hour.

The control used for charging plates is the gain in weight which normally amounts to 12% to 12.5% which represents fairly complete conversion. Methods have been established whereby plates for primary cells are converted almost completely to silver oxide with only a small proportion of silver peroxide present. This is necessary because of the tendency for silver peroxide to decompose during storage especially at temperatures experienced in the tropics. If and when this happens, considerable quantities of free oxygen accumulate in the plate which greatly decreases the plates ability to discharge at full efficiency where the requirement involves high rates of discharge and rapid activation.

Since the main purpose for which one shot batteries are normally required is their ability to remain inert in storage for long periods and to become available for use at a moments notice, the maximum consideration must be given to the production of mechanically and electro-chemically stable plate systems. As I have said, the argentic oxide is much less stable than argentous oxide and a primary plate will be most effective if as complete conversion as possible is obtained to the argentous state. In general, primary design is based upon this factor. Therefore, it can be said that weight for weight of active silver, the conversion efficiency of the positives in the primary cell is lower than in the secondary although the reduced separator requirement together with other factors ensures that volume for volume the performance of both primary and secondary will be of the same order.

Negative Plates

Negative plates for primary cells can consist of compressed zinc powder, zinc sheet, zinc wool, or meshes or can be zinc sprayed on various backings. All of these variants have individual advantages. The most important factor involved is the current density which the plate will withstand without becoming passive, coupled with the necessity of ensuring adequate electrolyte around and in the plate structure since a plate of this type operates almost entirely around the soluble reaction. High concentrations of zinc in the electrolyte adjacent to the plate face can mean a considerable restriction of the performance. Plates of zinc powder are more able to deal with this situation since they will in similar circumstances be able to provide capacity by oxidation. However, development has led to negative plates of zinc mesh able to work at current densities as high as 1.5 amps per square inch of geometric surface.

There is no doubt that in the silver zinc primary cell the performance of the negative plate is the limiting factor and that this is most affected by the electrolyte.

The electrolyte used in the commercial silver zinc cell is potassium hydroxide of 45% concentration. It is normal to use no additives such as lithium as no benefit can be detected.

In many cases, it has been impossible to accommodate conventional terminals in the space available. This problem has been solved by the use of strip connections to the plates which are brought through the lid and forge-welded to form a solid bar of silver which is used as a terminal. For series connections in a battery, only a short section of the strips is formed solid to allow a seal to be made, the individual strips being interleaved with the opposite polarity strips in the next cell and lightly clamped together. The surface area of such a connection is, of course, many times that of a conventional terminal and the losses and corresponding temperature rise which would normally be experienced are not present.

Until 1953, it was the custom in this country to enclose the positive plate in cellulose film and the negative in a tissue wrap to provide some measure of physical support to the negative material. Diffusion conditions in the positive compartment were, therefore, difficult; and due to the varying degree of diffusion, consistency between one cell and another was unsatisfactory. In 1953 a complete change was made in the system. The positive plate, having become a self-supporting sintered plate, was used in that form and the separator enclosure transferred to the negative plate. Diffusion of electrolyte to the positive plate was no longer a problem; and it was found possible, primarily as a result of this change, to upgrade all established cells by as much as 50% in terms of capacity.

In a special battery developed by Diamond Ordnance Fuze Lab, two separators of a non-woven rayon material are used between each positive and negative plate. One separator, which is thick and soft, acts as a cushion against high impact. The other separator, which is thin and dense, is designed to retain colloidal particles of silver oxide dispersed in the electrolyte after activation.

The negative plates are formed by electrodepositing sponge zinc on a metal grid from an alkaline bath containing a slurry of zinc oxide. After deposition, the zinc sponge is compressed, washed thoroughly, and dried. The grid material is brass wire cloth on which a coating of hard zinc had been electrodeposited. Otherwise, galvanic action between the active material and the grid structure after activation might result in evolution of hydrogen.

The procedure followed in preparing the positive plates is as follows: A silver oxide-water paste is applied to a grid of silver wire cloth, and the pasted plate, after drying in air, is reduced in a muffle furnace to sponge silver. The plate is then oxidized electrolytically to divalent silver oxide in a dilute solution of potassium hydrozide at a low current density. The final step in the process consists of heating the plate at 120° C for 24 hours in order to reduce the divalent oxide AgO to the monovalent oxide Ag_2O.

The positive active material is reduced from the higher to the lower oxide because of the greater stability of the lower oxide. An experimental determina-

tion showed that the pressure of oxygen in equilibrium with silver monoxide prepared in this manner is about 5 mm of mercury at 125° F, which is to be compared with the theoretical equilibrium pressure of 3.5 mm of mercury. This low value of the oxygen pressure of silver monoxide, which has been observed to remain constant over a period of observation of six months, is considered to insure that the residual vacuum will be sufficient for activation of the battery over indefinitely long periods of storage.

Apart from the problems of mechanical design of the battery, two significant technical problems were solved: (1) virtual elimination of hydrogen evolution from the zinc negatives after activation, and (2) measurement of the internal pressure or state of the vacuum in the battery before activation.

Since the activating mechanism of the battery did not permit hermetic sealing of the activated cell, it was essential that gas evolution be virtually eliminated. Otherwise, the development of high pressure would cause leakage of the electrolyte through the seal between the cell compartment and the reservoir of rupture of the reservoir. Leakage of electrolyte from either source would inevitably impair the operation of adjacent components.

Since galvanic couples from which hydrogen might be evolved had been carefully avoided, and since the steel container was protected from corrosion by a coating of tin, the sole remaining source of hydrogen was from corrosion of the zinc negatives by the electrolyte. By immersing the nagatives momentarily in a 7-percent solution of mercuric chloride, the average rate of hydrogen evolution from four negatives for the 7-day period was reduced from 10.5 cc per day to 0.6 cc per day.

Unless the state of the vacuum within the nominally evacuated cell compartment is known, the reliability of the battery is obviously uncertain. For measurement of the pressure within the battery before activation, advantage was taken of the fact that the electrical resistance of air, after the initial ionization breakdown, varies with the pressure. A minimum resistance occurs at 5 to 6 mm of mercury, depending somewhat on the shape and spacing of the electrodes. This pressure also happens to be the minimum pressure obtainable in the battery because it is approximately the pressure of oxygen in equilibrium with monovalent silver oxide.

The vacuum-testing device used was a modification of a commercial electronic manometer. It applies a voltage up to 1500 v and a constant current of a few microamperes between the test electrodes. The device measures the resistance of the corona discharge in the rarefied air between the electrodes and gives an indication of the pressure in the range of 5 to 200 mm of mercury. Pressures between 200 and 760 mm give constant readings and all batteries within this range are discarded.

In adapting the electronic manometer for estimating the pressure of the battery, the regular battery terminals were used. Before a measurement is made, it is necessary, of course, to insure that no leakage paths caused by contamina-

tion of the surface are present. For this measurement, the resistances between the terminals, and between the terminals and the case, are measured with a sensitive meter and a constant 100 v source. In practice, battery resistances of over 1000 megohms are considered satisfactory.

Now to consider torpedo applications. Here we will talk about the Mark 46 battery. Both Exide and Yardney make these batteries for the Bureau of Naval Weapons. Each have their own manufacturing methods. Plate sizes and cell dimensions vary. For individual cells, there are 18 positive and 19 negative plates. Exide dimensions are 2.9 x 4.7 inches; Yardney, 3.9 x 4.2 inches. At full power each cell delivers 1.3 volts 450 amps. Each cell container is constructed of molded, rigid vinylite approximately 1/8 inch thick. Figure 7 is a picture of the complete Yardney battery. Note that there is an outside steel case, a rubber heater blanket, an inner stainless steel case housing the individual cells. Above, is the electrolyte container. Figure 8 is a sketch of the Exide battery showing the cell arrangement and method of activation.

Finally, I have two slides giving some of the characteristics of the active materials.

I would like to point out again that I am not a battery expert. Most of the information I have given you is taken directly from the literature.

To summarize:

I have given you details of the construction of silver primary batteries used for torpedo propulsion. An urgent need exists to find a non-destructive method for examining these batteries. The only method I know is the one in use for the special Diamond Ordnance battery. In discussing this with Yardney personnel, they do not use vacuum activation because of critical timing and hence this method is not applicable. Perhaps you gentlemen can suggest a method.

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NONDESTRUCTIVE INSPECTION OF BOLT OF 7.62MM M14 RIFLE

BY

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The bolt problem arose as a result of weapon malfunctions at Ft. Benning and at a contractor plant in December 1960. Malfunctions were catastrophic in nature producing severe weapon damage. This necessitates requirement for nondestructive method to test components and to insure high degree of confidence in weapon and its component parts. Slide 1 pictures a failed bolt with a broken right locking lug. Preliminary metallurgical examination of this failed bolt indicated that it had been quenched below the upper critical transformation temperature of the steel used resulting in a high percentage of proeutectoid ferrite in the core of the bolt. Slide 2 illustrates the microstructure of the core, which structure exhibits poor fatigue strength and/or low impact resistance.

Subsequent investigations on failed bolts and observation of process methods employed by contractors indicated a most complex problem. Many variations were found in heat treat methods and procedures with resultant components developing a variety of structures and properties. Bolt specification lists the following requirements. These are shown in Slide 3.

Material:	8620 H Resulphurized Carburized Steel
Case Hardness:	Rc 54-59
Core Hardness:	Rc 35-42
Case Depth:	.012 - .018 inches
Temper Temperature:	425°F (max.)
Core Structure:	10% free ferrite (max.)

Gas carburizing and liquid carburizing methods are used in manufacture. Variations in equipment and heat treating practices at different plants plus variations introduced into components in an attempt to salvage otherwise rejected parts due to skipped operations, distortion, and inadequate heat treatment result in extreme variations in material properties. Process variables included wide variations in carburizing time and temperature, character of quenching oils and degrees of agitation, and choice of tempering temperatures to meet hardness requirements.

Establishment of acceptance criteria has been extremely difficult because the number of failures has been small and actual conditions producing failures are not known. Bolts not only have variations in structure, differences in case and core hardnesses, and depth of case but may also have machining deficiencies, poor surfaces, lack of fillets and other stress raisers. Probability is high that failures will only occur when some combination of conditions exist in the component. In order to clarify criteria for acceptance for this group, one should consider methods which could be used to assure that all specifications requirements are being met.

Many varied magnetic investigations have been conducted at the Armory in attempt to develop adequate nondestructive test methods for bolt inspection. In conjunction with tests a great number of bolts have been sectioned and metallurgically examined to obtain structure, hardness, and case depth data. Each new investigation required additional destructive examinations because once sectioned, the bolts with then known properties could not be used. Watertown Arsenal personnel have cooperated with the Armory in this program.

In the initial work, magnetic comparator type equipment was employed. Slide 4 pictures the equipment. Basically it contains a 60 cps generator, a pair of similar coils, an amplifier, filter and detector circuits for indicating the resultant coil output voltage. Each of the similar coil units contained a primary and secondary winding. The primary winding of each coil applies an a.c. magnetizing field to the sample placed in the coil. The secondary windings are connected in series opposition so that only the difference voltage between the two secondaries is measured by the indicator circuit. When like samples with identical magnetic properties are placed within the coils, the induced voltage in each secondary winding is equal, and the resultant output voltage is zero. In the actual test, a reference bolt was placed in one coil where it remained throughout the test. Bolts being compared were then inserted in the other coil.

One hundred and five (105) bolts manufactured using gas and liquid carburizing methods were tested initially. Four magnetic comparator set-ups were studied to determine which one appeared to give the widest differences in bolts examined. Wave forms with these set-ups were noted in order to study phase shifts and presence of harmonics. Filter net-works made it possible to measure the resultant secondary output in terms of the fundamental only, the 3rd harmonic only, the fundamental plus all harmonics, and all harmonics with the fundamental filtered out. Widest differences were noted employing the fundamental only and the fundamental plus all harmonics. Metallurgical examinations on 20 of the 105 bolts tested showed a general grouping, but with unaccountable differences within each group. Meter readings primarily representing a permeability measurement appeared to show that permeability increased with increasing percentages of free ferrite in the core and decreased with higher core hardness; however, too many exceptions were noted to assume any correlation.

Because permeability appeared to offer no correlation with core hardness, studies of other measurable magnetic properties, such as retentivity, coercive force, and residual fluxdensity were undertaken.

Laboratory equipment comprising a.d.c. power supply, magnetizing coil, and a gaussmeter was used in the measurement of retentivity which is the fluxdensity remaining after a magnetizing current sufficient to cause saturation has been applied and reduced to zero. This measurement was made on twenty (20) bolts heat treated as a group. In addition, the opposing current required to bring the retentivity to zero was recorded. A plot of this opposing current vs core hardness is shown in the following slide. Unfortunately, when bolts from various heat treat processes were tested and metallurgically examined, the linear relationship as indicated in the slide was non-existant. Numerous results did

not fall on the curves. For example, a bolt tempered at 500°F with core hardness Rc42 gave a current reading of 265 milliamps. It was concluded that the measurement was more affected by tempering temperature than core hardness.

Tests were next conducted using a.c. hysteresis loop measurements in an effort to distinguish variables from one another. A 60 cps generator was used to induce an a.c. field in a "U" shaped, high permeability, laminated iron core. A secondary winding encircled the bolt and the bolt was placed against the pole faces of the core thus completing the magnetic circuit. By means of a phase shifting network, the hysteresis loop was displayed on an oscilloscope. Coercive force and residual flux density measurements were recorded on nearly 200 bolts, first at saturation flux density, then at a constant flux density below saturation. The greatest sensitivity was indicated using the latter method. Before destructively examining any of these bolts, magnetic readings were recorded using a previous Magnetic Comparator set-up in an attempt to gather as much data on this group of bolts as possible. Sixty three bolts were sectioned and the metallurgical results were most carefully compared with the collected magnetic data. Results indicated promising correlation of coercive force versus core hardness, however, as with the retentivity measurements, it was concluded that coercive force was more affected by tempering temperature than core hardness. A literature review supported this conclusion.

An arrangement of the magnetic data which appeared significant was a plot of coercive force versus magnetic comparator readings. A boundary line was drawn on the data plot which divided good components from deficient ones. It was possible to separate bolts containing a high percentage of free ferrite from those which had been highly tempered or retempered. Previous magnetic comparisons had not differentiated these conditions. However, a few gas carburized bolts with comparatively low surface hardness and very high core hardness upset the correlation in that these plotted within a group of acceptable bolts. This lack of correlation proved most important because greater concentration was now given to the surface hardness variable. Because flux density is at a maximum on the bolt surface and decreases in an exponential manner into the core, it seemed most reasonable to more carefully consider surface hardness when examining the magnetic data with respect to core hardness.

At this time, 1800 bolts were received from Raritan Arsenal which required some type of a segregation due to the urgent need for supplying weapons to the field.

Magnetic permeability and Rockwell C surface hardness measurements were recorded on all 1800 bolts. Surface hardness, magnetic reading, and core hardness were plotted three dimensionally on 25 of these bolts. 24 of the 25 provided an accurate determination of core hardness within $\pm$ 1.5 Rc. The other was 5 points Rc harder than predicted. In conducting the magnetic permeability tests similar characteristic wave form distortions were observed on the oscilloscope. Additional investigations of these patterns revealed that core hardness was consistently harder than predicted when one characteristic pattern was present. Predictions with another pattern were excellent. Based on this, all

remaining bolts were re-examined with respect to wave form pattern. Five obvious patterns were evident, but many variations of these patterns were present. Obvious patterns are shown in the following slides.

Slide 6 - Waveform of bolt within specification requirements

Slide 7 - Shows characteristic distortion due to bolt with core greater than Rc42 (note sharp break in slope)

Slide 8 - High ferrite (25 - 40%)

Slide 9 - Borderline ferrite (5 - 15%)

Slide 10 - Tempered or retempered above 425°F

Slide 11 - Bolt containing decarburization

To further study these previously mentioned hard to distinguish variations, approx. 150 oscillograms were taken of the differing degrees of distortion. The corresponding bolts were sectioned and metallurgically examined. These oscillograms were then used to differentiate patterns in the subsequent bolt segregation program undertaken.

Using this test, 26,000 bolts have been segregated to date into the following groups:

Group		Total
A	- Core Hardness Rc 35 - 42.5	5795
B	- Core Hardness Rc 42.5 - 45	14581
C	- Outside Rc surface hardness	651
D	- High temper on retemper	2916
E	- Core hardness greater than Rc 45	87
F	- Core hardness less than Rc 45	-
G	- Core containing excess free ferrite	469
H	- Other unfavorable conditions	1898
		26,397

Though results and progress to date on the bolt segregation program have been most gratifying, it is felt that the combination of tests using case hardness, magnetic reading and waveform pattern is too complicated to employ as an in-process inspection method. Engineers who are experienced with the problem

and have observed properties found and waveform pattern differences are able to make suitable segregations, but test is not sufficiently refined to specify limits and procedures which could be incorporated in a drawing or specification for general inspection. In addition to possible test methods which might serve as in-process inspection methods, any idea on manner to establish acceptance criteria to assure satisfactory component service life is most desired. Tests conducted on bolts taken from various segregated groups give no assurance that components in any group will fail in service. Failures are few and conditions which produce failures are not known.

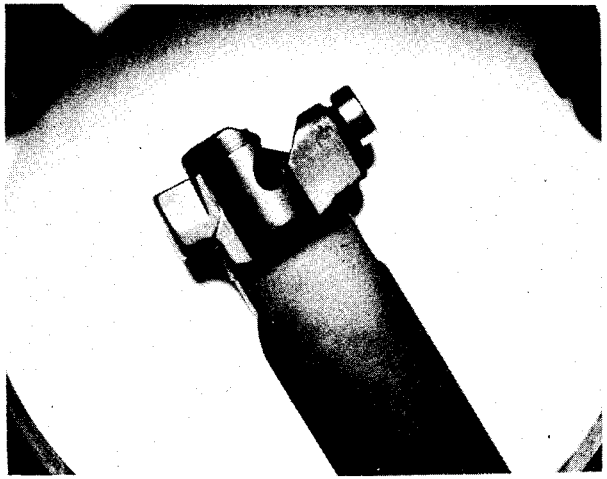

FIG. 1

FIG. 2

M-14 BOLT REQUIREMENTS

MATERIAL:	8620 H RESULPHURIZED STEEL
CASE HARDNESS:	R_c 54 - 59
CORE HARDNESS:	R_c 35 - 42
CASE DEPTH:	.012 - .018 INCHES
TEMPERING TEMPERATURE:	425°F (MAX)
CORE STRUCTURE:	10% FREE FERRITE (MAX)

FIG. 3

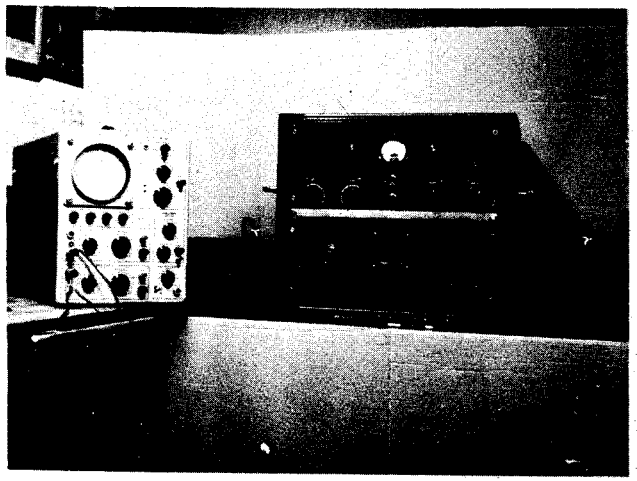

FIG. 4

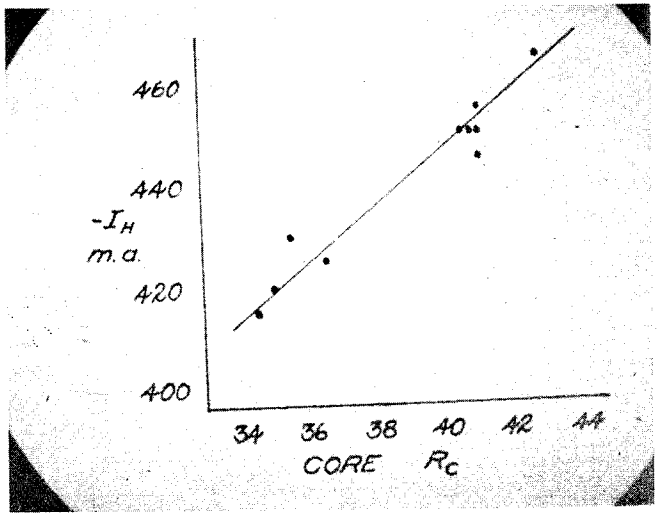

FIG. 5

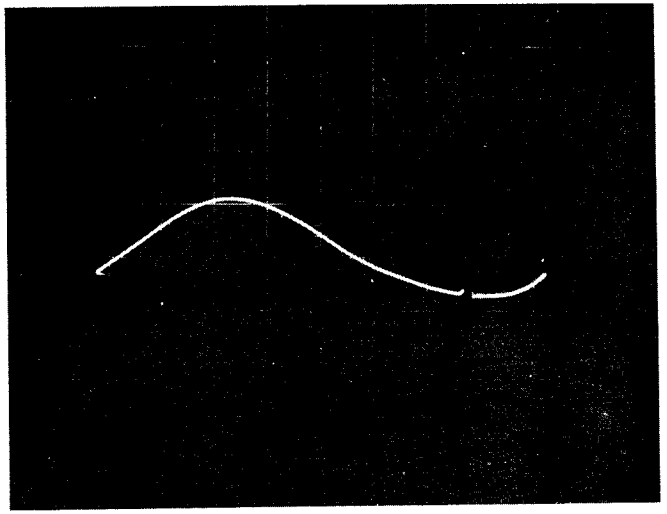

FIG. 6

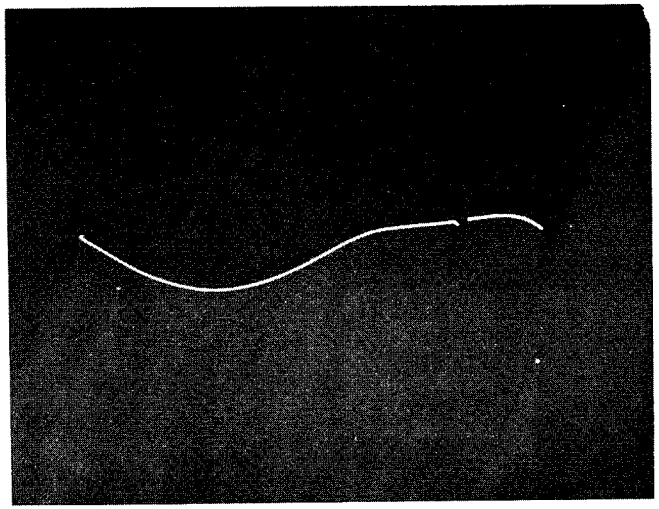

FIG. 7

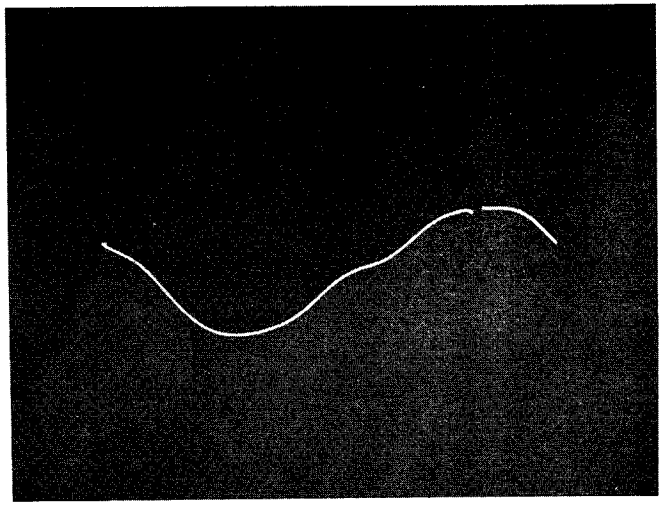

FIG. 8

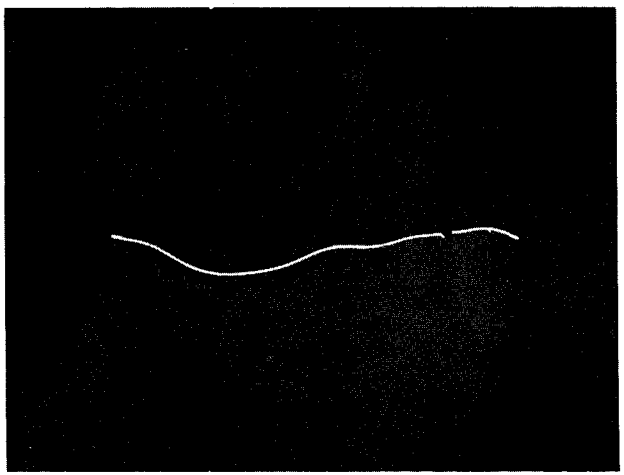

FIG. 9

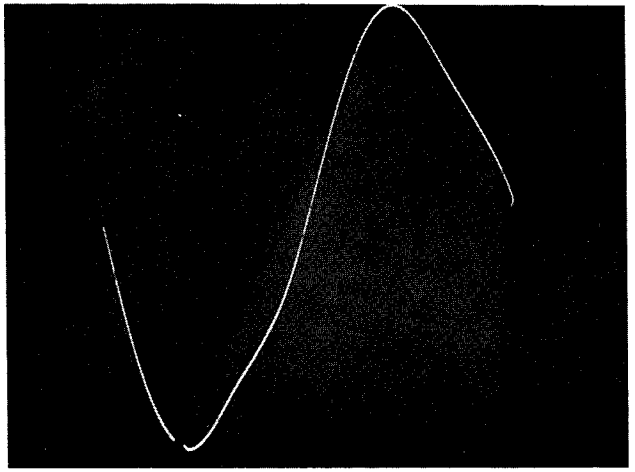

FIG. 10

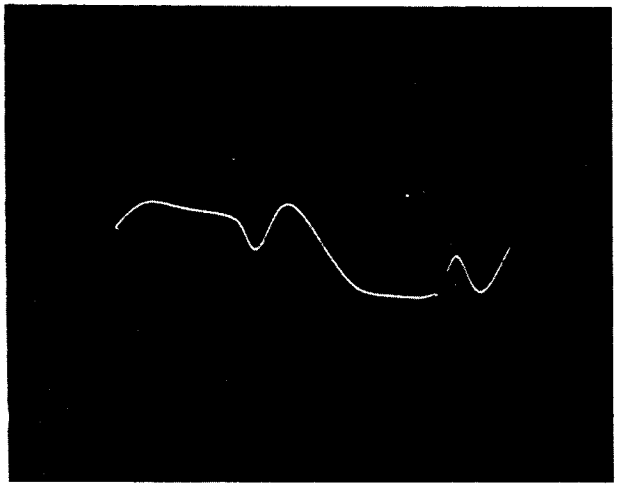

FIG. 11

NON DESTRUCTIVE TEST PROBLEM
Nondestructive Testing of Thickness and Quality of Zirconium Oxide Coating of XLR-99 Rocket Engine Thrust Chamber.

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Introduction

The XLR-99 Rocket Engine is the propulsion unit for the X-15 Aerospace Research Vehicle designed and built by North American Aviation Company at Inglewood, California. This vehicle was designed for NASA and the Air Force for purposes of studying hypersonic and extremely high altitude flight conditions, the effects of aerodynamic heating, exit and re-entry techniques, and physiological and physchological problems during sub-orbital flights.

The X-15 was designed to attain a speed of 4000 miles per hour and an altitude of 250,000 ft. The outer skin of the X-15 is constructed of inconel X material capable of withstanding 1200°F temperatures. The XLR-99 is a liquid propellant engine rated at 50,000 lbs of thrust. The X-15 on 30 March 1961 attained an altitude of 169,600 ft with Mr. Joseph Walker, NASA, the pilot. On 23 June 1961 the X-15 attained a speed of 3603 miles per hour, with Major White, USAF, the pilot. Skin temperatures in excess of 800°F have been recorded to date.

Description of XLR-99 Engine Components and Operation:

1. The XLR-99 RM-1 rocket engine is designed and manufactured by Reaction Motors Division, Thiokol Chemical Corporation, Denville, New Jersey. The main propellants consist of anhydrous ammonia fuel JAN-A-182 and liquid oxygen oxidizer MIL-P-25508, USAF. Major engine components are a turbopump assembly, a gas generator to power the turbopump, a single thrust chamber, a thrust chamber igniter, propellant valves, propellant lines, and miscellaneous control components. In operation, the turbopump assembly, which consists of two centrifugal pumps driven by a two stage impulse turbine, is supplied with propellants from low pressure vehicle tankage (not included as part of the engine). Upon discharge from the pumps, the propellants are delivered at high pressure to the thrust chamber and chamber igniter, with the fuel being used first to cool the thrust chamber wall. The propellants are injected at a nominal mixture ratio of oxygen to ammonia of 1.25.

2. The thrust chamber consists of 196 "spaghetti" tubes, carrying the fuel coolant and providing the thrust chamber internal profile. The tubes are fabricated of annealed 346 stainless steel with a 0.035 in. wall thickness and bonded together by a high temperature furnace braze to form the thrust chamber; hoop tension in the tube bundle resulting from chamber pressure is taken by a wire wrapping around the chamber exterior.

3. The gas generator is supplied with concentrated (90%) hydrogen peroxide from a small high pressure auxiliary tank. Engine thrust can be varied by the pilot over the range of 25,000 to 50,000 pounds at sea level at present, with plans to increase the throttling range from 15,000 to 50,000 lbs. The engine is capable of five restarts without additional servicing and will start and operate at any altitude. Rated life of the engine is one hour accumulated operating time or 100 starts, whichever occurs first. However, the present chamber life has been averaging about 18 minutes due to the loss of zirconium oxide and subsequent tube failures. This limitation has indicated the need for development of nondestructive inspection techniques for determining the condition of the thrust chamber prior to actual failure.

Thrust Chamber Materials and Manufacturing Processes:

1. Materials involved consist of 3/8" O.D. x .035" wall thickness 347 stainless steel tubing (annealed), Rokide Z zirconium oxide (lime stabilized), nickel chromium (60 Ni, 16 Gr, 24 Fe) in metallized form.

2. Manufacturing processes:

a. The stainless steel tubes are formed to make up the contour of the thrust chamber and furnace brazed together in the fillet formed by the tubes, then annealed and stress relieved to RB 87-88 condition.

b. Externally the tubes of the chamber are wire wrapped using an epoxy resin bond (reference photo #3, 865-61).

c. After mating of the tube sub-assembly to the combustion section by welding, the I.D. of the chamber is cleaned using high pressure grit blasting prior to metallizing.

d. The metallizing process consists of applying a coating of nickel chromium .004 to .008 inch thick while circulating water through the tubes at a flow rate of 2-3 gallons per minute at a temperature of 115°F ± 15°F.

e. Rokiding operation is then accomplished utilizing equipment similar to the metallizing equipment, applying a coating of zirconium oxide (lime stabilized 1/8" rod form) .007 to .015 inches thick over the metallized surface. The Rokide is applied in five layers with a rod fed Norton flame spraying gun which melts the rod in an oxy-acetylene flame (max temp 6500°F.), and the combustion gas stream then deposits the fused refractory on the metal undercoat. The tooling which rotates and holds the chamber is fully automated as is the gun traverse mechanism.

3. The manufacturer, RMD, relies primarily on operator technique to assure quality metallizing and rokiding of the chamber; visual inspection is performed during application and a hydrostatic test is accomplished on the welded assembly.

Nondestructive Inspection Requirements:

1. A nondestructive inspection method is needed for measuring the thickness of zirconium oxide (rokide) initially being applied to the metallized surface of the thrust chamber and after subject chamber has been operated in the field.

The application of rokide is for the primary purpose of aiding in reducing bulk temperature rise in the coolant (anhydrous ammonia NH3) fuel and in eliminating oxidation of the coolant tubes by the high temperature, high velocity gases. Estimated combustion chamber flame temperature in the critical plane, upstream of the chamber throat, is approximately 4300°F. During operation of the chamber loss of rokide is experienced in this plane (reference photo #3,867-61) which in turn is followed by erosion of the outer gas side wall of the 347 stainless tubing (reference photos 357L and 1780). Experience has indicated that this condition is accelerated by the application of rokide coating in excess of the desired maximum thickness .015" or less than .007" minimum. Following the loss of rokide insulating material inner wall cracking occurs, probably as a result of thermal stresses being applied to the tube in localized areas (reference photos 362L and 358L) coupled with a possible chemical change in the base metal. Microphotographic evidence from sectioned failed tubes indicate that tube temperatures rise to 1250°-1300°F when loss of rokide is experienced. This rise in temperature causes a breaking down of the NH3 cooling fluid and nitriding of the tube I.D. To summarize the resulting failure, it appears that progressive liquid side cracking combined with gas side erosion weakens the tube to the point of failure.

2. A nondestructive inspection method is needed for measuring the inner wall (gas side) thickness and the detection of minute cracks inside the tubing.

Minimum calculated wall thickness to sustain operating pressures is approximately 0.010". However, it is desirable to detect and attempt to arrest internal nitriding and stress corrosion before effective wall thickness is reduced to less than 0.020". This could be accomplished if a suitable nondestructive inspection method was available for field usage. Marginal chambers would then be removed from service and repaired by recoating prior to complete tube failure.

3. A nondestructive inspection method is needed to inspect the quality of weld performed on ruptured tubes prior to recoating.

The welding of ruptured tubes using Heliarc (Inert-gas) process and argon gas as a back up to prevent possible drop through is currently being evaluated. Also the use of radioisotopes are currently being evaluated as a possible nondestructive inspection medium.

Current Development Effort:

1. In view of the present average chamber life of 18 minutes, the Air Force and NASA are currently evaluating an alternate coating process which has been applied to two (2) repaired chambers. These chambers will be subjected to ground static engine operational tests. The new process consists of stripping the present coating by abrasive cleaning, welding ruptured tubes, pressure checking chamber for quality of weld and leak test. A metallizing undercoat of molybdenum and four (4) layers of graduated nichrome and zirconia as follows:

(1)	Molybdenum	0.002" - 0.0004"
(2)	70% Nichrome - 30% Zirconiz	0.002" - 0.004"
(3)	30% Nichrome - 70% Zirconiz	0.002" - 0.004"
(4)	10% Nichrome - 90% Zirconiz	0.002" - 0.004"
(5)	100% Zirconia	0.002" - 0.006"

Both the molybdenum and the nichrome zirconia layers are sprayed with a Plasmadyne plasma gun modified to completely shield the high velocity plasma arc with inert argon gas. Maximum temperature with the plasma gun is 13,000°F and the plasma arc velocity is also higher than the oxy-acetylene gases used in the flame spraying technique. It is thought that the refractory subcoat bond is a mechanical phenomenon and can be greatly improved by increasing the deposition velocity of the fused refractory particles. Coating density also appears to be a function of deposition velocity. Thus the plasma gun seems to offer several distinct advantages as a tool for coating applications for rocket chambers.

2. The Propulsion Branch, Air Force Flight Test Center, is currently conducting tests utilizing X and gamma radiography as a nondestructive inspection medium to attempt to provide a solution to the forementioned inspection problems. In addition a defective thrust chamber has been made available to be disected to provide laboratory physical samples of unsatisfactory tube conditions. Subject samples will be released to vendors of nondestructive inspection equipment (e.g.; eddy current and/or ultrasonic types) for test and evaluation.

Summary:

The problem defined concerns premature failure of the inner control surface of the thrust chamber on the XLR-99 engine. Development of improved coating procedures may alleviate this problem. But it is highly desirable to find adequate nondestructive inspection techniques to measure coating thickness and the condition of underlying tube walls, in order to permit timely repair or replacement of chambers which will least interfere with the X-15 Flight Research Program. Undoubtedly such inspection techniques, when developed, will have application in other areas.

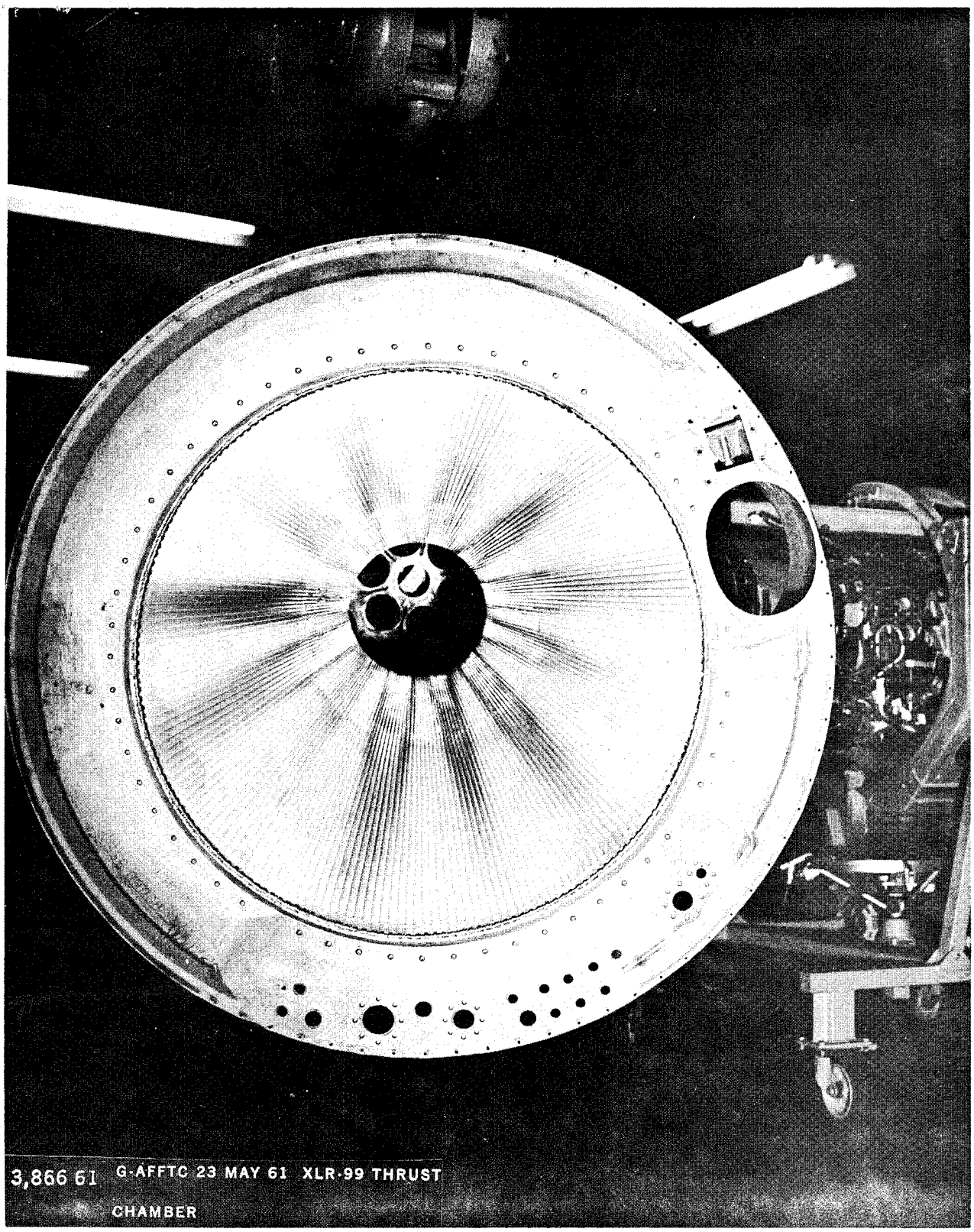
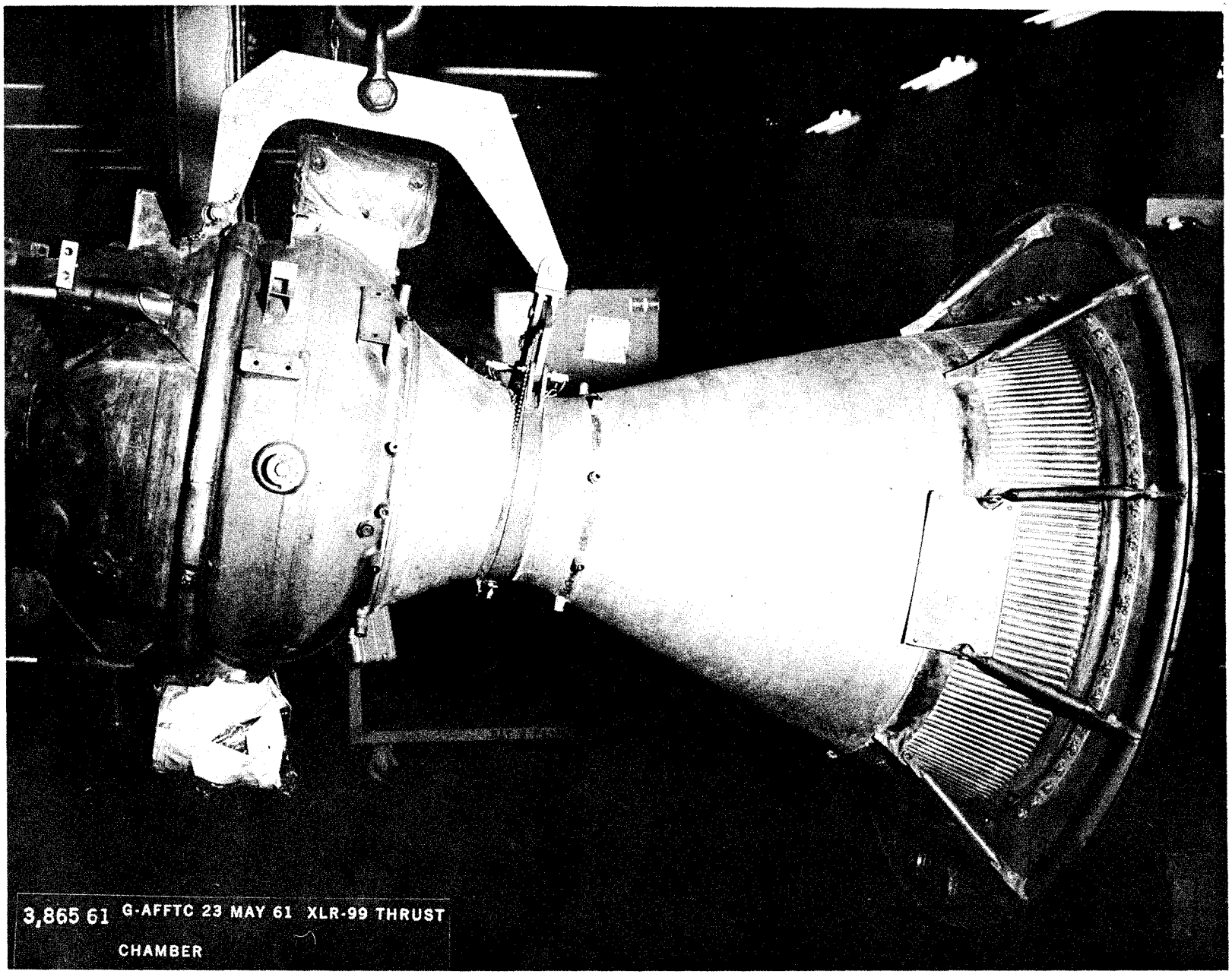
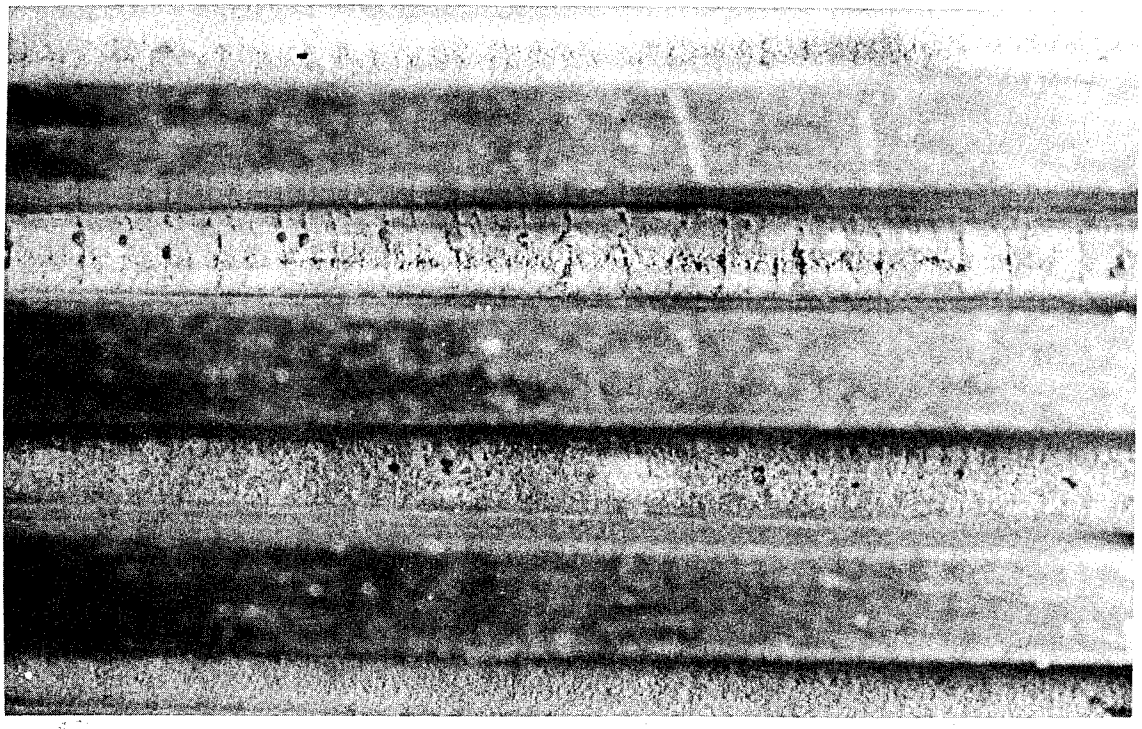


FIG. 1

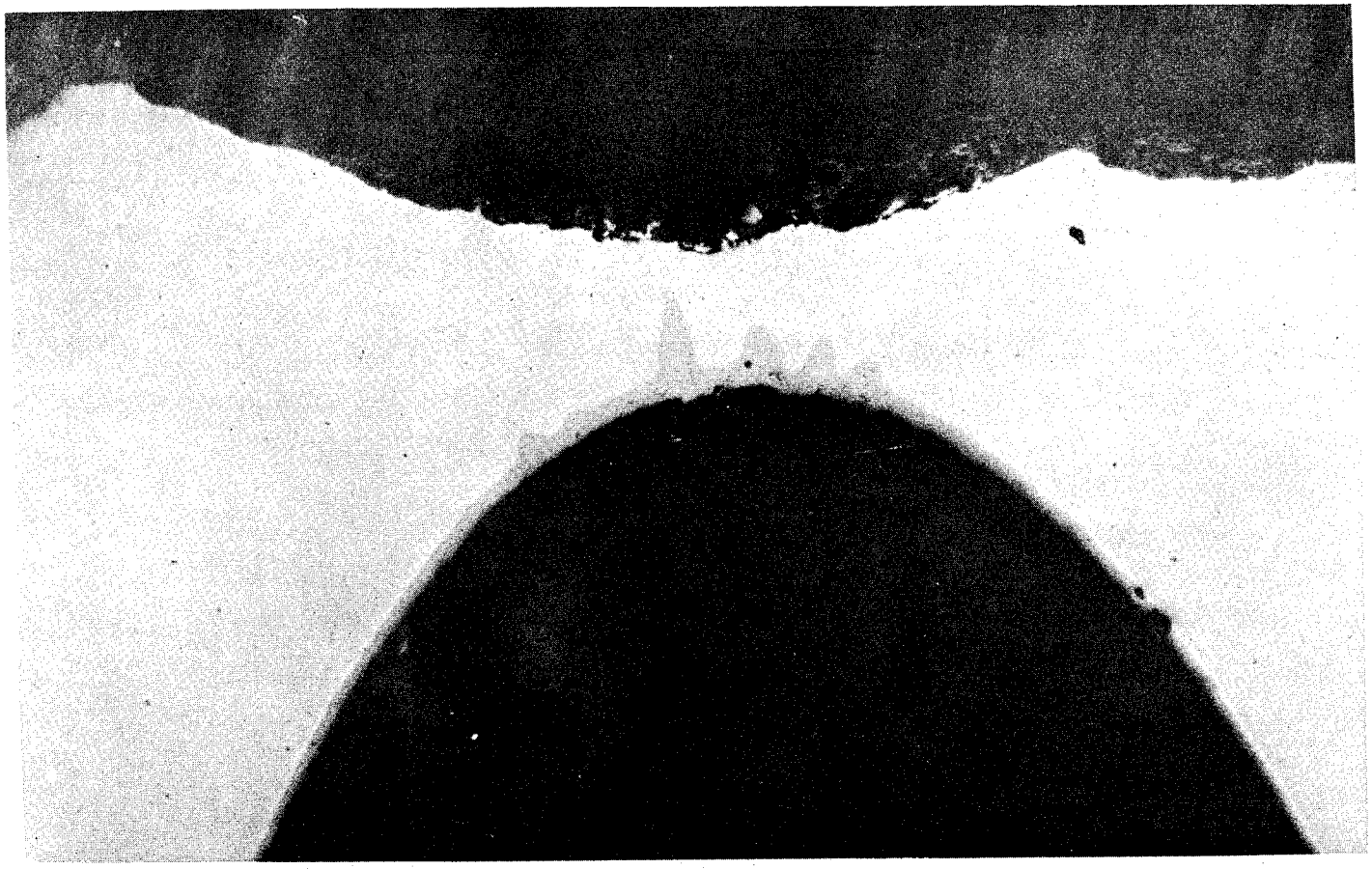

3,865 61 G-AFFTC 23 MAY 61 XLR-99 THRUST
CHAMBER



10X Photo No. 362-L

15X Photo No. 358-L

FIG. 3



100X Photo No. 1780

Photomicrograph of 347 stainless steel tube showing result of high-temperature erosion on the O.D. This occurred after the Rokide coating eroded away thereby exposing the tube. Note also the darker material on the I.D. (unetched).

FIG. 4

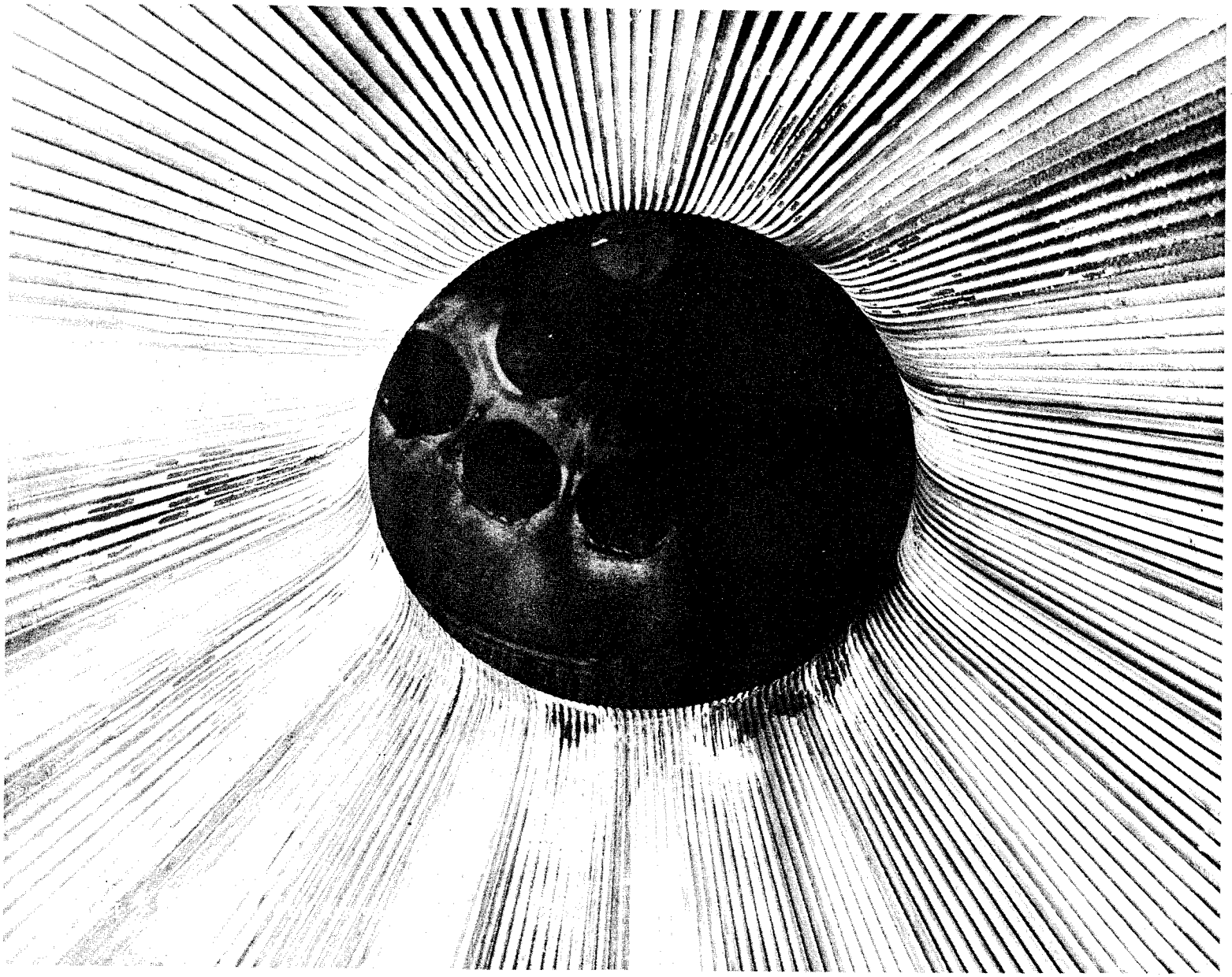
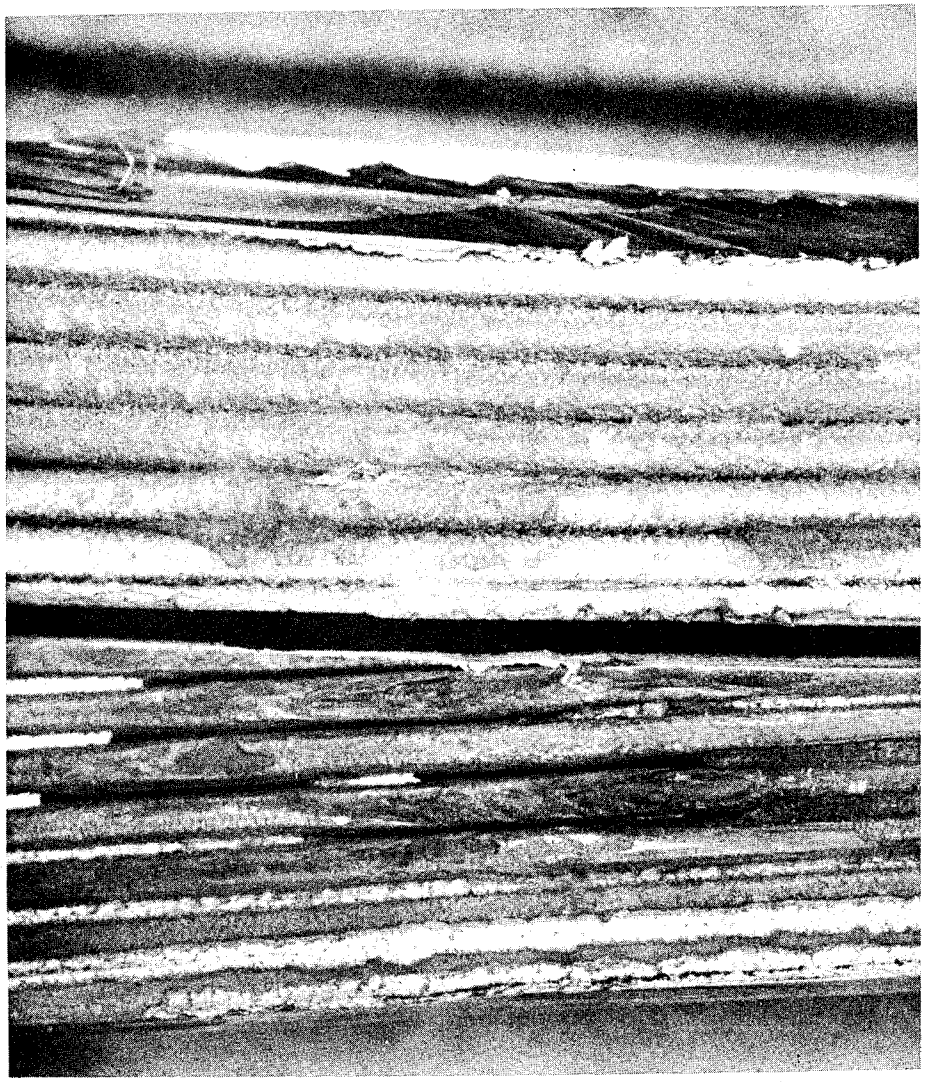


FIG. 5



1X Photo No. 357-L

Upper part of photomacrograph shows Rokide remaining on the tubes at throat. Lower part shows bare tubes (Rokide coating removed by erosion). Both tube sections were immediately adjacent at the throat section.

FIG. 6

REPORT ON NONDESTRUCTIVE TESTING IN PLACE INSPECTION OF FERROUS BOILER TUBES

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I. Description of Boiler

In Naval service, boilers are designed and constructed so as to produce the maximum steam generation for the minimum size and weight. Consequently, there is little, if any, extra space provided for accessibility. Basically, these boilers consist of a water drum and a steam drum connected by a large number of tubes that generate steam when heated. The water drum is of the order of two feet in diameter with an access opening of about eleven by fifteen inches in the end, just large enough for a man to enter. The steam drum is somewhat larger, about four feet in diameter, with the same size access opening. The tubes that connect the two drums are mechanically rolled into holes through the sides of the drums so that access to the water side (inside surface) of the tubes is limited to through the ends of the tubes from inside the water and steam drums. Access to the fire-side (outside surface) of the tubes is practically impossible since as many as twenty-six rows of fifty or more tubes are arranged in the tube nest with only a fraction of an inch between them.

II. Local Inspection Practice

At the present time, the inspection of ferrous boiler tubes on Naval vessels is conducted by removing specimen tubes from the boilers during overhaul periods. The specimen tubes are checked for blisters and ductility and then split and inspected for location, depth and type of water-side corrosion, and water-side deposits. Generally, some pitting type corrosion is found in all specimen tubes, however, the Bureau of Ships has set a criteria of fifty per cent penetration as the maximum allowable penetration. At times scale and sludge deposits are found on the water-side surface of the tubes.

In addition to the above conditions, seams and die marks are found in some of the specimen tubes and also in some new tubes prior to installation in the boilers. The seams are the results of welding procedures used in the manufacture of welded tubes. The die marks are the results of imperfections in the surface of the dies used in drawing the tubes. No reliable inspection method is presently available that will adequately indicate the severity of these conditions along the entire length of the tubes when inspected in place in the boilers.

III. Desirability of Method for In Place Inspection

A method for in place inspection of these tubes would effect a considerable saving in time, money and material. At present, the extent of tube replacement is not determined until exploratory blocks of a hundred or more tubes are inspected. This requires several days and at times additional tubes must be removed, depending upon the results of the inspection. The removal of tubes continues until the inspectors are satisfied that the extent of the harmful conditions has been determined and that all unsatisfactory tubes have been

removed from the boiler. The removed tubes must then be replaced and the boiler hydrastatically tested. Over the period of a year's time it is not uncommon for as many as forty boilers to be inspected in this way at our activity.

IV. Description of Material To Be Tested

The tubes to be inspected are low and medium carbon steel, one to two inches in diameter with 0.085 to 0.175 inch wall thickness, and 2-1/4 per cent chromium 1 per cent molybdenum alloy steel, one inch in diameter with 0.100 to 0.150 inch wall thickness. The carbon steel tubes vary in length from about 12 to 15 feet and have bends with radii that vary from 6 to 36 inches. The alloy steel tubes are from 15 to 16 feet in length and have bends with radii that vary from 3 to 9 inches. The inside surface condition of the tubes varies from one that is smooth and scale-free to one or more of the following conditions:

a. Thin metal oxide
b. Nonadherent powdery sludge
c. Hard oxide shells over localized pits (oxygen scabs)
d. Thick, hard heat scale

V. Past Attempts at Solution of Problem

Several years ago a representative from our laboratory visited Watertown Arsenal to look into the practicability of using a "magnetic flaw detector" developed by the Arsenal personnel for the inspection of gun tubes. It was learned at that time however that the device in its then state of development was not suitable because of lack of sensitivity and reproducibility. Recent correspondence with the Budd Co. revealed that an instrument produced by them the RADAC was possibly suitable for the inspection of nonferrous tubes but was not applicable to the inspection of ferrous boiler tubes. The problem was presented to the Bureau of Ships who in turn referred it to the Navy Boiler & Turbine Laboratory, which reported that possibly the use of glass fiber-optics and miniaturized cameras would be the solution to the problem. Currently, a Boston Naval Shipyard method involving radiography is used but with only limited success. Only the outside rows of tubes can be inspected by this method and only pitting can be evaluated.

VI. What is Desired

As a solution to this problem, it is desired to have a probe that can be inserted into the tubes in place in a boiler. The probe should be connected to an indicating device that could be used to evaluate the presence of deep scratches and seams, water-side corrosion and deposits, changes in ductility and the presence of bulges.

TESTING FOR HOMOGENEITY AND BOND OF BRAZED JOINTS

BY

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The Chemical Corps does not claim to be unique among the services charged with specific missions within the complex of the Department of the Army, but, like Signal Corps, Ordnance Corps, the Engineers and others, the Chemical Corps operates in a specialized field, and this specialization, in itself, does cause the Chemical Corps to be confronted with problems which are unique. Many problems peculiar to the Chemical Corps could be cited as examples - they would apply to both equipment and munitions - but only one has been chosen for discussion as being most applicable to the purpose of this meeting. This problem, which has been of considerable concern to the Chemical Corps, is how to evaluate the quality of brazed joints employed in toxic-filled munitions which would assure our obtaining homogeneity and, therefore, increased reliability of minimum or zero leaks of toxic liquids.

We have two sources of our problems - one is of our own making and one is not. In many cases the Chemical Corps works with an item "from cradle to grave" ... that is, from research through procurement to eventual obsolescence. Other times, items are given to us for "adoption."

To satisfy certain logistic requirements, on one hand, the Chemical Corps, as often happens, might be charged with full responsibility for research, development, and procurement of a specific kind of toxic-filled munition. That is, a certain agent, which is known to have certain affects, would be required for a specific tactical application, to be used in some prescribed manner. It then becomes a comparatively simple matter for the Chemical Corps to control the design of the container so that leakage cannot occur, or will be held to a minimum within certain acceptable limits. Putting it another way, we are saying that toxic-filled munitions must not leak! But, to be practical, a safe and measureable limit is established and determined through the use of The Standard Helium Leak Test. This test says that a leak in excess of 1×10^{-6} cubic centimeters per second at standard atmosphere, which is about $2\frac{1}{2}$ milliliters a month, is too much, or is cause for rejection of a manufactured item. Less than this amount of leakage can be tolerated since normal atmospheric dissipation renders the agent comparatively harmless...but any leaks are undesirable.

In the design of these containers - and in many other items - the Chemical Corps resorts to different kinds of seals and joints incorporating brazing; special techniques for forming and brazing have been developed by the Corps, for the application on hand, to insure that good seals will result. But, even so, there is not always an assurance that homogeneity of the brazed joint or bond is obtained.

But, on the other hand, there are many occasions when the Chemical Corps is asked to take some item from another service, already designed, already degugged,

and which has been or is being procured as an explosive or non-liquid, non-hermetically sealed item, and to build this into a toxic-agent munition. Little, if any, change can be made to basic configuration or design. One example of this situation is the M23 Land Mine. You have sketches of this mine in your handouts and there is a cut-away sample here for your examination. It is labeled with the experimental indentification, E5, but it is the M23.

The M23 Chemical Land Mine was developed in response to a requirement for a toxic chemical land mine which could be utilized in area contamination in conjunction with high explosive anti-tank and anti-personnel mines. The use of chemical mines in high explosive minefields greatly increases the difficulty of the enemy in his advance. The high explosive mines slow his advance, thereby exposing him to the agent for longer periods, and the agent slows operations by forcing clearance personnel to wear protective masks and clothing. The M23 Mine had to conform in external configuration to the M15 Ordnance Anti-tank Mine and to utilize its standard fuzing and firing devices! This conformance to the M15 Ordnance Mine allows the M23 Chemical Mine to be planted and armed by existing and available mechanical means.

Since the item was to conform externally to the M15 Anti-tank mine and have the same activator wells, the fundamental problem was to design a bursting system into the M15 Mine to properly disperse the toxic agent.

However, concurrent with burster design investigations, the design of the external container of the mine had to be studied since the M15 Ordnance Land Mine was inadequate because it was not designed to be hermetically sealed. Ordnance requirements for leak tests are neither the same nor as stringent as required for chemical or toxic-filled munitions. Ordnance, with its explosive mine, is more concerned over excessive amounts of moisture passively seeping into the item, causing it to become merely inoperative, or less effective, whereas Chemical must be concerned with the liquids already inside, which can - and often do - build up internal pressures under certain conditions, and their leaking out of even the tiniest hole.

If this were an ordinary munition, we could merely dispose of the leakers if and when they occur. But this filling is colorless, odorless, and extremely toxic. A single droplet on the skin, if unnoticed, would be fatal within a few seconds!

From the study of the container a design resulted which eliminated as many unnecessary joints as possible, but which was still externally identical to the M15 Mine ... the amount of changing permitted was extremely limited.

So, after the few small changes that were permitted, there are still three critical areas which employ brazing in the land mine: they are shown in details "A", "B", and "C" in the sketch, and are labeled on the cut-away sample.

In the Chemical Land Mine, and other items using brazed joints, it has been and is possible to make a joint that will pass all tests, but, since a minimum amount of brazing material can be deposited at some point or points resulting in

a thin or hair-line seal and leaving voids, this brazing can become defective to the point of permitting an excessive leak to occur, without any effect whatever on the strength of the joint.

These brazed joints in the land mine are checked for quality, to the extent possible, through the use of the Standard Helium Leak Test, the use of coupons taken from sections to check for strength of bond, pull and shear tests, and x-ray which is not wholly satisfactory.

A special investigation was performed to determine the feasibility of using adhesives, alone or in conjunction with interrupted soldered joints, to obtain an air-and-moisture-tight, structurally sound bond between the body housing and the retainer ring of the mine. Similar investigations have been performed on other items, but the results have not been satisfactory.

The brazing method of fabrication is used on many Chemical Corps items, and in all cases an adequate evaluation of the brazed joint is needed. Evaluation can be done on a slow, one-by-one basis, or other not wholly satisfactory, destructive methods as mentioned before - as well as the completed item being subjected to helium leak test. But the evaluation should be performed at the earliest possible stage in production where detection of faulty material would preclude it from being further incorporated into assemblies.

We believe this can be done by nondestructive testing methods. We are looking for a nondestructive test to determine the homogeneity of the braze in the joint and the quality of the bond ... a test which can be applied to at least 50 land mines an hour ... a method, or device, which will be adaptable to a production line, require minimum maintenance, and be able to be used by semi-skilled operators.

By discussing some general problems and those of the toxic-filled land mine, we have considered some of the brazing applications where testing is difficult or costly - or both. We feel that a successful application of nondestructive testing to the brazing on the land mine could be applied to the inspection of many other Chemical Corps items, and higher over-all reliability could be realized.

10969-04

"NONDESTRUCTIVE TESTING OF TEFLON BLADDERS FOR LONG TERM CONTACT WITH HYDROGEN PEROXIDE"

by
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U.S. NAVAL UNDERWATER ORDNANCE STATION
Newport, R. I.

The first problem to be considered is "Nondestructive Testing of Teflon Bladders for Long Term Contact with Hydrogen Peroxide". The bladder of which I speak is shown in figure 1. It is hemispherical in shape - approximately 20" in diameter and 7 - 10 mils thick. On the flattened end, it has a 6" diameter opening equipped with a flange for securing it to a bulkhead. The bladder is made by spraying or dipping Teflon dispersion onto a disposable form and subsequently sintering this coating. This process is repeated as often as necessary to obtain the required wall thickness before the form is removed. The next two figures (figures 2 and 3) show a prototype of this bladder in the inflated and collapsed conditions. Preliminary cycling indicates that the bladder collapses in a random manner and not along the same lines or folds each time.

In use, this bladder will be collapsed and surrounded by a high strength oxidizing medium (figure 4). Maximum storage time while in this state will be six months at temperatures which may range from -65° to +160°F. The bladder will be expected to function at temperatures down to +30°F.

These then are the design features and operational environment. Now, what type of defects do we expect to encounter? Basically, we expect only two types:

1. Pin holes in the bag structure as a result of fabricating techniques.

2. Cracking of the material due either to fabricating techniques or storage in the collapsed condition.

The first type of defect - pin holes - can probably be located by a number of techniques such as

1. Pressurization and testing with a halogen torch.

2. Triboelectric tests such as Statiflux.

3. Medium voltage (150-500V) spark testers.

However, each of these has a drawback and none are considered completely satisfactory. The second type of defect, cracking, is more difficult to detect. In all probability, it will not be present initially, but will arise during functional testing of the bladder.

What we are looking for in a nondestructive test then is a method which can detect the above defects without altering the properties of the bladder material nor its compatibility with the oxidizer. It should be capable of performing this function fairly rapidly. This examination will not be a field operation, but will be performed by laboratory personnel.

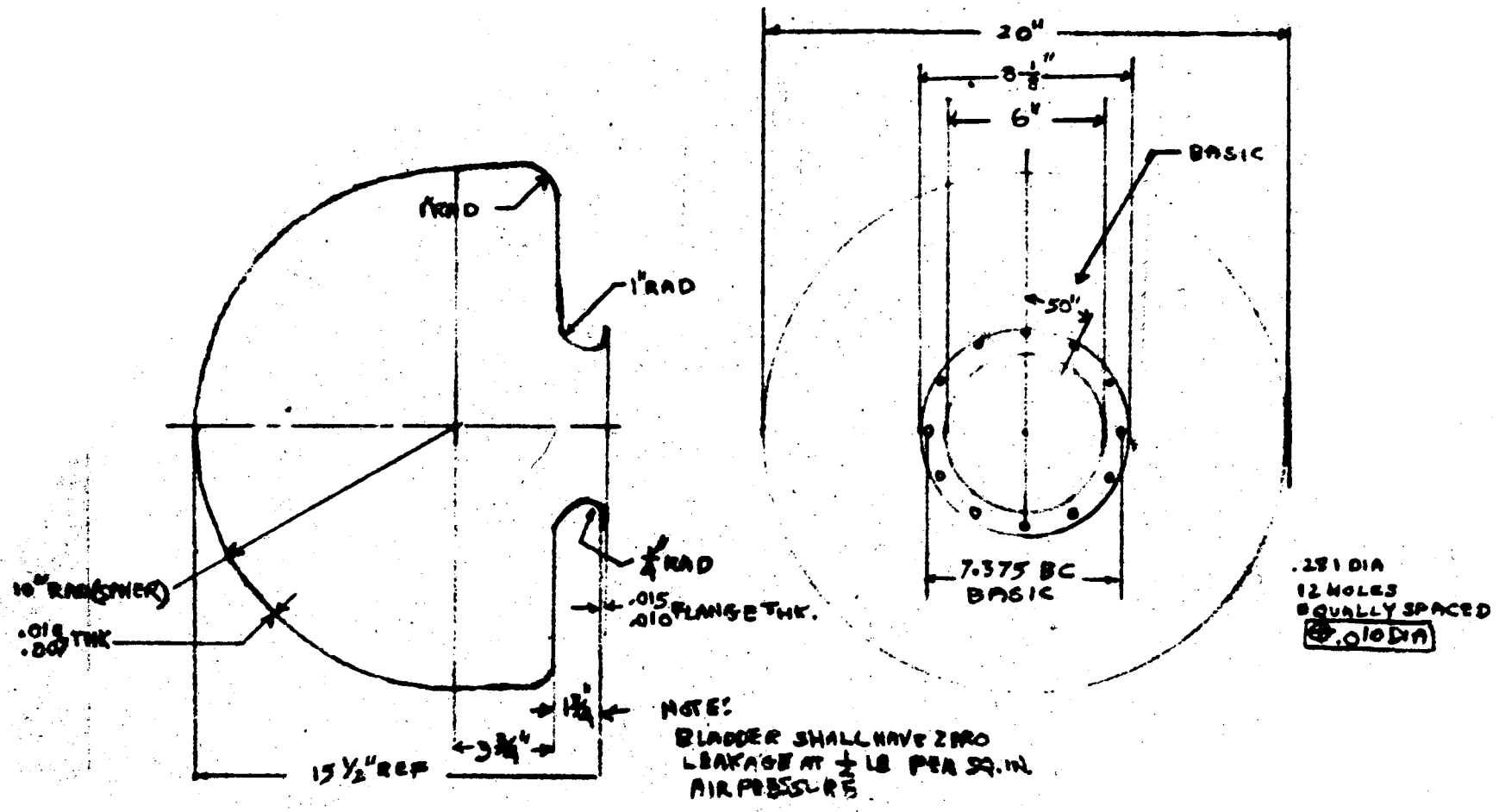
1" RAD
1" RAD
10" RAD (OTHER)
.018 .002 THK
¼ RAD
.015 .010 FLANGE THK.
1¾"
3¾"
15½" REF
20"
8⅛"
6"
BASIC
.50"
7.375 BC BASIC
.281 DIA
12 HOLES
EQUALLY SPACED
.010 DIA
NOTE:
BLADDER SHALL HAVE ZERO
LEAKAGE AT ½ LB PER SQ. IN.
AIR PRESSURE

Fig. 1

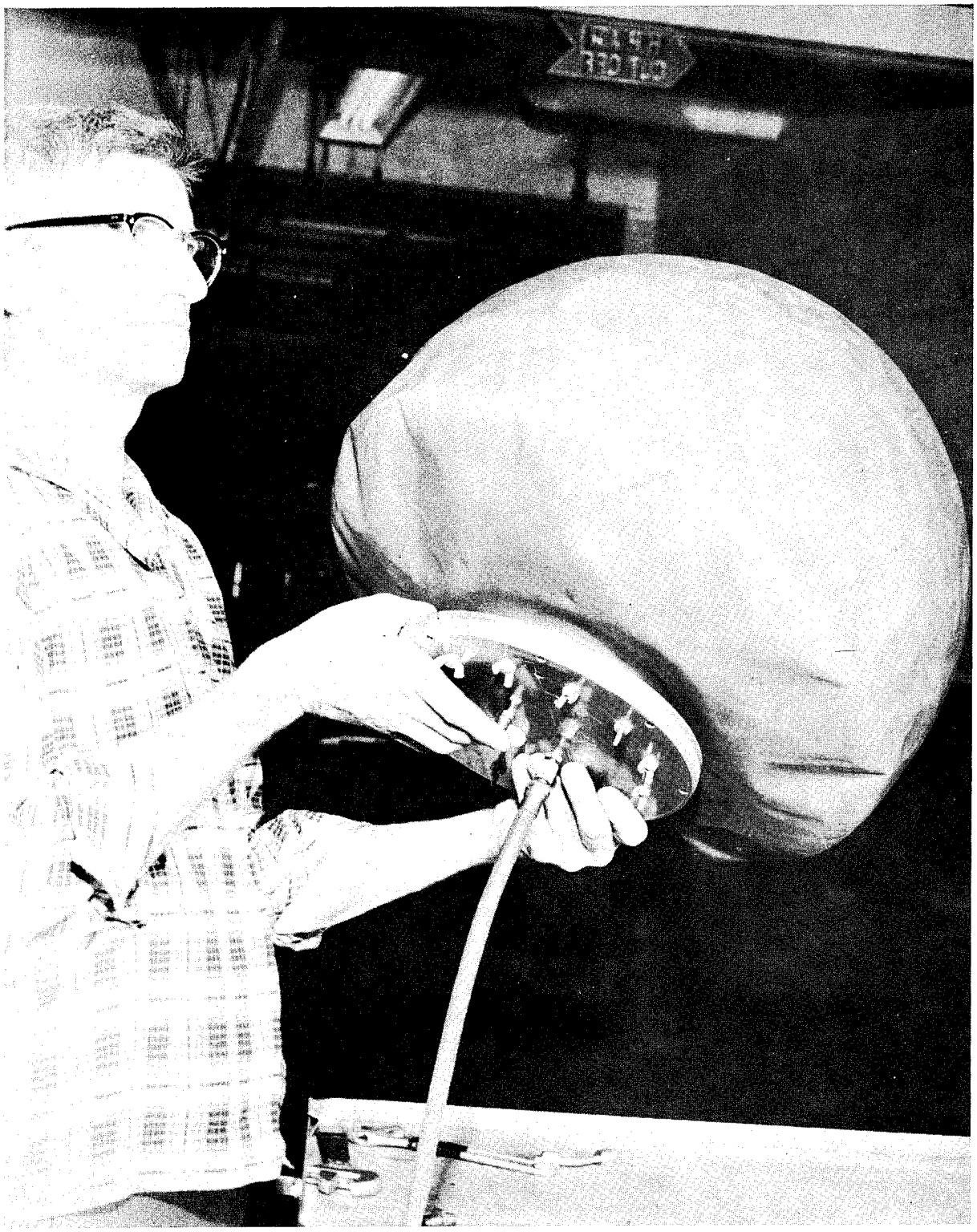

FIG. 2

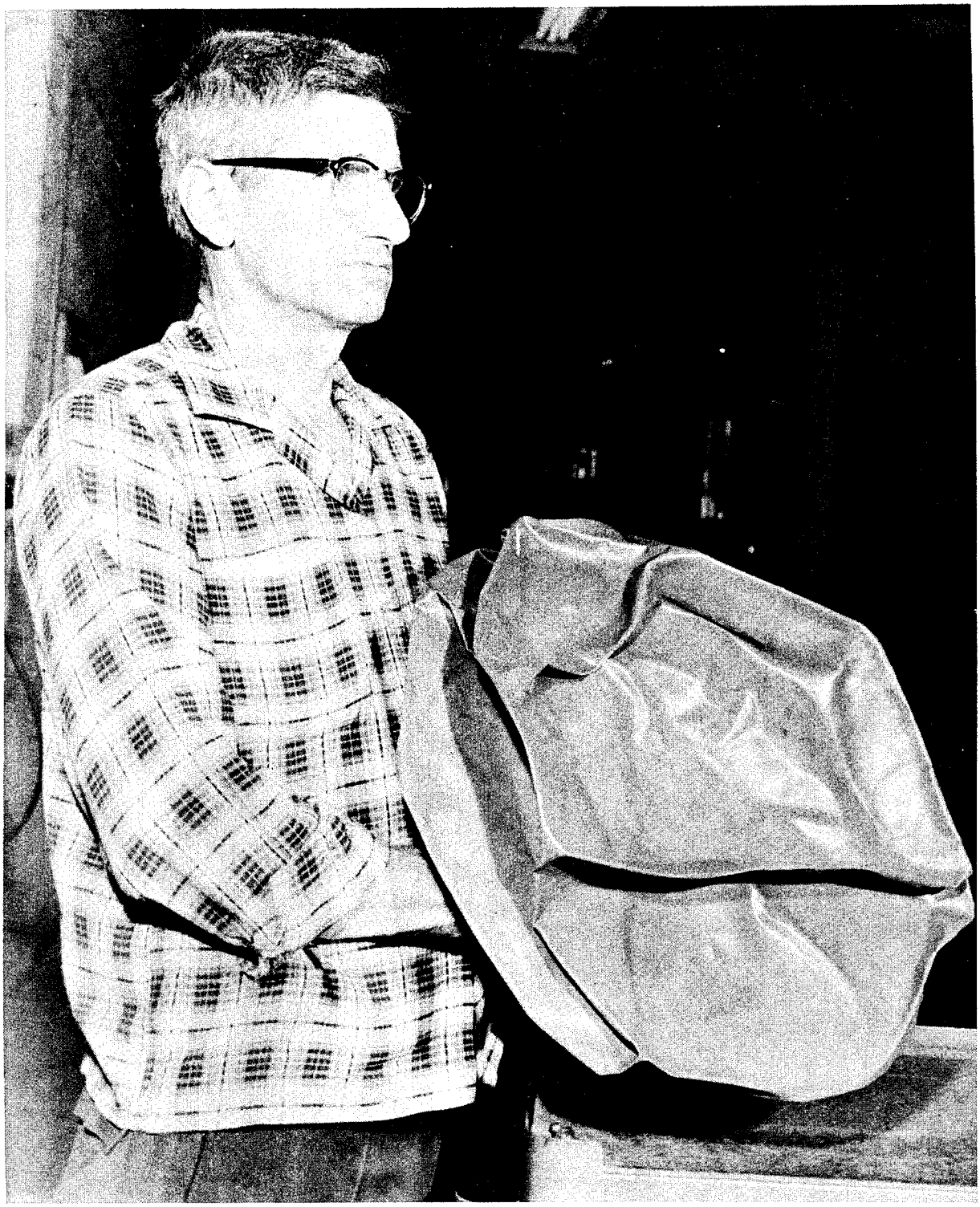

FIG. 3

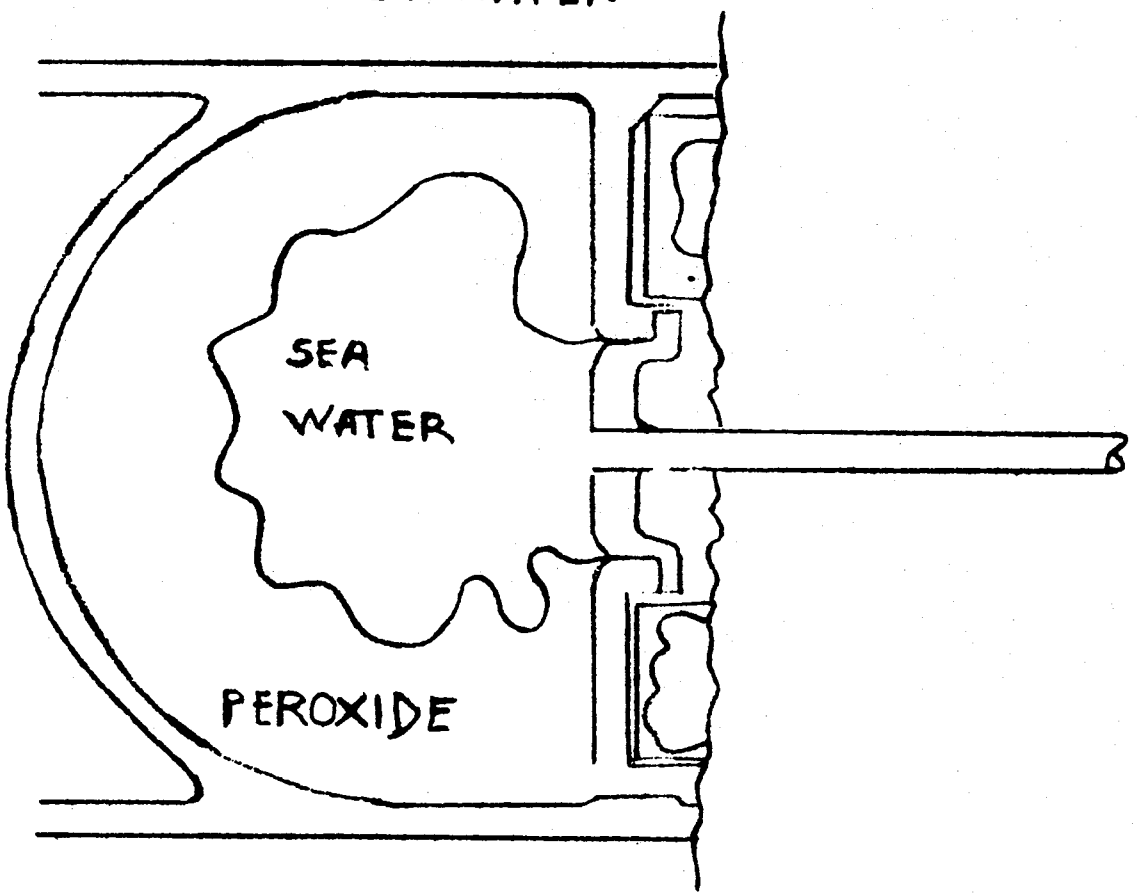

Fig 4

NONDESTRUCTIVE DETERMINATION

REQUIRED TO SEPARATE STEEL BALL FROM ALUMINUM HOUSING

(Not presented because of security reasons.)

NONDESTRUCTIVE INSPECTION FOR

PRESENCE OF HYDROGEN EMBRITTLEMENT IN METALS

INDUCED BY VARIOUS PLATING PROCEDURES

(Not presented due to nonavailability of presentor.)

10969-05

"NONDESTRUCTIVE METHOD FOR DETERMINING IMPACT STRESSES AND WATER ENTRY ANGLES OF SMALL AIR DROP MISSILES".

by
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U.S. NAVAL UNDERWATER ORDNANCE STATION
Newport, R. I.

The second problem area in today's discussion is to find a simple, inexpensive method for determining impact stresses and water entry angles of small air dropped missiles. There are a number of configurations within this missile grouping; the simplest nose shape is shown in figure 1. This unit is 18" long 3" in diameter, and when loaded weighs approximately 7 lbs. It is dropped from a height of 150 feet from an airplane traveling 250 knots/hr. Impact velocity is estimated at about 175 knots/hr (300 ft/sec). While the air drop trajectory can be computed mathematically, the deviation from trajectory angle due to a sideways motion (yawing) cannot be so determined. The size of the unit is such as to preclude use of photographic devices at least of the capabilities available at NAV UNDERWATER ORDSTA. Electrical type indicators for measuring entry angles or impact stress cannot be accommodated because of insufficient space within the unit. What little space is available is used for "pringers" which are necessary in locating and recovering test units from the water.

What is desired is some manner of obtaining at least a semi-permanent record of how the unit strikes the surface of the water and what maximum force is imposed on it at this time. We have two methods which may yield information along these lines. The first is the application of brittle lacquer coatings to the nose piece and the second is a rather crude mechanical indicator.

Some preliminary work has been done to see if either of these two methods is feasible. The first step was to construct a carriage and slide test rig to be used as a type of screening device. This equipment is shown in figure 2. The slide is constructed of aluminum channel stock; is about 10 feet long and is adjustable to produce angles from 0-90°. The next figure (#3) is a close-up of the test carriage which carries a nose section suitably weighted to simulate the entire missile. We have applied several types of brittle lacquers to the nose piece and impacted them at different angles against a sheet of moderately hard rubber. It was apparent from these results that the mechanical abuse on an exterior coating would be too severe and that better results would be obtained by applying the coating on the interior of the cap. In this manner, the coating would be put in tension during testing a direction which produces the best indications. In addition, we plan to reduce the thickness of the steel nose piece from its present 0.150" to 0.060" to increase the sensitivity of the indication.

The mechanical indicator which I referred to is a variation of the above technique which, at best, will yield approximations of the water entry angle. Figure 4 illustrates what is involved. A disc of coarse grained garnet paper is placed on the nose section and overlaid in succession with a disc of carbon paper and one of plain white bond paper. Over these discs is placed a flexible

plastic cap to hold them in place and keep out the water. On impact, an impression is registered on the white paper by the grain of the garnet paper pressing against the carbon paper. An illustration of this type of indication is shown in figure 5. These results are crude, but they do serve to show large differences in entry angle and may have application in gaining an idea of what range of values the entry angle encompasses.

If interest continues in this problem, we plan to go to a different type of testing device as shown in figure 6. Here, we can vary the entry angle either by changing the indication of the slide wire, as shown, or by using a vertical slide wire and changing the mounting position of the test piece. In the former case, some adjustment would be required to compensate for changes in terminal velocity.

These then are two of our problems and the methods under consideration to help solve them. They are perhaps not the best methods and certainly not the only ones. Any suggestions for refining, improving, or substituting for these methods would be greatly appreciated.

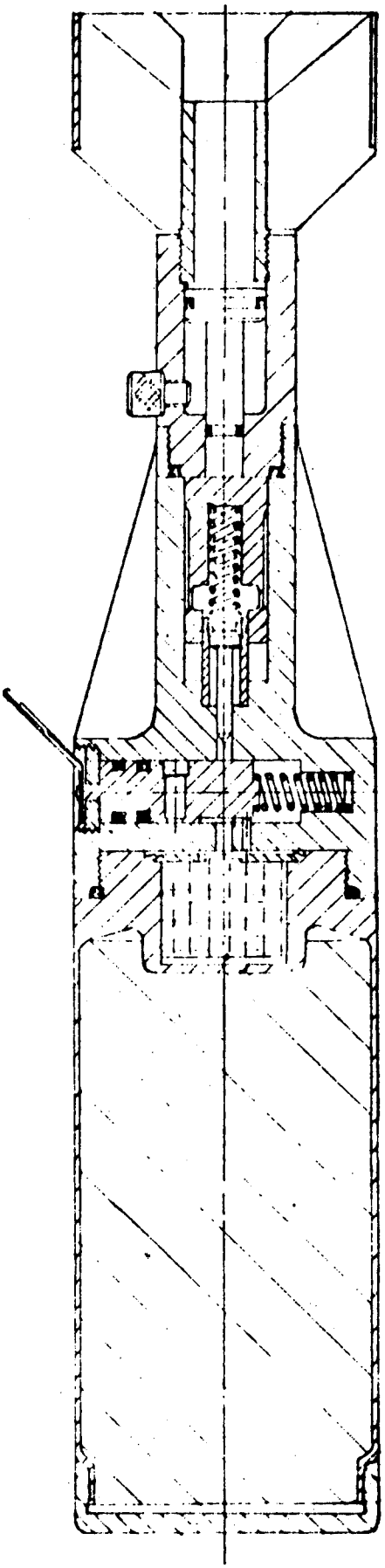

FIG. 1

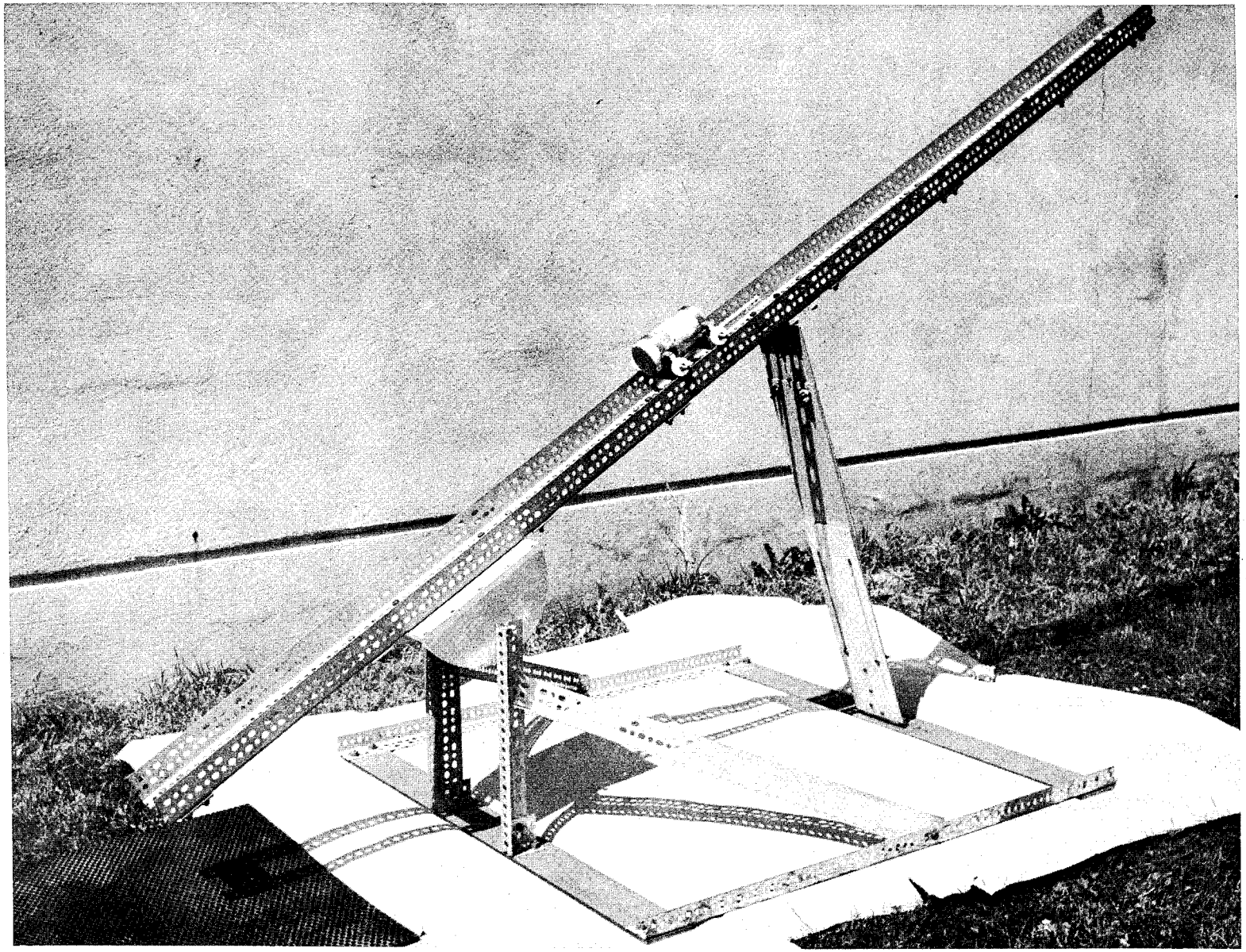

FIG. 2

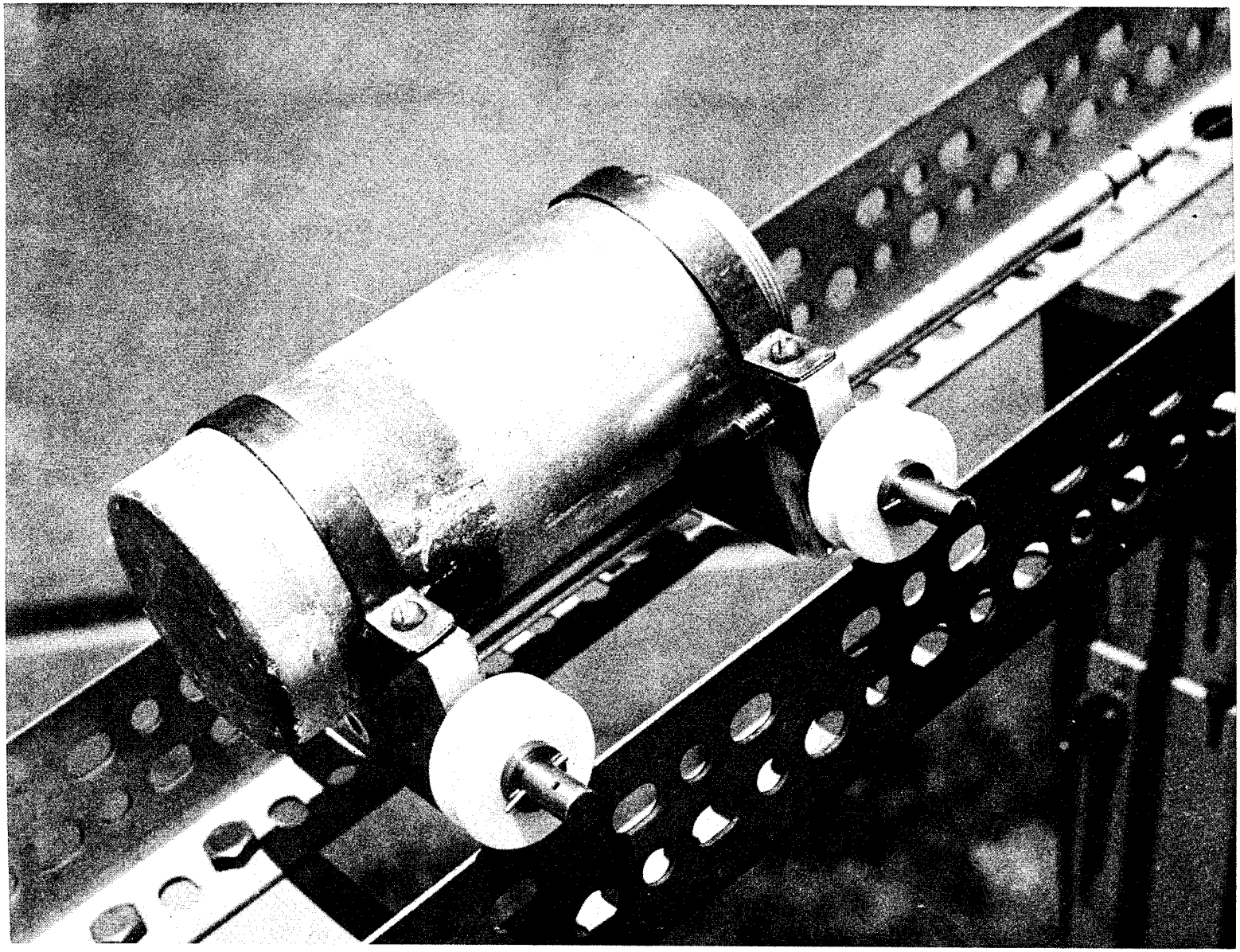

FIG. 3

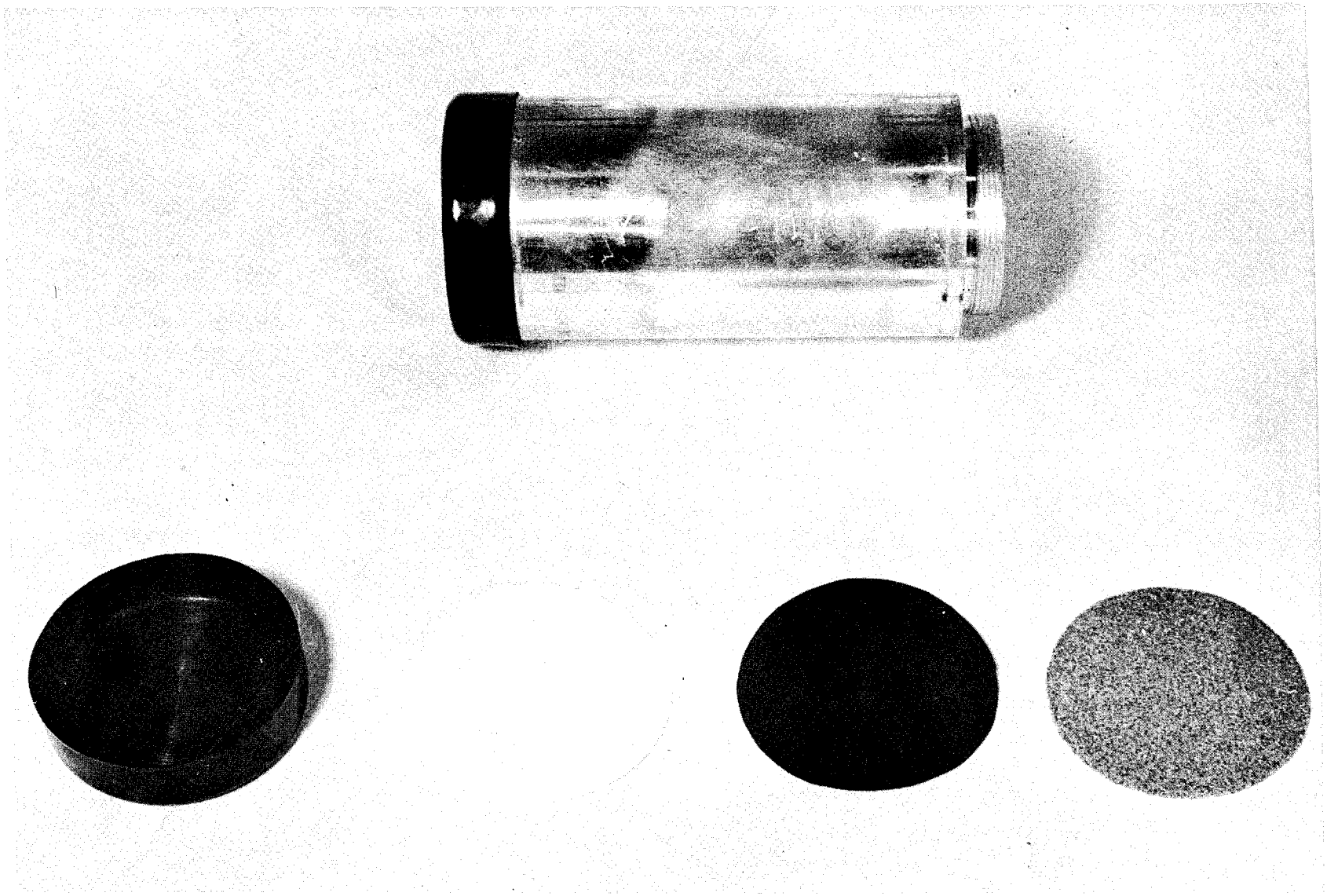

Figure - 4

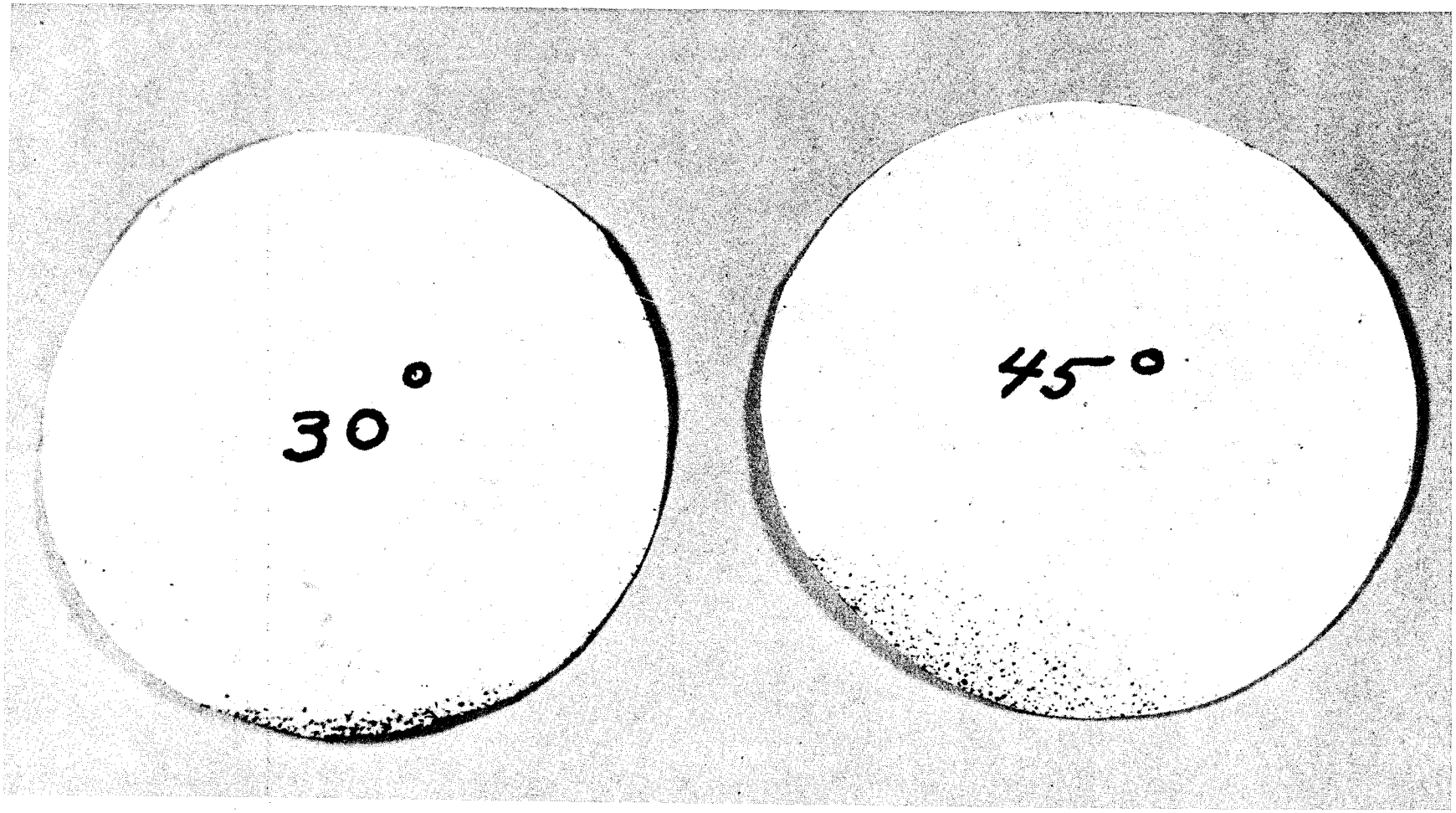

FIG. 5

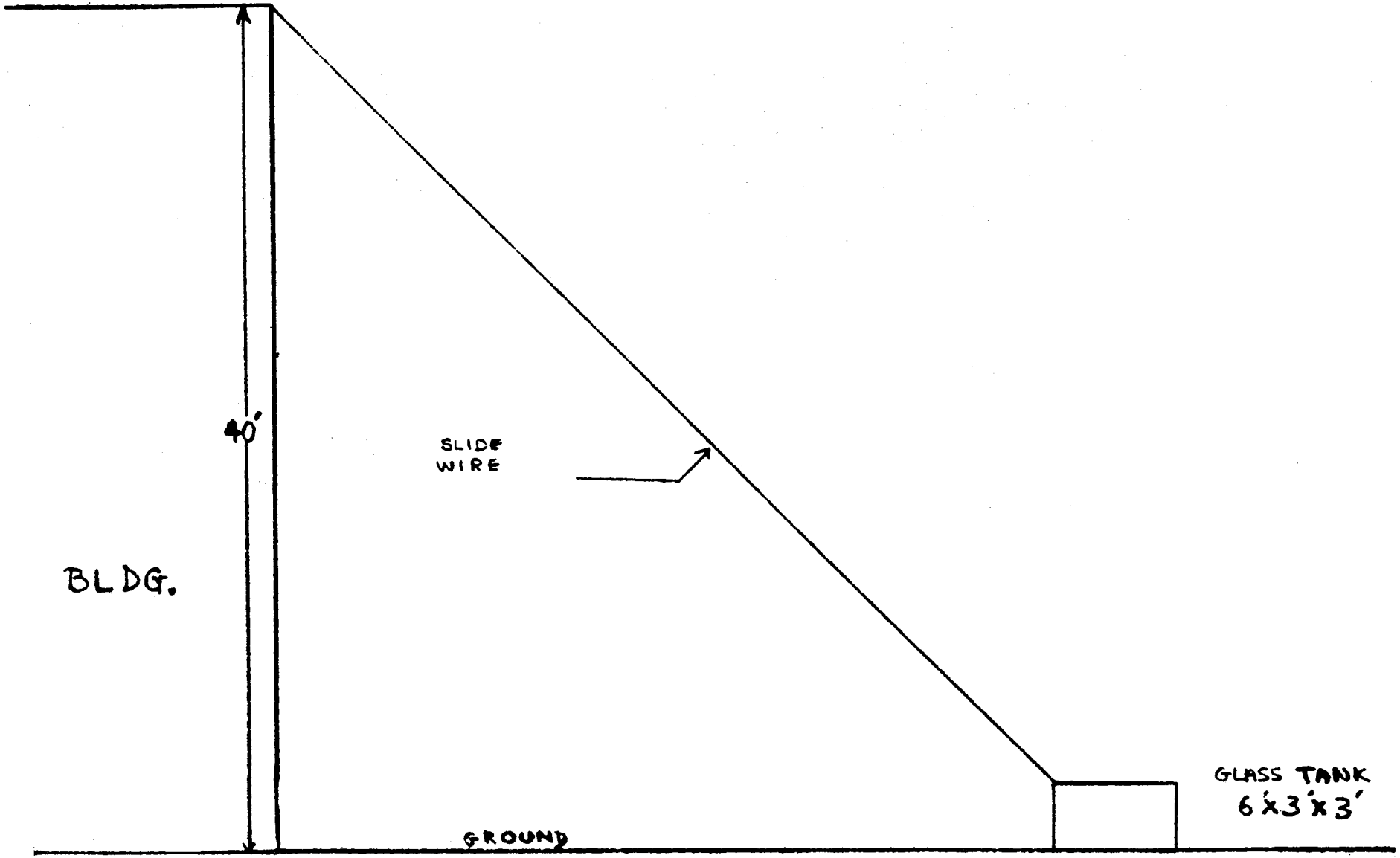

FIG. 6

NON DESTRUCTIVE TEST PROBLEM
Evaluation of Aircraft Tie Down Chains

ERNEST T. NICOTERA
Materials Branch, Service Engineering Division
Olmsted Air Force Base, Pennsylvania

Introduction

An unsatisfactory report from a field activity concerning the failure of a tie down chain used for cargo on military aircraft precipitated the problem. An incident of a chain failure occurred during unloading which started mass Xraying and magnaflux inspection of chains, the results of which were most revealing and left a problem which as to date has not been satisfactorily resolved. Fortunately, no one was injured during the incident and there was no risk in safety of flight.

The intensive inspections which took place resulted in our receiving small bits of information, Xray pictures and a few chain exhibits. Reports stated that over 60% of available chains had to be rejected based on Xrays or other examinations which were made. In addition, an interim directive was imposed requiring double chaining on all cargo. This resulted in a serious shortage of tie down chains which affected MAAMA and prompted us into doing the work we did.

Chain Manufacture

Chains are manufactured from bar stock shaped into open links which are looped through each other, the bar ends which in turn are butt welded together. It's a resistance type weld and no filler material is used. (see Fig I) The actual link that failed never was submitted for review; consequently, the location of the fracture has never been established. Perhaps it is only normal but because of the failure the welds are under severe scrutiny and are the areas given most of the attention.

Problem

In reviewing the Xray films received from various sources, a wide range in the degree of severity and frequency was noted. These can be noted by a review of the Xray film available. Some indications we considered legitimate and serious while others could be negligible. Right then we realized that evaluation by Xray was becoming too complicated simply because of the personal judgment factor and because too many people were judging. No specific standards were in existence and none had been established for this screening. As a consequence, what one person would accept another might reject and in the meantime chains were becoming scarce.

With the comparatively few exhibits and corresponding Xray film available, a correlation study was undertaken. All chains received, both the 10,000 lb and the 25,000 lb capacity types, were re-Xrayed at MAAMA and our film was compared to those originally submitted with the chains. Basically, there was good agreement with respect to the major looking defects but with the fine details certain discrepancies were noted. However, this was not without good reason. It was

found that trying to either pick up surface indications or to eliminate them was rather tricky and could account for the discrepancies. As illustrated in Fig II the configuration of the chain and the orientation of the surface defect with respect to the film will either magnify the seriousness of it or it may not even be seen at all. The situation is further complicated by trying to make a second shot in a direction 90° to the first. The individual links have a tendency to flop over when being positioned and unless each link is marked with dykem or other suitable material there is no assurance that the second shot is not the same as the first.

Magnaflux

It was felt that magnaflux might give a more positive indication, cheaper and more convenient to interpret but after a few attempts the operation was given up because of the numerous false indications which were noticed. Magnaflux paste always seemed to hang between the links and sometimes extend beyond the overlap. It was felt that interpretations in this case would not be positive and because of the variation in patterns it would be guess work trying to label certain patterns as false and others as real ones. This would mean reviewing and judging each individual link which would be no relief.

Proof Testing

Proof teating was next tried and very interesting but complicating results were obtained. Specifications were used as a guide, which require that the 10,000 lb chain be able to withstand a 10,000 load for 30 seconds without deforming and 15,000 lb without fracturing. The 25,000 lb chain requirement called for a 25,000 lb load for 30 seconds without deforming and an ultimate of 35,000 lb. Both requirements forbid reuse of the chain if exposed to a stress beyond their yield points.

Based on visual appearance and our Xray evaluations the chains selected as most likely to fail prematurely were chosen for the pull tests. The first 10,000 lb chain selected for test had definite serious looking indications on the Xray film and, in addition, an 8 link section in which 1/3 of its cross sectional area was worn away. It was the type chain which would normally be discarded based on visual appearance alone. It was proposed to test this chain in its entire length and so it was placed in a rig attached to a dynamometer. Unfortunately, the dynanometer, the largest available, was built for a maximum capacity of 15,000 lbs. The chain held at 10,000 lbs for 30 seconds and never broke when subsequently loaded to 15,000 lbs.

Since this first chain had been stressed beyond 10,000 lbs and some set was noticed a portion of a second chain with links showing similar indications but no worn areas was tested to rupture in the laboratory on a tensile machine. It broke at 15,800 lbs load but the break did not occur at the welds or any of the indications. It broke instead at one of the links held by the grips. The grips were the vise type which drew tighter as more tension was applied. The pinch created a notch and fracture occurred through this notch.

A 25,000 lb chain section with severe looking indications was also tested on the tensile machine. It sustained the 25,000 lb load test for 30 seconds and ultimately broke at 38,000 lbs. Again, the fracture was due to the action of the vise grips and did not occur at the weld or through any indication.

Conclusion

All chains are proof tested to the respective 10,000 and 25,000 lb requirements prior to acceptance but now that we have seen Xrays of some we feel that proof testing is not telling us the true condition of the chain. From what we have seen we have become somewhat skeptical about approving their use without some additional criteria. What is needed is some medium which can be incorporated at field level for evaluating chains on some positive schedule. To date we have only been able to suggest 100% Xray coupled with a statistical plan for pull testing those with Xray indications. We are looking for a better method but it must be commensurate with economy and the facilities which can be made available at the field or operating level.

ERNEST T. NICOTERA
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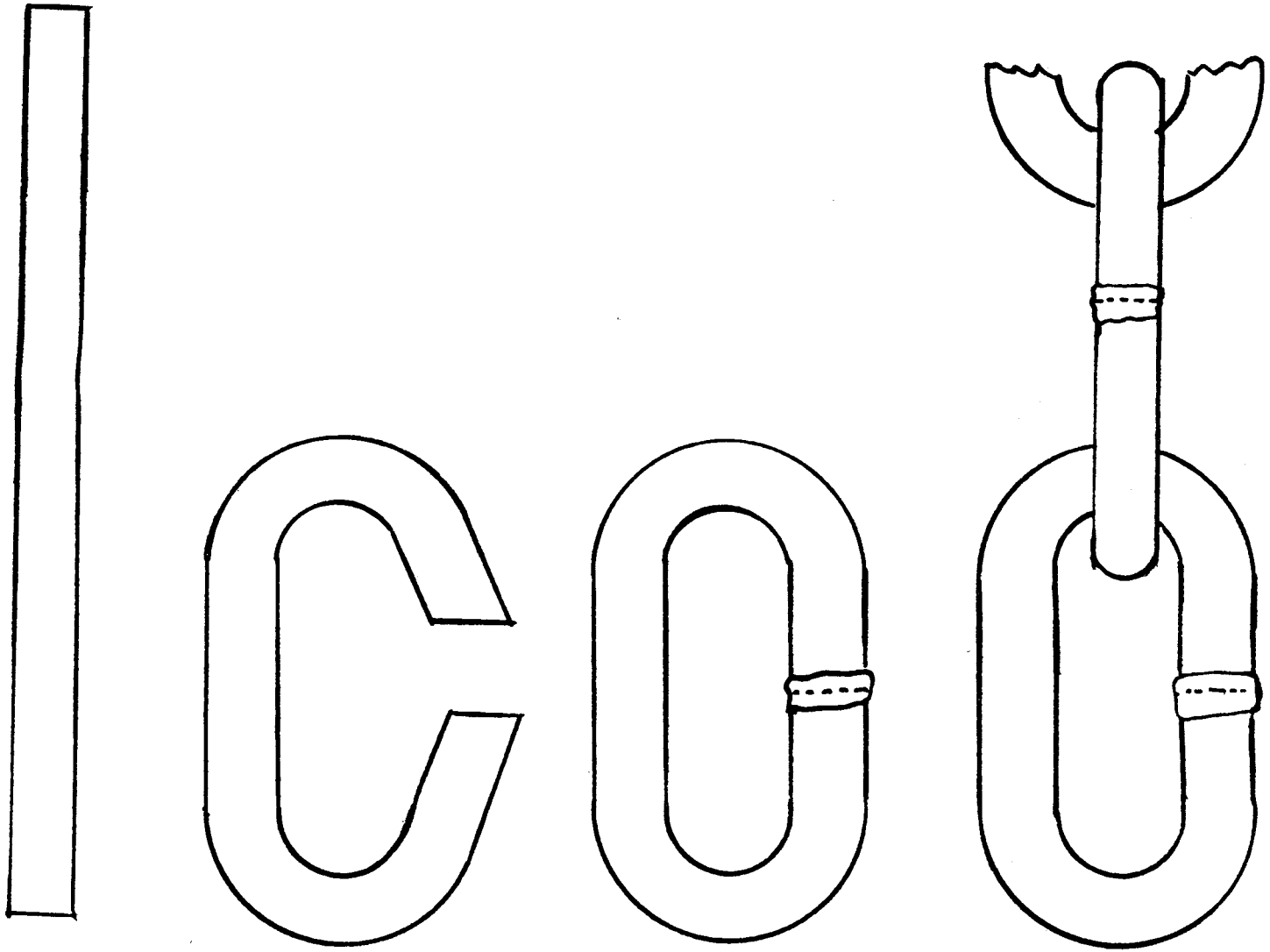
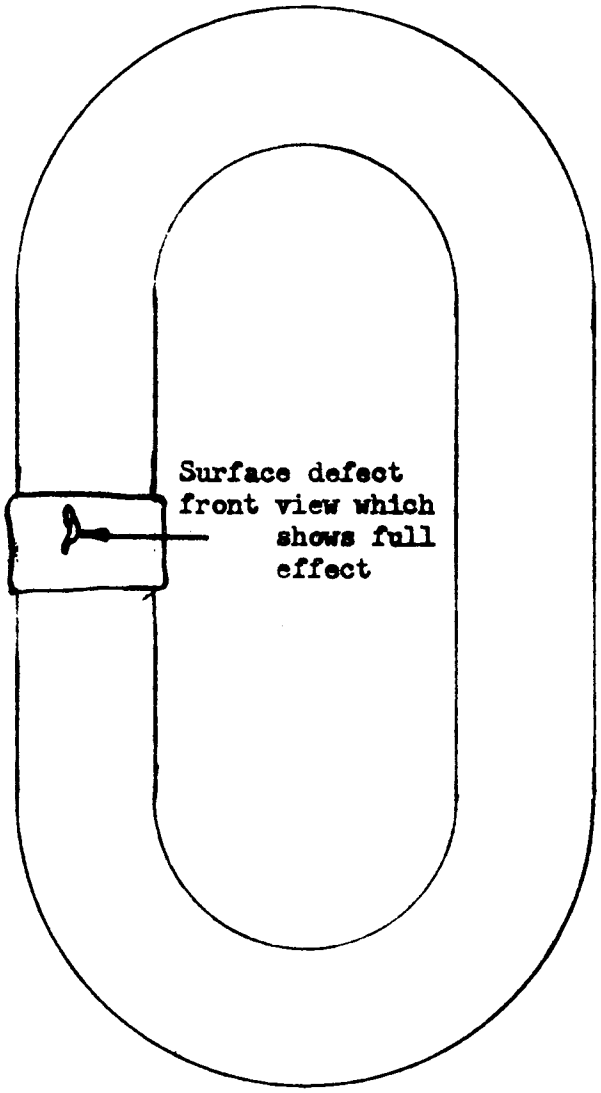
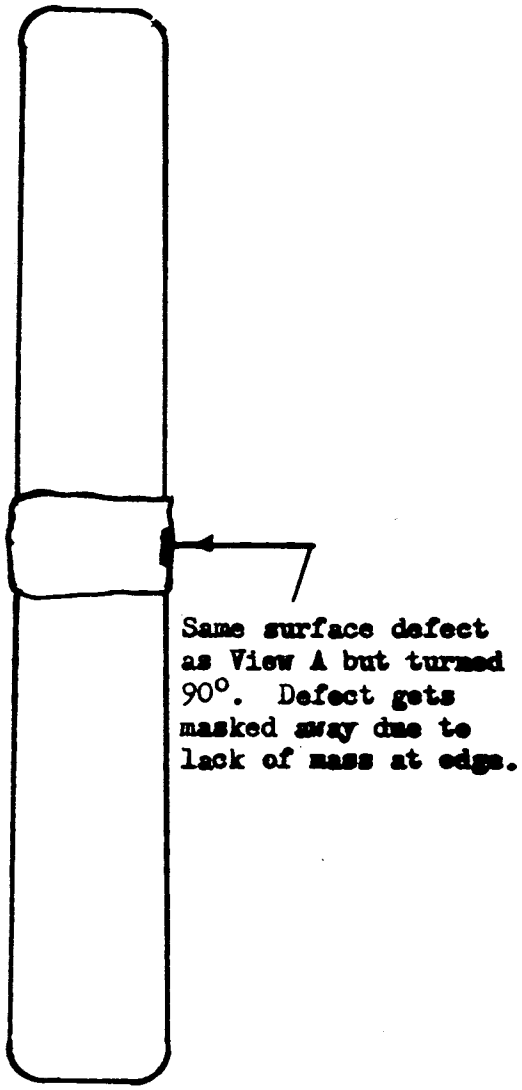


Bar Stock | 1st Bend | 2nd Bend & Weld | Linking & Weld

Fig I - Steps in Chain Manufacture



View A



View B

Fig II - Two Views of Same Surfact Defect
Illustrating Ultimate Effect on Xray Film

PANEL MEMBERS

PENETRANTS AND MAGNETIC PARTICLES PANEL

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RADIOLOGICAL PANEL

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PROBLEM:

Loss of Strength of Nylon Parachute Material due to Ultra Violet Light Degradation

PANEL REPORTS:

1. Penetrants and Magnetic Particle

Penetrant and magnetic particle methods were considered not to be applicable in the solution of this problem.

2. Ultrasonic

Ultrasonic inspection was considered not to be applicable to the solution of this problem.

3. Radiological

The Radiological Panel considered that X-ray defraction or spectro-analysis may be applied on sample fibers taken from critical areas of the parachute material. Mr. S. Goldspeil, New York Naval Shipyard, Brooklyn, New York, offered to undertake an experimental approach to this problem if samples of the materials were furnished. The Middletown Air Materiel Area agreed to make this material available to Mr. Goldspeil.

4. Others

The Others Panel considers that this problem requires an approach different from that applied to metals. The following are methods which are considered as possibilities:

a. Use of attached test strips or strip areas which are removed at various time intervals for physical or chemical testing of degree of deterioration. However, this was considered as impractical due to interference in deployment of parachutes.

b. Examination by UV (Black) Light for detection of changes in fluorescent color due to deterioration. This could be expanded to spectral analysis of fluorescent light.

c. Use of fugitive dyes on fabric and periodic measurement of changes in dye color as a measure of atmospheric exposure. This approach is applicable only to new parachutes.

d. Periodic measurement of some pertinent optical property such as index of refraction as a measure of deterioration. Report WADC 60-253 and Report of National Bureau of Standards of work done from 1950 through 1952 for Quartermaster Corps on the analysis of the degradation of vinyl and nylon polymers are reference documents of basic work on this subject.

PROBLEM:

Nondestructive Inspection of Potted Electrical Multipin Connectors for Flight Environment Adequacy

PANEL REPORTS:

1. Penetrants and Magnetic Particle

This problem was first discussed with respect to determing short circuits by penetrants and magnetic particle methods. For this requirement penetrants and magnetic particle methods are considered as not applicable. With reference to air bubbles and trapped moisture, magnetic particle methods were considered not to be suitable. Penetrant fluids could result in detrimental conditions which might interfere with the serviceability of the connectors. Penetrants, therefore, are not considered as a satisfactory solution. "Radiflow", a pressurized radioactive gas penetrant conceivably might detect bubbles and moisture. The helium leak detector was discussed and may be used in solving this particular problem. It was suggested that individuals at Frankford Arsenal and the Army Chemical Center be consulted concerning this method. High definition fluoroscopy, while not germane to the penetrant and magnetic particle methods, may be used to detect bubbles and moisture.

2. Ultrasonic

The complex configuration of these cable connectors precludes ultrasonic inspection using currently available techniques. Tensile testing for continuity and the measurement of electrical resistance or capacitance were suggested as possible solutions of this problem.

3. Radiological

The present X-ray inspection method is considered to be the only practical approach for this particular problem. As suggested by Doctor Bujes, the X-ray tube or meter is placed as close as possible to the connector cables. Additional research should be considered to produce a greater degree of separation of the connector pins, wires, and voids utilizing one of the recently developed magnification techniques. This would permit films which could be more easily read and interpreted. Another theory on this was the possibility of magnification through radiography. Mr. Holloway of the Naval Ordnance Laboratory agreed to study this problem and to conduct tests to define magnification requirements. As radiographic techniques have been tried, Dr. Bujes offered additional suggestions for improvement of present methods. Primarily, place the radiation source in closer proximity to the materials being inspected. Interpretation will remain very difficult and numerous radiographs will be required because of the orientation problems.

4. Others

Presence of visible cracks and discontinuities may be checked by use of low power stereo microscope. For other defects, use of Statiflux, Magnaglo,

or induced sonic vibrations is recommended. Corrosion of connectors after soldering may be detected by use of radioactive plated films. As previously indicated, leak test may be performed by using radiometric or mass spectrometric methods.

PROBLEM:

Nondestructive Examination of Ag-Zn and Ag-Mg Type of Dry Charge Batteries for Degradation of Active Materials after Extended Storage

PANEL REPORTS:

1. Penetrants and Magnetic Particle

The problem to determine degradation of silver-zinc (Ag-Zn) and silver-magnesium (Ag-Mg) batteries was not considered feasible for detection by penetrant and magnetic methods. Further discussion indicated that the thermo-gravimetric techniques similar to that developed by the Navy may provide a useful inspection tool. Specific reference regarding this technique will be furnished the presentor. It was further stated that Dr. Harris of the Bureau of Standards may be able to provide assistance in the solution of this problem.

2. Ultrasonic

Ultrasonic inspection was determined not applicable to the solution of this problem. Here it was also suggested that measurement of electrical resistance or capacitance might be used to solve the problems.

3. Radiological

This problem was discussed at great length with the presentor. Due to the complex nature of the construction of the batteries and the orientation of the plates, and the requirement that the batteries be fully charged at all times, some doubt existed as to whether this could be handled by radiographic procedures. However, it was considered that, through research, radiographic procedures might be developed for this inspection. It was suggested that radiographs be taken of known batteries of a full charge at various planes and at various intervals to provide a technique for examining and correlating plate separation and resulting loss of charge. Through this technique it can be determined what has happened to the gap space between the plates. It would not be possible to determine whether the plates have deteriorated to the point that they will not function. The Naval Ordnance Laboratory has conducted considerable research and tests on a different type battery. Mr. Holloway, Naval Ordnance Laboratory, stated that if the presentor of the problem would send batteries to the Naval Ordnance Laboratory, this Laboratory would use these batteries for research and formulation of X-ray methods considered applicable to the solution of this problem.

4. Others

It is the considered opinion of the presentor that the primary deteriorating agent was atmospheric moisture which reacts with the Zn or Mg and produces oxides or hydroxides, decomposition products and useless to generating electromotive force. If the enclosure is gas tight, the amount of undesirable chemical reaction may be gauged by the amount of hydrogen generated. There are detection devices available to determine this. This approach would not work if

the chemical reaction results in the formation of a hydrate. The most promising solution is by preventing the ingress of atmospheric moisture by making the case gas tight or by sealing water openings with a breakable, impermeable membrane of Saran or Mylar, and using dessicant granules with dye indicator. Periodic surveillance of state of dessicant and renewal would assure minimum deterioration from atmospheric moisture. The Ag-Mg battery is damaged by exposure to high temperatures during storage. It was suggested that a maximum thermometer, mounted visibly inside the case, would record the maximum temperature to which the battery had been exposed. Based upon the date of manufacture and the periodic inspection for temperature, suspect batteries could be replaced. A more elaborate method would be a small, battery operated temperature recorder with a chart covering a period of one year to indicate level of thermal exposure.

PROBLEM:

Nondestructive Inspection of Bolt of 7.62 mm M14 Rifle

PANEL REPORTS:

1. Penetrants and Magnetic Particle

The problem as presented was concerned with finding the core hardness and the percent of ferrite in the bolt. It was concluded that penetrants were not applicable to this problem. The present method of using magnetic property analysis was considered the optimum method. However, improvements to this method were not known. Therefore, no suggestions for improvements could be made.

2. Ultrasonic

Variation in hardness or the ferrite content may be revealed by ultrasonic measurement; that is, through variation in attenuation. It was recommended, also, that components be examined for evidence of grain growth which could increase the brittleness of the material.

3. Radiological

Radiological methods were considered as not applicable to this problem. However, it was suggested that more rigid process quality control and qualification of the heat treatment might help in this problem.

4. Others

The extremely small incidence of failures, makes it probable that they were not due to failure to comply with specifications but to external, fortuitous causes such as discontinuities, inclusions or minute differences in radius of fillets at critical points such as reentrant angle on back lug. It was pointed out that at this point which is the apex of a 90° angle, the bolt would be mechanically operable with the radius at base of the angle 1/32" or less. However, a very small radius of the order of .001 or .002 inches (approaching a sharp 90° notch) would impose much greater local stresses at the base of the notch than radii of .020 or greater. A simple check on this can be made on an optical comparator. Another consideration is that the hardness of the case-hardened bolt is given in Rockwell C scale. This produces a severe indentation and, on a case-hardened material is a complex function of case and core hardness. A Rockwell C scale reading of 45 might be obtained on a thin but extremely hard case (C 60) with soft core (C 30) or on a deeper, softer case (C 48) with a relatively hard core (C 40). Although the overall reading of Rockwell C would be the same, the first example would be a less desirable bolt than the second. The use of the Rockwell A scale, superficial scale, or the Tukon was suggested to differentiate the two conditions cited above. Also, the possibility of the use of a more severe proof testing procedure was suggested; the theory being that borderline conditions would be eliminated in proof testing. Those passing had a reasonable expectancy of full service use.

PROBLEM:

Nondestructive Inspection of Thickness and Quality of Bond of Zirconium Oxide of XLR-99-RM-1 Rocket Engine Thrust Chamber

PANEL REPORTS:

1. Penetrants and Magnetic Particle

This problem was considered in several different parts. The most important part of this problem is the determination of Rokide thickness. Penetrants, of course, were considered not applicable and emphasis was placed on electro-magnetic techniques. It was felt that with the numerous commercial instruments available today using electro-magnetic techniques one might provide the capability for thickness measurements. It was requested that the presentor consult the National Bureau of Standard Handbook 77, Vol. 3 since conceivably one of the techniques outlined might be useful. While not applicable to current problems, it was suggested that different colors or dyes be placed in each coating layer during deposition. Through the use of such a technique the relating thickness could be determined during errosion or ablation.

The inner wall gas side thickness and tubing ID leaks are another phase of the problem. Penetrants are not suggested for this use and magnetic methods are not known which would be sufficiently accurate for problem solution.

The last phase of the problem is the quality of repair welds in tubes. For this specific problem fluid penetrants are suggested as an inspection tool. Magnetic methods are not considered to be applicable.

2. Ultrasonic

The surface contour, roughness and the high attenuation characteristics of Rokide were considered to be serious deterrents to the application of ultrasonic inspection, however, it is conceivable that special techniques could be developed utilizing focused transducers and additional advanced concepts. The use of the Dermatron, which is an eddy-current instrument, was suggested. Here a small transducer is utilized to measure the thickness of the dielectric (Rokide). The Armour Research Foundation, working under Contract No. AF 33(616)-6396, is studying ultrasonic methods for nondestructive evaluation of ceramic coatings. This research is directed primarily to the evaluation of strength and integrity of ceramic conditions under idealized conditions, however, it is possible that they may be able to offer technical guidance in solution of the XLR-99 problem.

3. Radiological

It was suggested that back scatter X-ray be explored as a possible technique for thickness determinations. Sample material will be forwarded to Dr. J. Bujes at NOTS, China Lake, California. Dr. Bujes will explore the feasibility of using this principle. Mr. Goldspiel of the New York Naval Shipyard has agreed to determine the feasibility of depositing nickel by

electrolysis on the surface to be inspected as a means of introducing an isotope tracer. This will permit the use of a counter to determine the wall thickness of the tubing.

4. Others

This problem was divided into the successive steps which occur in use as follows:

a. Erosion of original Rokide protective layer.

b. Excessive heat buildup inside of tubes.

c. Conversion of liquid ammonia into gaseous form.

d. Nitriding effect of gaseous ammonia on stainless steel of tube wall.

e. Failures of nitrided (embrittled) stainless steel at points where Rokide layer has eroded away.

f. Pinhole leaks merely feed ammonia into combustion chamber; larger leaks extinquish flame.

For the detection of thin spots in Rokide protective coating use Dermatron, ED 500 Magnaflux, or Fokker Sonic Attenuator for checking thickness of ceramic layer on combustion side of tubes. To locate weak spots in tubes, apply proof pressure of approximately fifteen percent of normal operating pressure, using hydraulic liquid. This should break tubes at incipient weak spots. It will be possible to remove Rokide layer heliarc weld, and recoat with Rokide. On new motors, incorporate radioactive coating on stainless steel tube surface under Rokide and use detector for measuring coating thickness. This will require careful calibration and check on loss of radioactivity due to operating temperature.

PROBLEM:

Nondestructive Inspection of Cracks in Ferrous Boiler Tubes

PANEL REPORTS:

1. Penetrants and Magnetic Particle

The discussion covered the testing of the ferrous boiler tubes during overhaul for corrosion, cracks, seams, bulges, and loss in ductility by testing the water side of the tubes. It was concluded that the use of penetrants could result in objectionable residual fluids which could conceivably interfere with boiler functions. The Norfolk Naval Shipyard made an unsuccessful attempt to apply both magnetic particle and magnetic analysis probe techniques to the inside diameter. Mr. B. Hoffman, Frankford Arsenal, suggested the use of a Magnaprobe. It was suggested that the presentor submit a sample to Mr. Hoffman for his analysis.

2. Ultrasonic

Mr. Hart, U. S. Naval Research Laboratory, said that he had done some work with ultrasonics on a similar problem. He used an angle beam probe on a flexible drive. This method was capable of detecting pits, cracks, and bulges. This process would require precleaning of the tubes prior to the testing which is an important aspect of this problem. Once the scale has been removed and the bare metal is exposed, you can use magnetic means to detect discontinuities on the surface. You can also use optical means such as fiber optics or prism optics. Both are quite complex but nevertheless possible for examining the sidewall of bent or complex shaped tubes.

3. Radiological

One of the problems related to this problem is inaccessibility of the boiler tubes. Very few of the tubes are accessible through the port. The extent of inspection is based upon what is found during the inspection of those tubes which are readily accessible. The present method is to wrap film around the tube and check. The penetrant method is used, also. It was suggested that a scanning device be investigated. It was stated that there is only a one-half inch space between the tubes. Dr. Bujes, U. S. Naval Ordnance Test Station, has a pick up unit which required only one-fourth of an inch clearance on the outside of the tube. Dr. Bujes offered his services and suggested that the presentor furnish both good tubing and tubing which is unacceptable. However, this still does not eliminate the problem of the inaccessible tubes. Dr. Bujes would advise the presentor if the scanning device were more economical and faster than the present method. However, if the decision were made to check further, it would be necessary to tear out the bulkhead in order to inspect the inaccessible tubes. It was suggested, further, that a recording device might be supplemented with a recorder system to reduce overall inspection time and costs.

4. Others

Naval steam plants are water tube boilers consisting of a water drum approximately 2 ft. in diameter and a steam drum approximately 4 ft. in diameter connected by a series of 200 or more water tubes with a diameter between 1 and 2 inches, joining the drums. These tubes have a bend of some 20° at each end. The problem is to determine when a water tube has reached a condition requiring replacement.

a. Forms of damage to water tubes are:

(1) Boiler scale (lime-magnesia deposits, ferrous corrosion products);

(2) Bulging of tube, from creep at high temperature and pressure; and

(3) Pitting of sidewall by corrosion

b. Suggested approaches were:

(1) Pass a dial indicator bore gage through tube, scale on inside of tube will cause the bore to be smaller than nominal. Bulges in tube will be detected by sudden increase in internal diameter.

(2) Clean out scale with regular tube cleaners.

(3) Examine inside tube wall with low magnification periscope or fiber optics; the head to carry its own source of illumination.

(4) Another tool for sidewall thickness at this stage would be a magnetic probe which would respond to sudden decrease of sidewall thickness, (corroded areas and pits).

NEW CONCEPTS IN THE NON-DESTRUCTIVE TESTING OF EXPLOSIVE SYSTEMS

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I. Introduction

A. Nature of Explosive Systems. Two very important characteristics associated with the functioning reliability of explosive systems are that they have a statistically distributed response to input stimuli and their reaction to stimuli are irreversible. The consequence of irreversibility is that a functioning test cannot be performed on a given individual system without altering or destroying it. The effect of the statistical nature of explosive systems is that the response of an individual system must be stated as a statistical probability based on the tested response of supposedly identical systems. Where reliability must be high, as in modern missiles and space vehicles, the cost of the destructive testing is exhorbitant but essential.

Another aggravating aspect in determining to what degree a system will respond to a given stimulus is that only two outward responses are available. Either the explosive fires from the stimulus or it fails. Quantal data are not obtained; the margin of success or failure of a single test is never known.

B. Testing an Explosive System. A number of go/no-go types of destructive tests have been used to assess explosive system functioning reliability. Among these have been the Bruceton test (1), the Probit test (2), and the run-down test (3). In addition, wherever possible, non-destructive tests have been employed to eliminate duds, maintain control of functioning reliability, and maintain the uniformity of output power. For example, such tests as bridgewire resistance and insulation resistance measurements, radiographic inspection, density and gravimetric determinations, and leak testing have been used.

C. New Tests. The purpose of this paper is not to discuss the tests that have been used in the past but to describe two techniques of recent vintage which hold much more than the usual promise to increase uniformity, to improve reliability, and also to reduce the cost of the destructive testing necessary. The two new techniques are (a) the characterization of electro-explosive devices through thermal parameters and (b) the application of penalty testing to assess the functioning reliability across explosive interfaces.

II. Electrothermal Parameters.

A. History. The initiation of a primary explosive is basically a thermal process (4) in which a sufficiently large volume of explosive (hot spot) must be heated to a temperature where self-sustaining reactions occur. Early tests on electro-explosive devices showed that if the bridge was heated adiabatically,

the energy required for firing was directly proportional to the heat capacity of the bridge.* This fact was first utilized to perfect a test called the "twinkle test" wherein the temperature rise of individual bridges (prior to explosive loading) resulting from a fixed energy pulse into the bridge was used to measure bridge uniformity (5,6). It was found that under non-adiabatic conditions more energy was needed (7). Later, the entire heating-cooling characteristic of the bridge-explosive system was postulated mathematically (8,9). It is this mathematical postulation which is the basis of the new test.

B. Explanation of the Technique. It has been found that the heating-cooling action of a wire bridge electro-explosive device can be closely represented by the differential equation:

$$C_p \frac{d\theta}{dt} + \gamma\theta = P(t) \qquad (1)$$

where: C_p = the heat capacity of the bridge/explosive system

γ = heat loss factor

$P(t)$ = power input

θ = temperature rise above ambient

t = time.

By using instruments developed specifically for the purpose, the constants C_p and γ can be evaluated for individual explosive devices and related to their uniformity.

The value for C_p, although it includes a thin layer of explosive about the bridge and the effects of wire attachments, is primarily a measure of the uniformity of the mass of the bridge. The temperature rise attained by a bridgewire when firing under adiabatic conditions is:

$$\theta = \frac{\text{Energy Input}}{C_p} .$$

Since it is the temperature rise (assuming constant initial temperature) which causes the explosion, each individual C_p value is an indicator of the firing characteristic or the sensitivity of the device with which it is associated. Furthermore, if C_p is measured for a group of devices, all made to the same specification, the uniformity (or non-uniformity) of C_p is a measure of the

*Proportionality was in fact established between the bridgewire mass (or volume for a fixed bridge material) and the energy which caused 50% of tested samples to fire.

uniformity of the firing characteristic of the lot. Those units with the lowest C_p values will give the greatest temperature elevations and will therefore be the most sensitive (most likely to fire from a given input stimulus) and conversely the highest C_p values are associated with the least sensitive devices. It would be possible to cull out, if desired, items having values of C_p too far removed from the lot average value.

γ is the heat loss factor which accounts for the losses from the wire by conduction and convection both into the wire supports and into the surrounding explosive layer (losses by radiation for usual firing temperatures are negligible). Under steady state temperature conditions the differential equation assumes the form

$$\gamma \theta = P(t). \tag{2}$$

γ can thus be evaluated by determining the power necessary to maintain the bridge at a fixed elevated temperature. The value of γ has been found to be heavily dependent on the wire surroundings such as the intimacy of contact between the wire and the explosive. It is thus a measure of whether or not the explosive is present in intimate contact with the bridgewire in individual devices and how uniformly the explosive has been loaded throughout a lot of supposedly identical items (a major cause of duds in electro-explosive devices is inadequate contact between bridge and explosive. Items within a lot can be sorted out if so desired, on the basis of the γ value.

It should be noted that both the measurement of C_p and γ are dependent upon the temperature elevation measurement. A fortunate and simple method of marking the temperature elevation measurement is be using the bridgewire as its own resistance thermometer. Conventional bridgewire materials such as nichrome, tungsten, and platinum-iridium alloys have a sufficiently large thermal coefficient of resistivity to allow this approach. The relationship between bridge resistance and temperature is given by:

$$R_\theta = R_0 (1 + \alpha\theta) \tag{3}$$

where: R_θ = resistance at temperature θ.

R_0 = resistance at ambient temperature.

α = thermal coefficient of resistivity of bridge material referenced to ambient temperature.

θ = temperature elevation above ambient.

C. Existing and Planned Instrumentation. In order to determine the values of C_p and γ for wire bridge electro-explosive devices, several pieces of apparatus have been developed. They are:

1. A self-balancing power bridge (8).

2. A third-harmonic generator (10).

3. A cooling-curve generator (11).

4. A constant-current generator (12).

The value of C_p may easily be obtained by deliverying a known quantity of energy into the bridge under adiabatic conditions and measuring the temperature rise. Of course, the quantity of energy must be less than that required to fire or alter in any fashion the firing characteristic of the electro-explosive device. If the energy is delivered adiabatically by capacitor discharge such as by the circuitry shown in Figure 1a,

$$C_p = \frac{\text{Energy stored in Capacitor}}{\theta} .$$

The basic physical concepts by which the determination of C_p is made are indicated in Figures 1b and 1c. Since the energy stored in the capacitor is $CV^2/2$ microjoules and

$$\theta = \frac{R_\theta - R_0}{R_0 \alpha} \quad * \tag{4}$$

then

$$C_p = \frac{CV^2 R_0 \alpha}{2(R_\theta - R_0)} \tag{5}$$

where: C is the capacitance in microfarads

V is the potential in volts.

C_p is therefore the system heat capacity and, in this case, is given in microjoules/degree temperature change. It is related directly to the average temperature elevation of the total bridgewire system. The technique used for measuring C_p is not restricted to input by capacitor discharge. Other energy burst sources may be used such as square-wave constant-current bursts or half sine-wave bursts.

The quantity α may be determined by measuring on a self-balancing bridge the power necessary to maintain a steady temperature (above ambient) at the

*The value of α can be determined experimentally. Let the resistance be measured at ambient temperature (indicated 0) and at some elevated temperature θ, then

$$\alpha = (R_\theta - R_0)/R_0\theta.$$

In lieu of experimentally determined values, a handbook constant for the bridgewire material could be used. Experience to date indicates that it is probably best to use individually determined values, unit-by-unit.

bridgewire. Figure 2 shows a layout of the instrumentation used. Under these conditions

$$\gamma = \frac{P(t)}{\theta} = \frac{P(t)\, R_0\, \alpha}{(R_\theta - R_0)} \tag{6}$$

If P(t) is determined in microwatts γ is given in microwatts/degree temperature change.

Instrumentation (10,11) has also been built which can check independently the ratio of C_p/γ. If the bridgewire is elevated in temperature and the power input discontinued

$$C_p \frac{d\theta}{dt} + \gamma\, \theta = 0 \tag{7}$$

and

$$\theta = \theta_0 \exp\left\{-\frac{\gamma}{C_p} t\right\} \tag{8}$$

where θ_0 is the temperature elevation when power was discontinued. The ratio C_p/γ can be measured experimentally by oscillographic recording of the resistance transient using a small trickle or monitoring current passed through the bridgewire during the cooling phase. This ratio is τ, the thermal time constant. It not only serves as a check for the measured values of C_p and γ, but sheds some additional light on how the variation of C_p and γ are occurring. For instance, it is expected that

C_p varies as $d^2 \ell$ (volume dependent)

γ varies as $d\,\ell$ (surface dependent)

τ varies as d. (from the ratio C_p/γ)

Here d and ℓ designate bridgewire diameter and length. Thus, if C_p and γ change due to changes in bridgewire length no change will be observed in τ but if changes in C_p and γ occur due to diameter variations τ will also vary.

D. Example. Electrothermal parameters have been measured for the Squib Mk 1 Mod 0 which is a typical wire bridge electro-explosive device. A cross section of this device is shown in Figure 3. The average values obtained for the thermal parameters are:

C_p = 2.4 microwatt seconds/°C

γ = 600 microwatts/°C

τ = 4.0 milliseconds.

To determine how well the electrothermal model fits actual squib firing using the parameters determined and assuming the hot-spot theory (4)* is correct, experimental firing results were compared with predictions of the Equation (1) solved for constant-current and capacitor-discharge inputs. The results are shown graphically in Figures 4 and 5.

III. VARICOMP Penalty Testing.

A. History. An explosive train consists of a number of distinct explosive charges arranged to give a chain of explosive actions leading from the response to the initial firing signal to the ultimate functioning of the system or weapon. Ordinarily, the train includes some method of inhibiting the completion of this chain of explosive action until required by the tactical situation. The inhibited arrangement is termed "SAFE" or "SAFED" and the readied condition is termed "ARMED". The proof of the validity of the explosive train design must include a demonstration of how reliable explosive action will propagate when the train is "ARMED", and a demonstration of assurance that explosive action will not propagate when the train is "SAFED".

A particular segment of the problem of demonstration of train safety/reliability is the demonstration of the probability of transfer of detonation across an interface between two high-explosive charges. The usual problems of go/no-go response apply. The problem is further complicated by the need for demonstration of very high reliabilities and safeties. Often requirements are encountered such as:

Reliability to be $>$ 99.99 percent at 95 percent confidence.

Probability of unsafe action to be $<$ 0.01 percent at 95 percent confidence.

Demonstration of such probabilities by direct, brute force testing would require exorbitantly large samples. (In either of the above requirements a minimum of 30,000 trials would be required.)

In order to reduce the testing effort that would be required for direct demonstration, some form of penalty testing is required; i.e., if a way can be found wherein the probability transfer of detonation can be determined under adverse conditions, then a higher probability can be expected under design conditions.

In the past, penalty testing has been achieved by geometrical modification of the interface between the two explosive charges by such devices as:

Interposition of inert barriers.

*For typical electro-explosive devices, the hot-spot temperature has been determined to be about 500°C.

Increase of air gap distance.

Misalignment of charges.

However, such procedures are apt to lead to faulty conclusions when these physical changes greatly modify the explosive mechanisms or phenomena by which detonation transfer takes place.

Another method of penalty testing is one which involves the alteration of the explosive composition. The desensitization of explosives by the addition of inert compounds is almost as old as the history of explosives. The use of diatomaceous earth to form dynamite is an example. Penalty testing of an explosive system can be accomplished by using desensitized explosives. For instance, if detonation is observed to propagate across an interface into an insensitive acceptor or target explosive, then it can be reasoned that transfer will occur more readily when the target explosive is more sensitive (more easily detonated). Therefore, it is logical to penalty test an explosive which is a known amount less sensitive than the design explosive. A demonstrated reliability under these penalized conditions can be used as a basis of quantitative extrapolation to a higher reliability under design conditions. The idea of VARICOMP (13) (VARIable explosive COMPosition) to bring about penalization of detonation transfer was originally advanced about 1953, but has developed slowly due to the complex statistical analysis necessary for assessing the results.

B. Explanation of Technique. VARICOMP can be considered as a pseudo-non destructive test of an explosive system in that the prediction of performance of the final design does not require the expenditure of samples of the final design. Rather, the prediction is based on data related to the final design by principles of analogy and obtained on simplified mock ups of those elements of the system directly representative of the explosive interface. It depends upon the measurement of the response of a penalized system with a relatively small sample size, and extrapolation to the performance of the design explosive knowing quantitatively the degree of penalization.

In the VARICOMP method of penalty testing, two considerations are important:

> A knowledge of the mechanism of explosives initiation. In the case of high explosives initiation is accomplished by means of a high intensity shock. If the shock intensity is strong enough, initiation will grow to detonation; if the shock intensity is relatively weak, the reaction will die out. This varies from explosive to explosive, and a given explosive can be diluted or sensitized to affect its sensitivity. Good calibrations (good measures of the response to stimulus distribution functions) of a number of explosives of widely differing sensitivities must be available for substitution in the explosive penalty test.

C. Instrumentation. The instrumentation of penalty testing consists of a test arrangement that gives adequate representation of the system to be tested

(only partial representation is necessary); means for detecting whether or not each test had produced a go or a no-go; a test plan (staircase up-and-down or run-down type of statistical design); and explosives properly calibrated, for use in the test.

The explosives, of varying sensitivities, can be either standard explosives or can be specially compounded for the particular needs, as in Figure 6. It is currently believed that the tailor-made (specially compounded) explosives should provide the basis for more precise determinations than might be the case with standard explosives.

A number of sensitivity tests exist which might be used to calibrate the VARICOMP explosives:

Small Scale Gap Test, Figure 7 (14).

NOL Booster Sensitivity Test (15).

NOL Propellant Sensitivity Test (16).

Drop Hammer Impact Test (17).

The choice of test is governed by the degree of similarity between the mechanisms of initiation characteristic of the test compared with the mechanisms characteristic of the transfer across the interface.

An inexpensive but appropriate mock up of that part of interest must be designed and available in quantity for the tests. (The mock ups must be inexpensive since each one tested will be destroyed in the test.)

E. Example. The analysis of detonation transfer reliability across interfaces in a typical warhead configuration will be given as an example of the VARICOMP procedure. The main warhead charge is H-6, cast in a 1/16-inch cold-rolled steel case. As shown in Figure 8, the charge configuration is roughly that of a flat cylinder with a concentric well to accept a cylindrical booster. The booster is separated from the main charge case by a 1/8-inch-wall central tube which is also closed at the bottom end. A 1/16-inch radial air gap exists between the booster and the central tube. Another 1/16-inch radial air gap exists between the central tube and the main-charge case.

H-6 and TNT were calibrated in a variable steel-barrier gap test. It was assumed that the response of the acceptor explosive could be described as being normally (Gaussian) distributed with decreasing gap. (On the basis of experience this assumption can be shown to be highly conservative.) The results of the calibration tests are shown as the two slant lines in Figure 9, and have each been drawn in a consistently conservative manner. The effect of this conservatism is to cause the lines to converge more closely (in the zone of response to a gap which most closely resembles the design gap) than would be indicated by the observed responses.

Ten weapon mock ups of the type shown in Figure 10, were loaded with TNT, and were tested with the standard booster charge. All ten trials were interpreted as having exhibited full initiation of the TNT, this being based on the amount of deformation of the witness block. At 97.5-percent confidence 10/10 successful trials indicates a minimum reliability of 69.2 percent; i.e., there is but one chance in forty that the observed 10 successes out of 10 trials with TNT could have been observed on a sample drawn from a population whose reliability is less than 69.2 percent.

From these results it can be deduced that the explosive drive in the mock up must be at least as much as the shock strength in the calibration tests indicated as the vertical line drawn through point A of Figure 11. Point A is the intersection of:

> A horizontal line drawn at a coordinate value of 69.2 percent which represents the lower limit estimate of reliability. The slant line labelled TNT which is the conservative estimate of the response vs. shock function for TNT. (By conservative in this case is meant the most sensitive that it might be expected to be at better than 95-percent confidence.)

The vertical line drawn upward through point A intersects the slant line representing the H-6 calibration at point B. (The H-6 line is a conservative estimate for the response vs. shock function by being drawn to indicate the most insensitive that it might be expected to be at better than 95-percent confidence.) The reliability coordinate of point B is 99.95 percent. This is a lower limit estimate of the reliability of detonation transfer across the booster-warhead interface with at least the following chain of independent elements of conservatism.

1. H-6 Calibration -- better than 95-percent confidence.
2. TNT Calibration -- better than 95-percent confidence.
3. Mock Up Test -- 97.5-percent confidence.
4. Mock Up Test Configuration (judged to be more difficult to initiate than the warhead system) -- ? percent confidence.
5. Assumption of calibration distribution function which suppressed the sensitivity distinction between H-6 and TNT -- ? percent confidence.

The VARICOMP estimate of reliability, on the basis of 10 samples, is 99.95 percent at some unknown,* but high, confidence provided basic assumptions of analogy are valid. A direct demonstration of this reliability at 95-percent confidence would require about 6,000 trials. This is an amazing saving in effort

*Unknown because statistical techniques are not yet at hand to determine the confidence associated with a combination of a chain of conservative estimates.

and, in fact at first sight, this appears to be something for nothing. Actually the VARICOMP process is a systematic method for combining quantitative data, experience, and engineering judgement in a methodical fashion and for this reason is not a blue-sky guess of the system performance.

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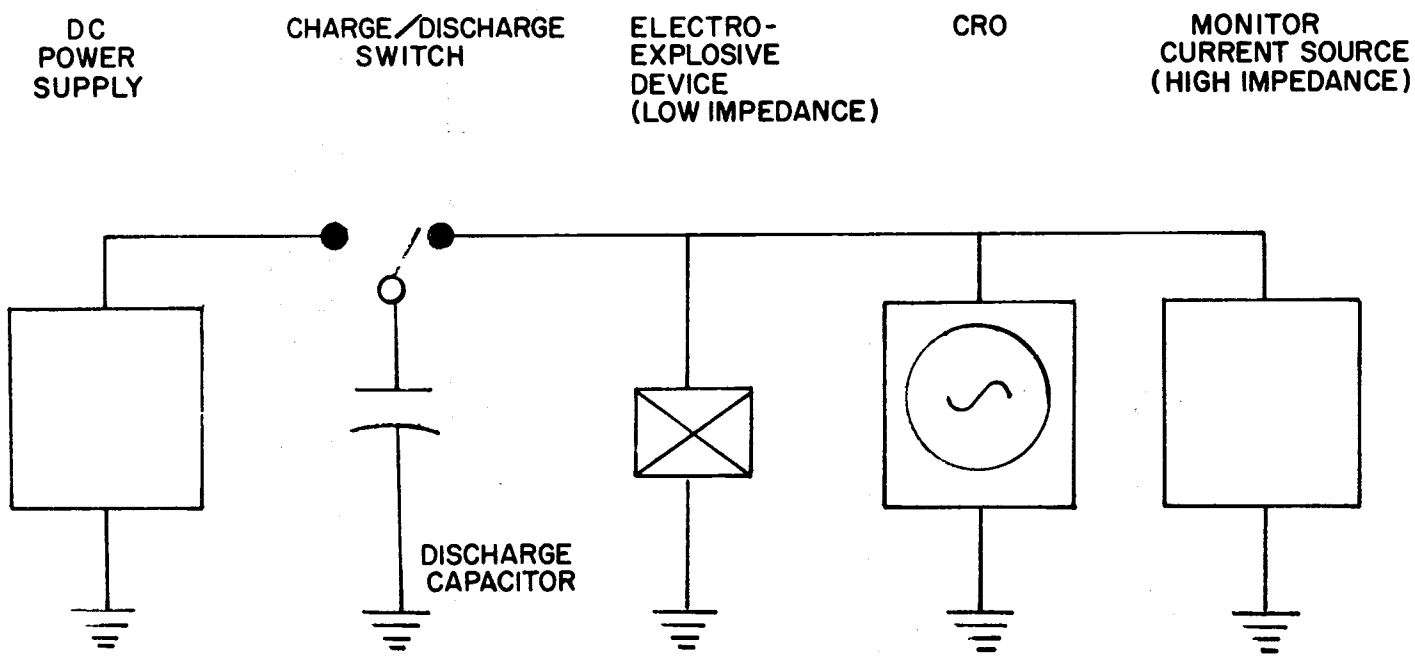


FIG. Ia CAPACITOR DISCHARGE, DETERMINATION OF Cp; INSTRUMENTATION

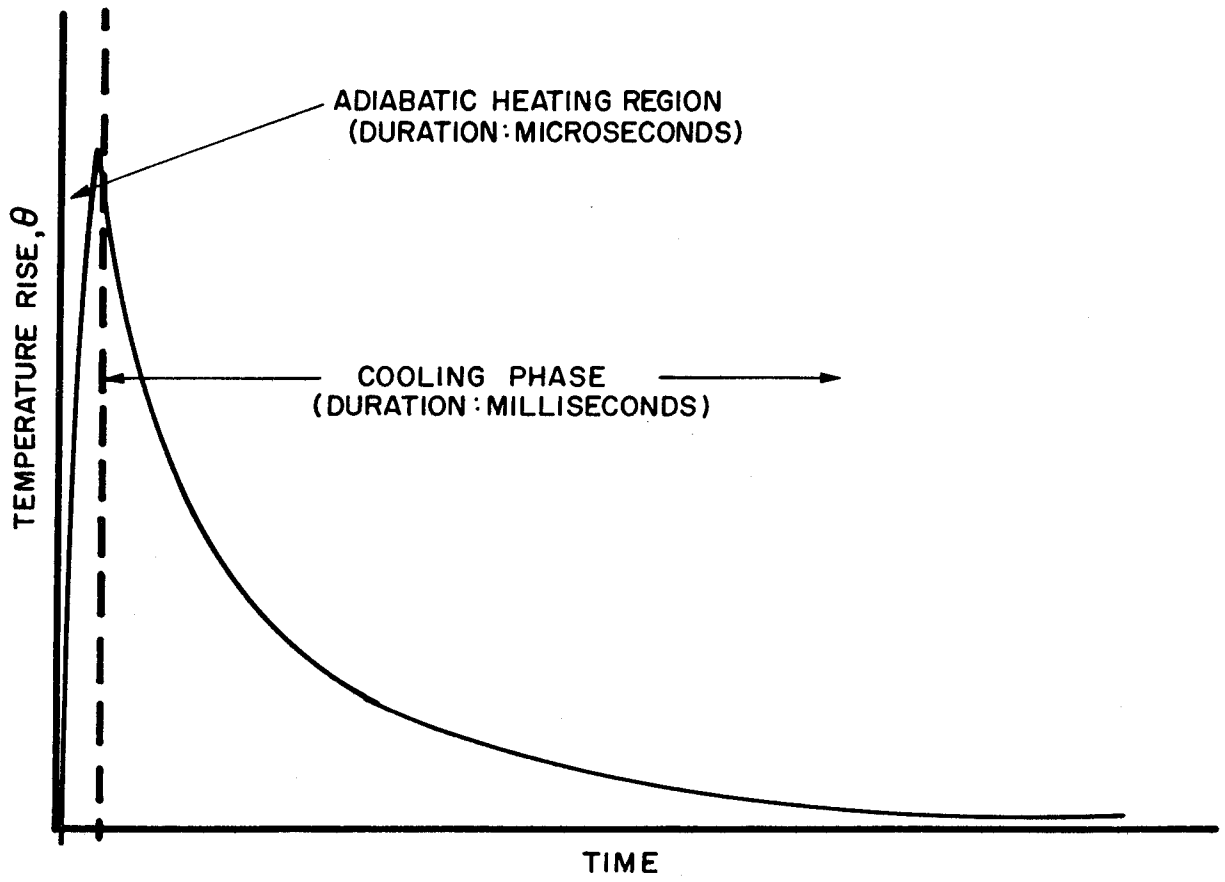


FIG. 1b CAPACITOR DISCHARGE DETERMINATION OF Cp;
TEMPERATURE – TIME TRANSIENT

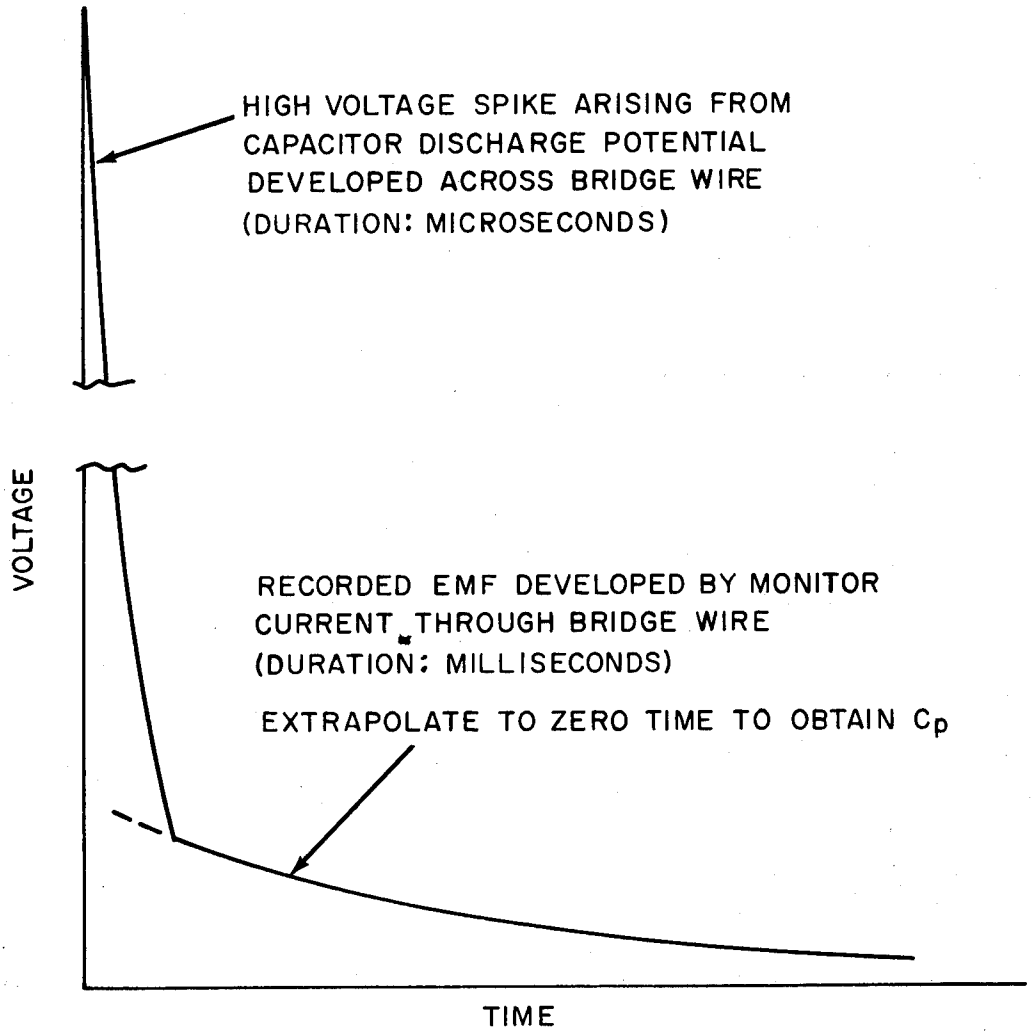
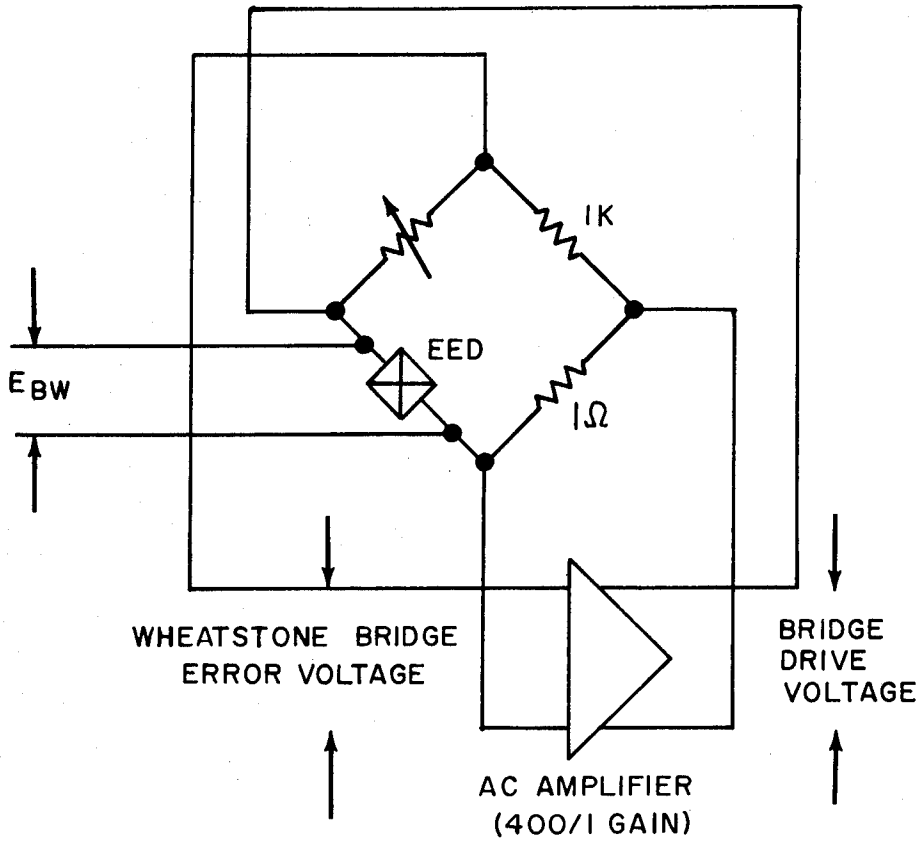


FIG. Ic CAPACITOR DISCHARGE DETERMINATION OF C_p: VOLTAGE TRANSIENT OBSERVED ON CRO

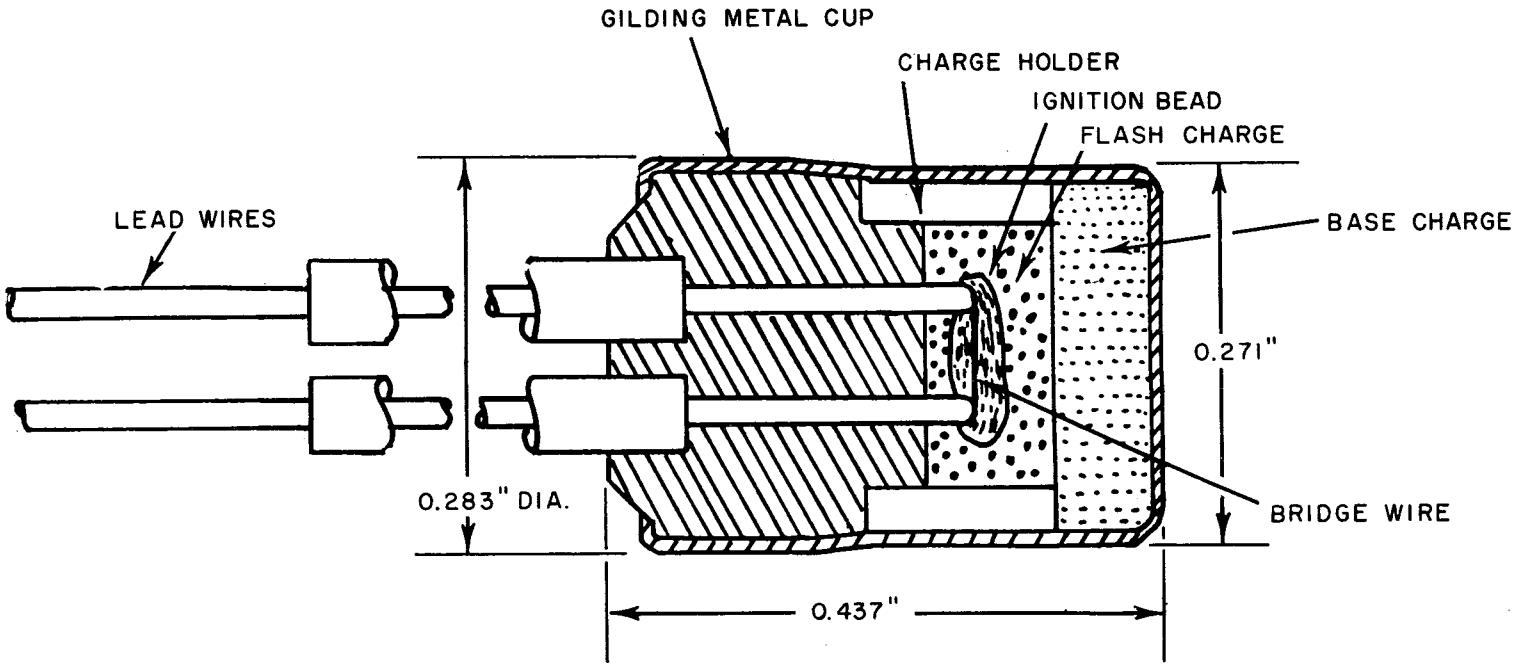


$$\gamma = \frac{\alpha E_{BW}^2 R_0}{R_\theta (R_\theta - R_0)}$$

R_0 AND α, BRIDGE WIRE PARAMETERS PREVIOUSLY MEASURED

R_θ DETERMINED FROM WHEATSTONE BRIDGE RATIO

FIG. 2 SELF-BALANCING BRIDGE DETERMINATION OF γ



NOTES:

1. IGNITION BEAD - APPROX. 5 MG DDNP/$KClO_3$
2. FLASH CHARGE - APPROX. 45 MG BLACK POWDER
3. BASE CHARGE - APPROX. 45 MG BLACK POWDER
4. BRIDGE WIRE - 0.001" PLATINUM-IRIDIUM 0.060" LONG

FIG. 3 SQUIB MK I MOD O

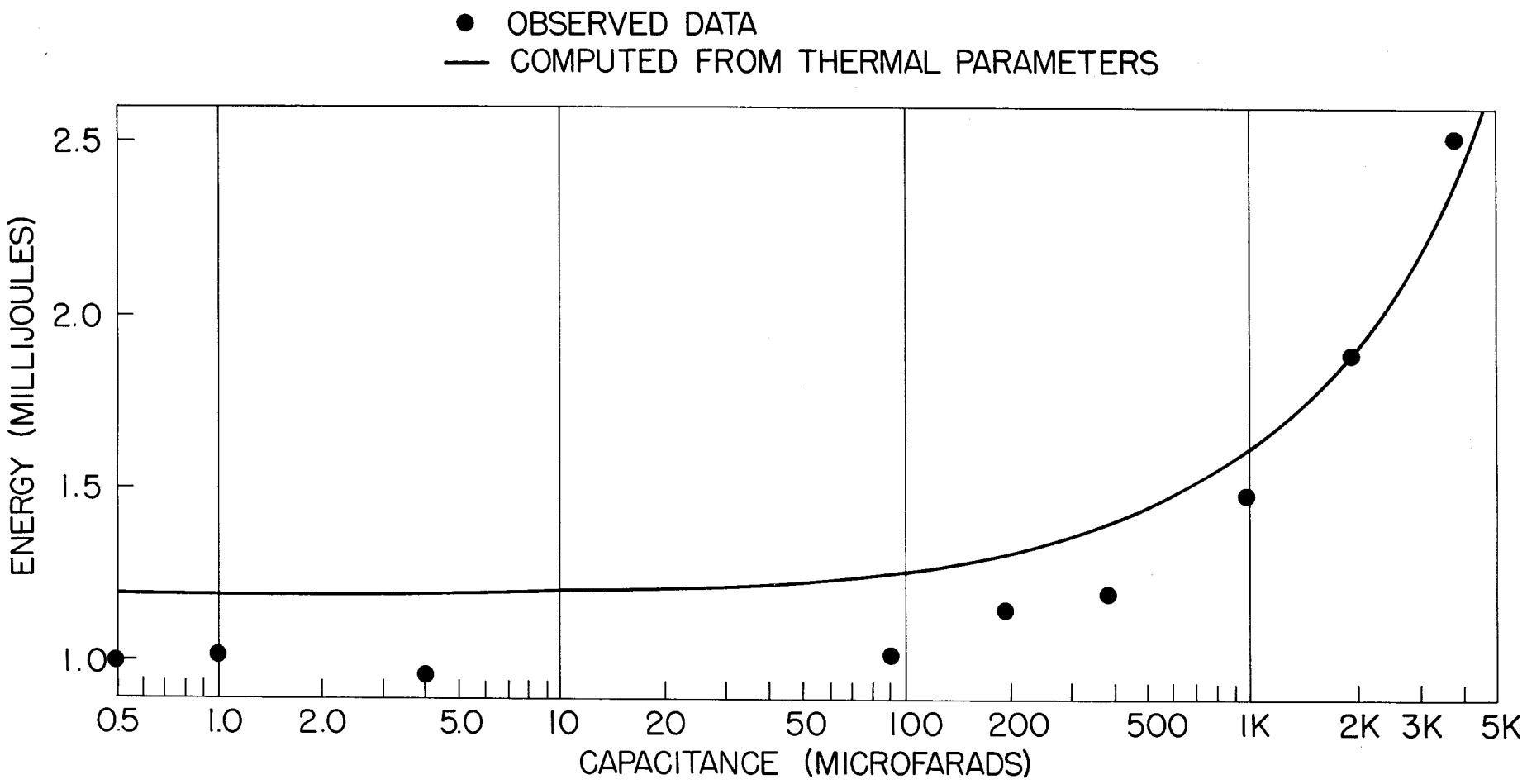
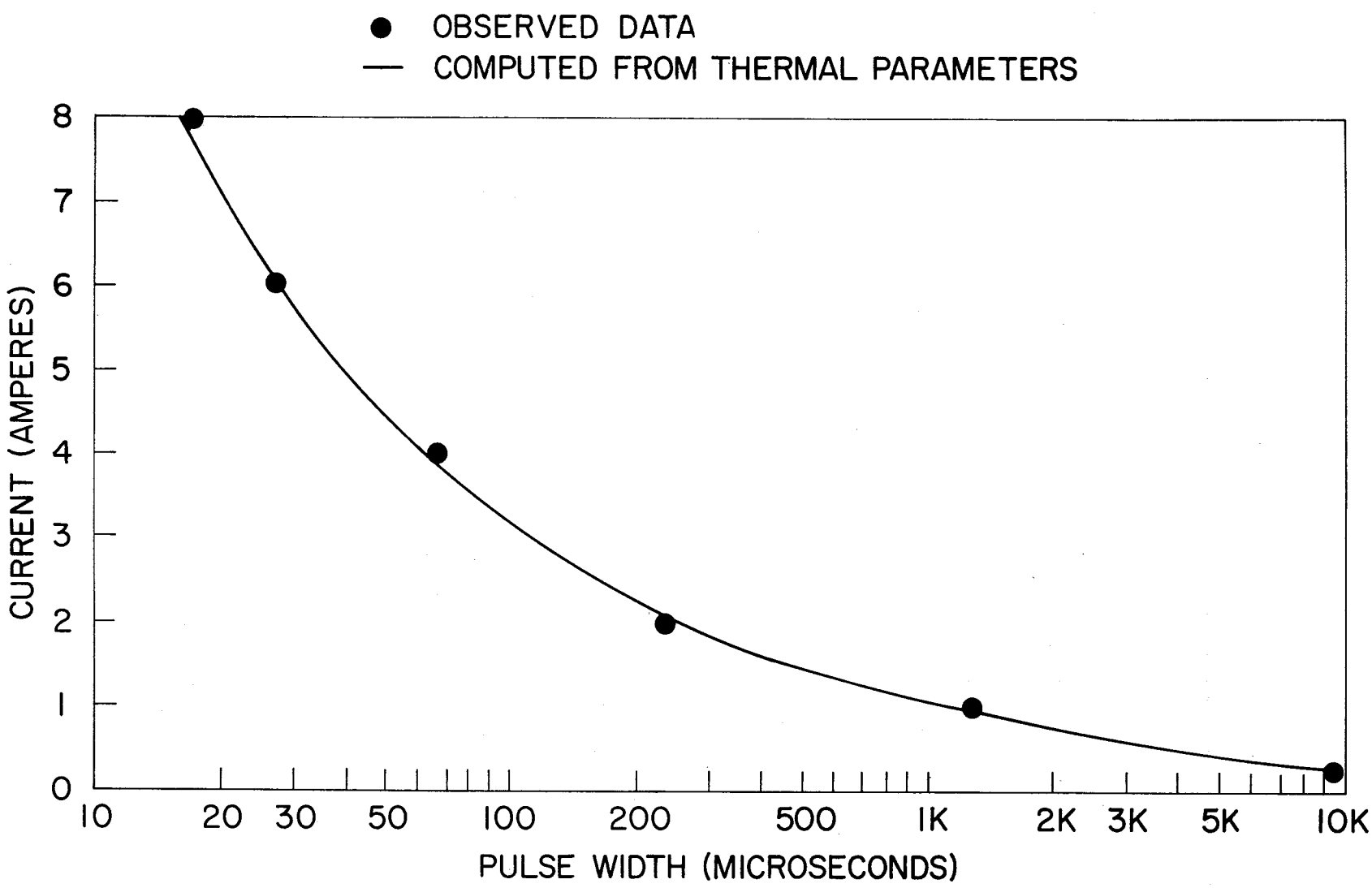


FIG. 4 CAPACITOR DISCHARGE FIRING
COMPARISON OF 50 PERCENT FIRING ENERGY WITH ENERGY NEEDED TO RAISE BRIDGE-WIRE TEMPERATURE 500° C



FIC. 5 CONSTANT CURRENT FIRING
COMPARISON OF 50 PERCENT FIRING CURRENT WITH CURRENT NEEDED TO RAISE BRIDGE-WIRE TEMPERATURE 500° C

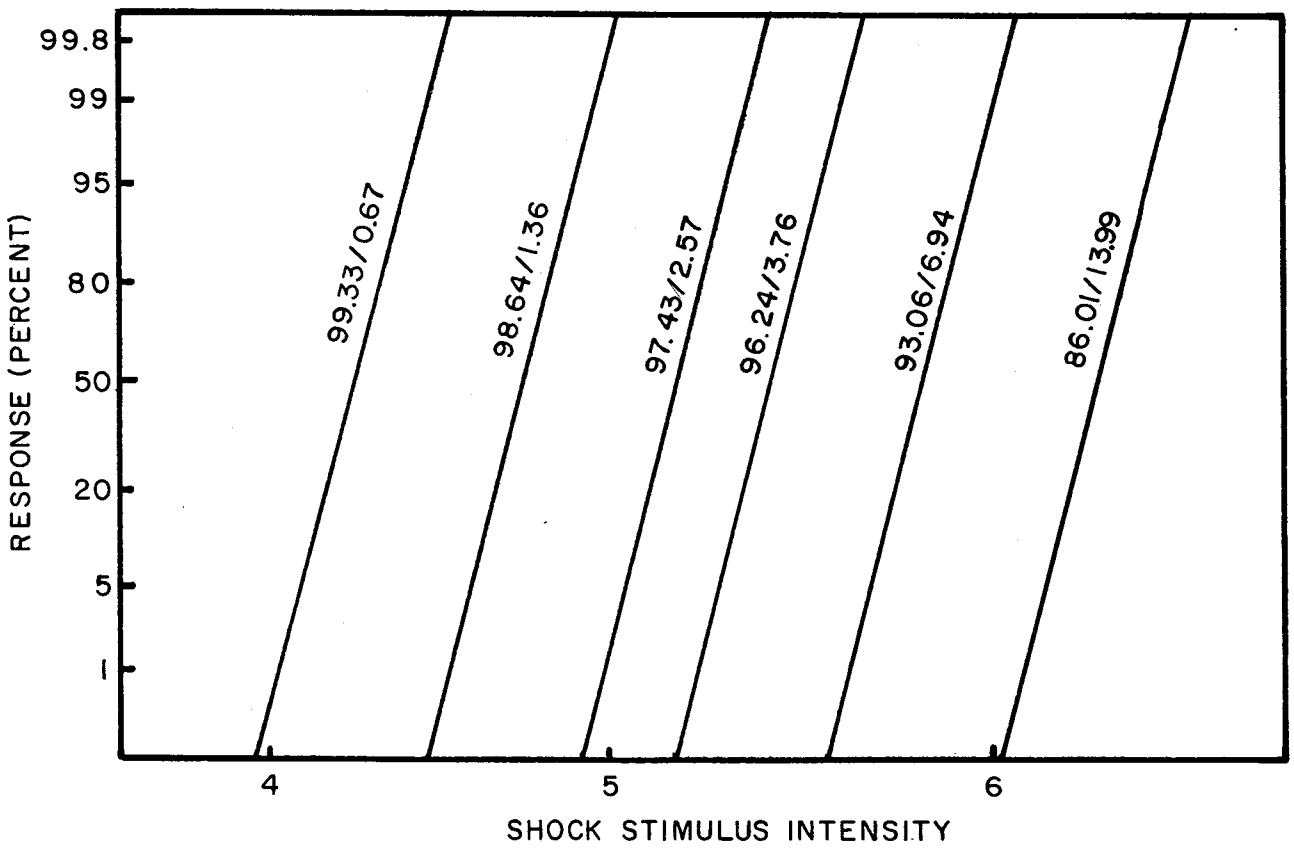


FIG. 6 SENSITIVITIES OF VARIOUS RDX/CALCIUM STEARATE MIXTURES

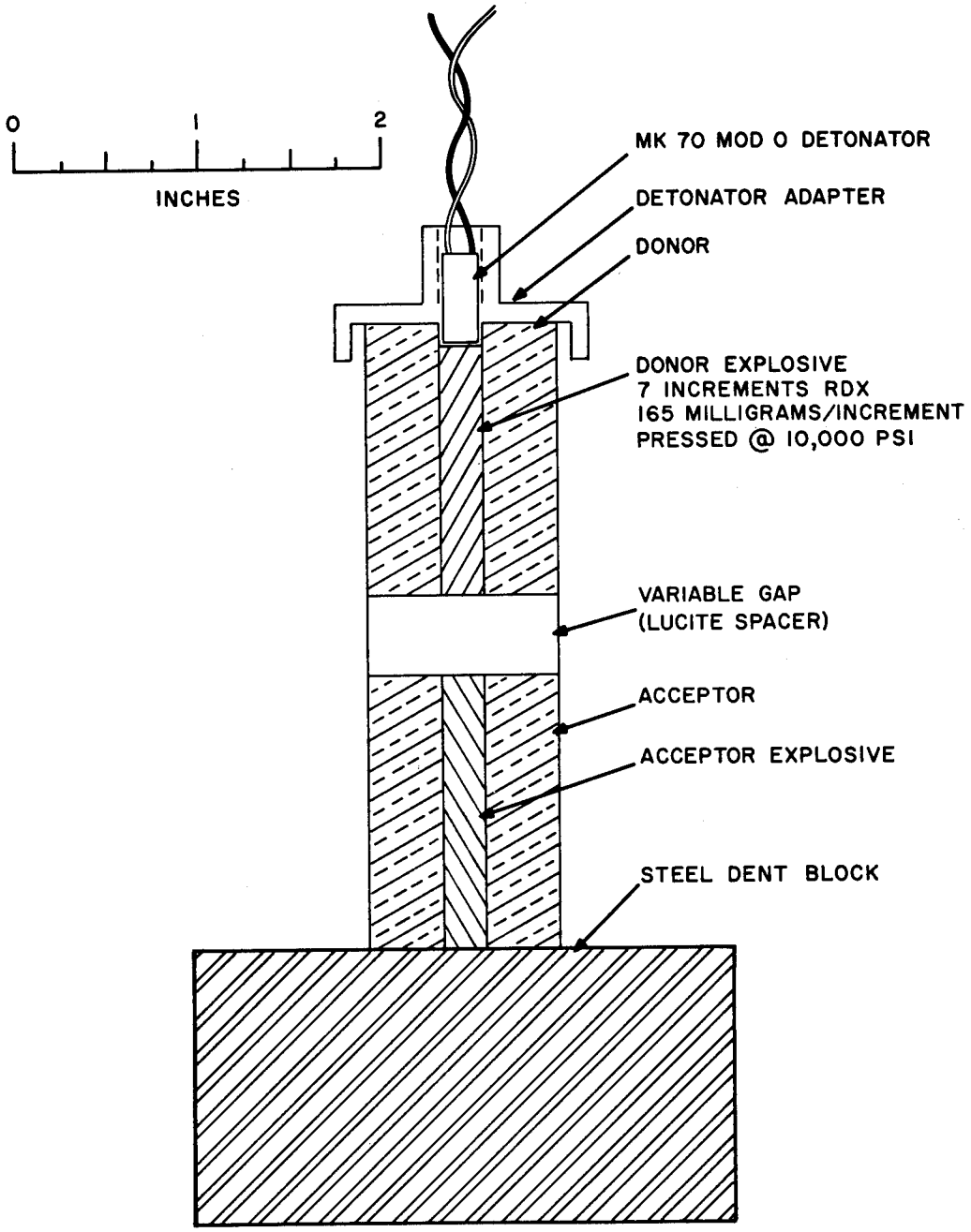


FIG. 7 SMALL SCALE GAP TEST

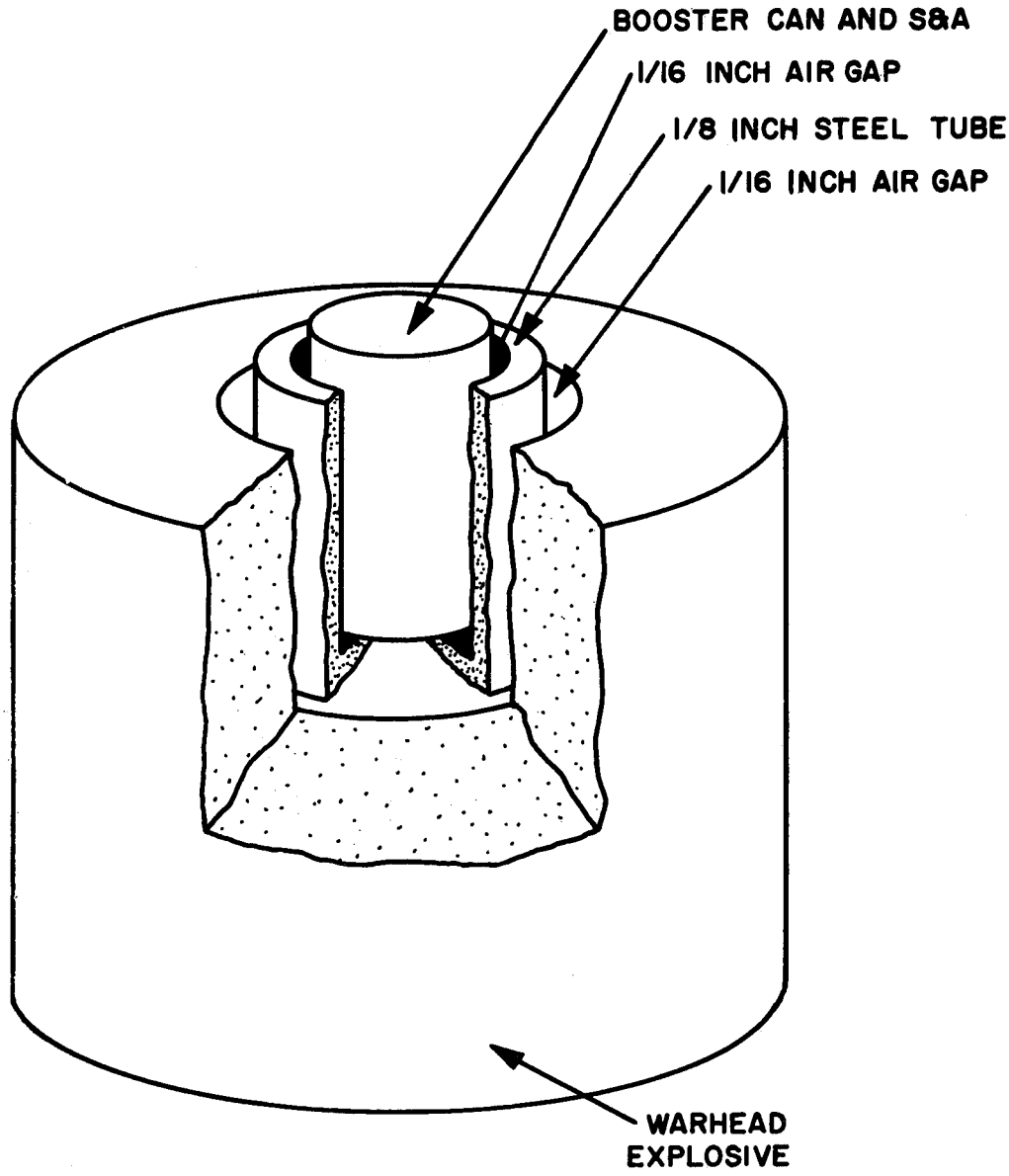


FIG.8 BOOSTER/WARHEAD CONFIGURATION

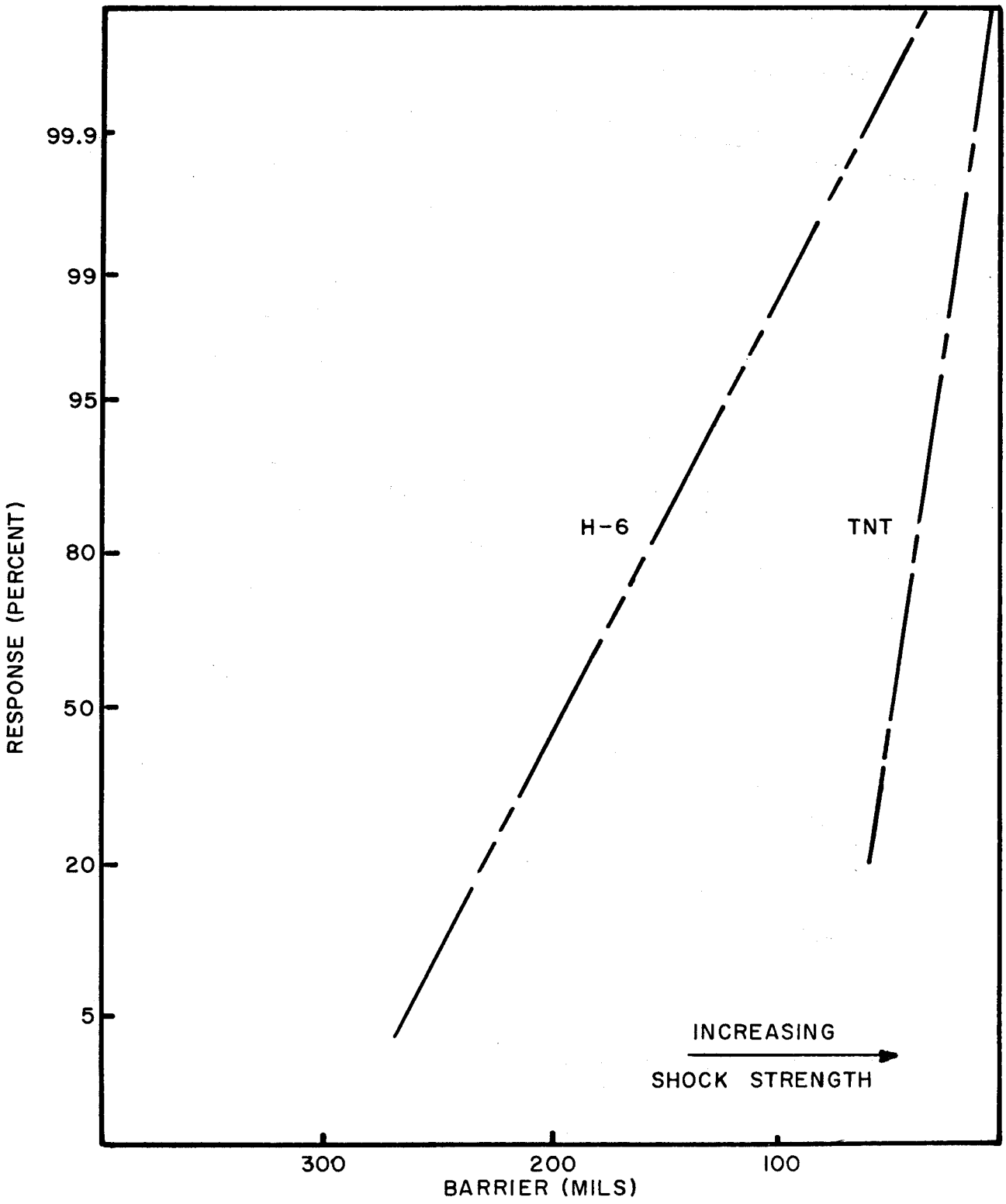


FIG. 9 CALIBRATION OF H-6 AND TNT

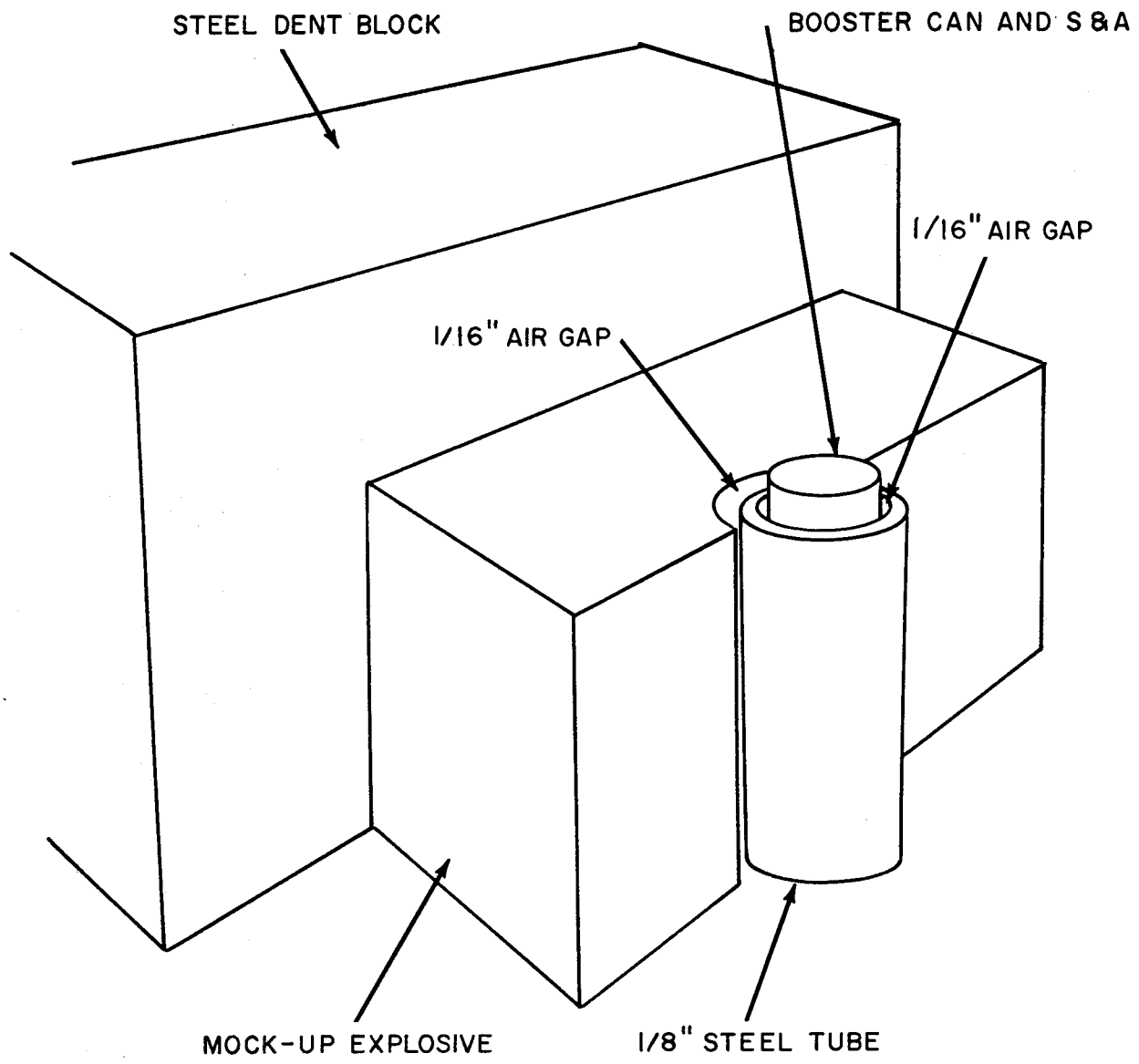


FIG. 10 BOOSTER/WARHEAD MOCK-UP

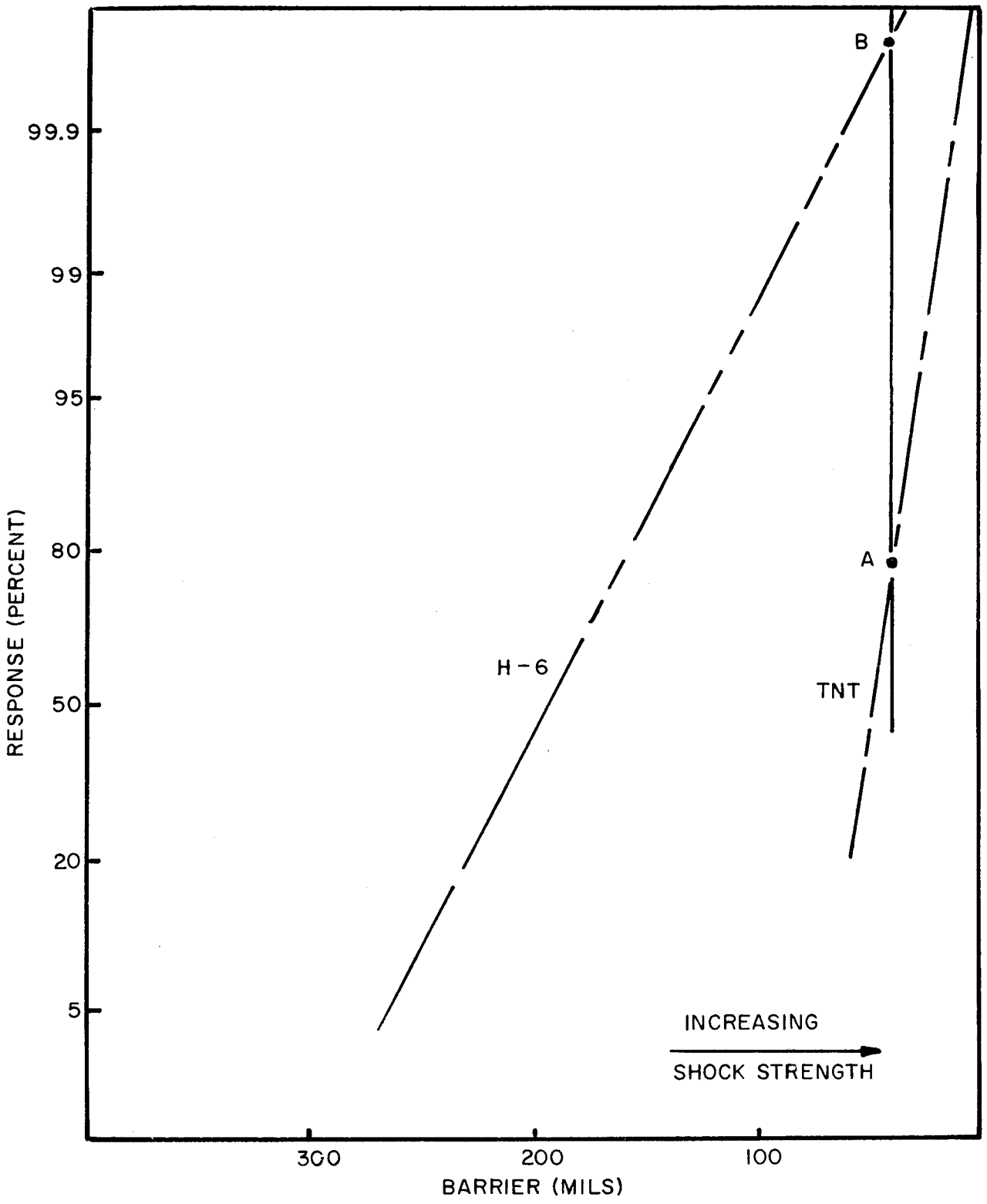


FIG. 11 BOOSTER/WARHEAD RELIABILITY ESTIMATE

INTERNAL INSPECTION OF LARGE PROPULSION UNITS BY ION CHAMBERS AND SCINTILLATOR DETECTORS

by

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INTRODUCTION

The difficulty of internal inspection of propulsion units by radiography grew with the design of ever larger solid propellents. Even 2-million volt X-ray machines become inadequate to penetrate the total thickness of a modern propellant. This difficulty was circumbented by inserting the X-ray film cassette in the perforation of the propellant so that the X-rays penetrated only one half of the total propellent thickness.

Fig. 1 illustrates the position of the film cassette inside of a POLARIS.

This method, however, fails if the perforation does not provide enough space for inserting the film cassette or when the propulsion motor is assembled. The latter condition prevails when the motor is reinspected internally just before firing on one of the launching sites such as Atlantic Missile Range. The same condition exists also when the motor undergoes its final inspection before delivery for tactical use.

On the other hand, 100-percent radiography by 22-MEV betatron X-rays of the straight part of the first-stage POLARIS, not including radiography of the interface separations and end closures, requires a minimum of about 90 manhours and about 400 X-ray films, 14 x 17 inch. Of course, those figures are much higher for the first-stage MINUTEMAN, whose straight part is twice as long as that of POLARIS, and also the outside diameter is considerably larger. Under these conditions, it was only natural for the Naval Ordnance Test Station to resort to a faster monitoring detection system and apply radiography only where the detectors would indicate a defect. At this point, I would like to emphasize that there is no substitute of radiography if an internal pictorial view of a test object is required.

However, scanning detectors such as ion chambers and scintillators were to be introduced for reduction of radiographic inspection time and cost.

DESCRIPTION OF DETECTORS

Ion Chambers

The performance of the ion chamber is based on ionization of a gas when affected by any radiation, such as alpha particles, beta rays, gamma rays or X-rays. The ion chamber itself is, in general, an airtight container with two electrodes of opposite polarities. During ionization, the ions of different polarities converge on the electrodes of opposite signs and the so obtained electric output can be measured or fed into a strip recorder. The output is a function of the radiation and efficiency of the chamber.

Scintillators

A crystal, e.g., NaI, shows the property of luminescence when exposed to invisible radiation, such as alpha particles, beta rays, gamma rays and X-rays. The light flashes or scintillators affect the cathode of a photo-multiplies and in cascading over a series of electrodes, so-called dynodes, multiply the cathode input about 10^6 time at the anode output.

Performance equation

Before discussing the equation, some of its parameters will be analyzed.

The detectors are electrically connected in a balance circuit according to the diagram in Fig. 2.

It is evident that the balance is changed when one of the detectors is irradiated differently than the other one. A differential output will thus result and can be transmitted to a recording strip. Since all detectors have an inherent "noise" resulting from irradiation, the signal to noise ratio determines the quality of the detector.

Another parameter is the minimum defect to be detected. Since radiography detects a one-inch deep radial crack and 0.001-inch wide, the same is expected from the scanning system. However, with the present equipment, only a 0.002-inch wide crack can be detected in reasonable time.

Fig. 3 represents the performance equation on the basis of 95% confidence level.

Performance Equation:

$$\frac{S}{\sigma_i} = \frac{\sqrt{E} \cdot \sqrt{\eta} \cdot A_c \cdot \sqrt{RC} \cdot (A) \cdot (B)}{2\sqrt{A_p}}$$

The parameters follow:

S = signal height

σ' = noise rate

Φ = Flux (photons per roentgen and cm^2).

η = Efficiency of the detector

A_c = Area of the defect in cm^2 perpendicular to the radiation beam.
A_p = Collimator area at the detector in cm^2.
RC = Time constant (resistance in ohms, capacity in farads).

$A = 1 - e^{-\mu x}$ = Radiation absorbed in the defect.

e = base of natural logarithm.

μ = X-ray absorption coefficient in cm^2 of the penetrated material for nonenergetic X-rays equivalent to peak photon energy.

$x = \frac{t}{RC}$ = the radial depth of the defect.

$B = 1 - e$ $\quad t = \frac{l}{v}$, l = detector collimator aperture in the scanning direction.

v = scanning speed per second.

Analysis of the performance equation

It is evident that the signal/noise ratio is the higher, i.e., more significant, the higher photon flux; efficiency; the larger the crack area; the larger RC; the smaller A_p.

The following analysis is based on 22-MEV betatron radiation, as the scanning detection systems were originally designed for the POLARIS and MINUTEMAN motors which required a high energy radiation source.

The factor Φ is a function of monoenergetic X-rays equivalent to the continuous energy spectrum of the 22-MEV betatron radiation. It was found by experiments that for a practical "narrow beam" the equivalent photon energy is 8 MEV and the absorption factors can be computed from the table in Fig. 4 as published in Handbook 55. National Bureau of Standards.

Experiments conducted with both detector types, ion chambers and scintillators, on first-stage POLARIS and MINUTEMAN revealed that, at present, the

scintillators are superior to ion chambers. The results were obtained by determination of the efficiencies from the performance equation for identical signal/noise ratio. The efficiencies ratio was 8:1 for scintillators and ion chambers, respectively.

The data on MINUTEMAN scanning with ion chambers were obtained from Boeing document no. D2-9958. The data for the scintillator was obtained from experiments with scintillators on first-stage POLARIS by the Naval Ordnance Test Station.

The scanning speed for the ion chambers and scintillators were 0.34 inch/sec and 0.68 inch/sec, respectively. The minimum defects of the same radial depths detected by ion chambers and scintillators were 0.06-inch and 0.071-inch width, respectively.

Figs. 5 and 6 illustrate the recording graphs obtained with scintillators on first-stage POLARIS and two different scanning speeds. In both cases, the defects were simulated by propellant slivers of the indicated size. Under consideration of the two different detector efficiencies, it was computed that for the inspection of the total straight part of the first-stage POLARIS, it would be required 8.5 hours with ion chambers, and only 1.4 hours with scintillators.

The physical size of both detectors is illustrated by Fig. 7.

The weight of both detectors is about 70 lbs for the scintillator, 700 lbs for the ion chambers.

Missile perforation

Because of the propellant perforation pattern of the POLARIS and MINUTEMAN, the radiation beam path varies considerably depending on the central beam angulation. The minimum thickness in the path of the X-ray beam, called "web", is so much smaller than the maximum thickness, called "star finger", that the detection time of the same defect e.g., in the first-stage POLARIS, 2.5x faster in the "web" than in the "star finger".

Scanning procedure

The most attractive method appears continuous scanning on a helical curve. But as difficulties arise, when the peripheral and longitudinal speeds are not precisely synchronized.

Therefore, it was decided to scan longitudinally, without rotation, and index at each stop for the desired angulation. By this method, it is also possible to apply different scanning speeds to the web and the rest of the propellant areas.

Because the firing performance of the propellant grain is more affected by defects in the web area than in any other part of the propellant, it is necessary

to detect the finest radial cracks in the web area. On the basis of this assumption and tests, as described in this presentation, it was calculated that 3 lengthwise scannings of the first-stage POLARIS, 60° apart will require 3.4 and 7.6 hours to detect 0.003-inch and 0.002-inch wide cracks respectively.

Fig. 1 illustrates a typical cross-section of a large missile solid propellant. The 2 detectors are located in adjacent circumferential sections and cover, e.g., the total width of the web. Both detectors can be affected by radial cracks in 2 different parts of the web. It is evident that the scanning for the web has to be performed only in 3 positions, 60° apart, because the X-ray beam penetrates in one position two diametrically located webs.

On the basis of the before illustrated performance equation, it was computed that, in three webs, the detection of 1-inch radial cracks 0.003-inch and 0.002-inch wide will require 3.4 and 7.6 hours respectively. The operation cost amounts to $200 including 10-percent radiographic retake. The rest of the propellant sections can be longitudinally scanned in 500 seconds, considering the minimum crack 1/8-inch wide.

Cost of 100-percent radiography of the straight body of POLARIS will require 29 hours at the cost of $1200 including films and assessment.

Handling of the POLARIS missile for internal inspection

In the following moving picture are illustrated all significant phases of handling the POLARIS missile and the betatron X-ray machine.

Cadmium selenide crystal as X-ray detectors

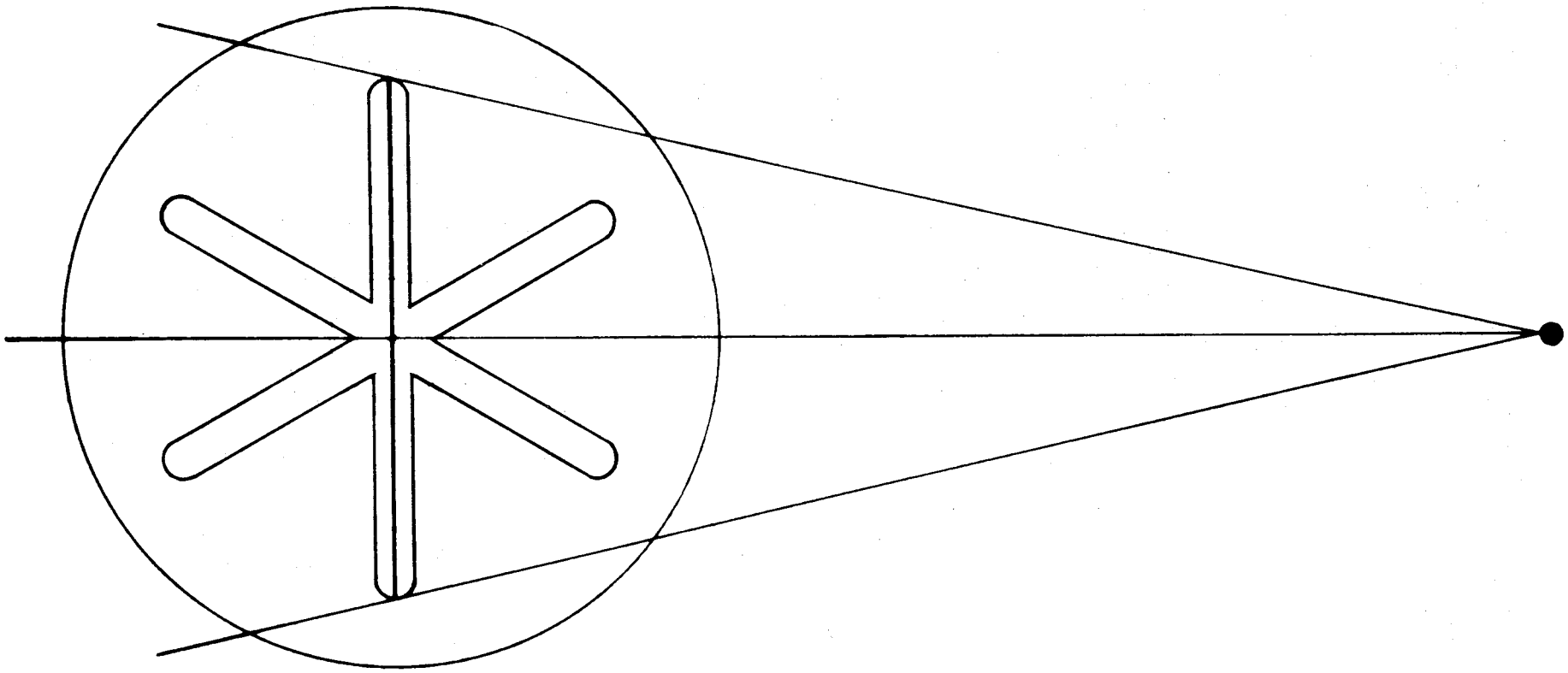
In conclusion, I would like to report preliminarily on a detector which might prove feasible.

The size and the shape of the crystal is demonstrated by Fig. 8.

The sensitive area is about 0.6 x 1 millimeters, therefore the width is about equal to the presently detectable minimum defect width, as I mentioned before.

Fig. 9 illustrates the detection results and indicates the parameters of the experiment. It is noteworthy that for betatron radiation, the ion chamber is 8-inches long and 1-1/4-inches in diameter, the sodium iodide crystal of the scintillator is 1-1/2 inches long and 1-1/2 inches in diameter in comparison with 0.024 x 0.04 inches of a cadmium selenide crystal. The detection of X-rays by the crystal which is a semiconductor is effected by reduction of its resistance, which is analog to the amplification of a transistor.

Our investigation will continue, especially as to the feasibility of combining a multitude of crystals to a mosaic pattern and increase thus the efficiency of such a unit.



X-RAY FILM CASSETTE
IN PROPELLANT PERFORATION

Fig #1

B,C = DETECTORS

E,F = BATTERIES

D = RECORDER

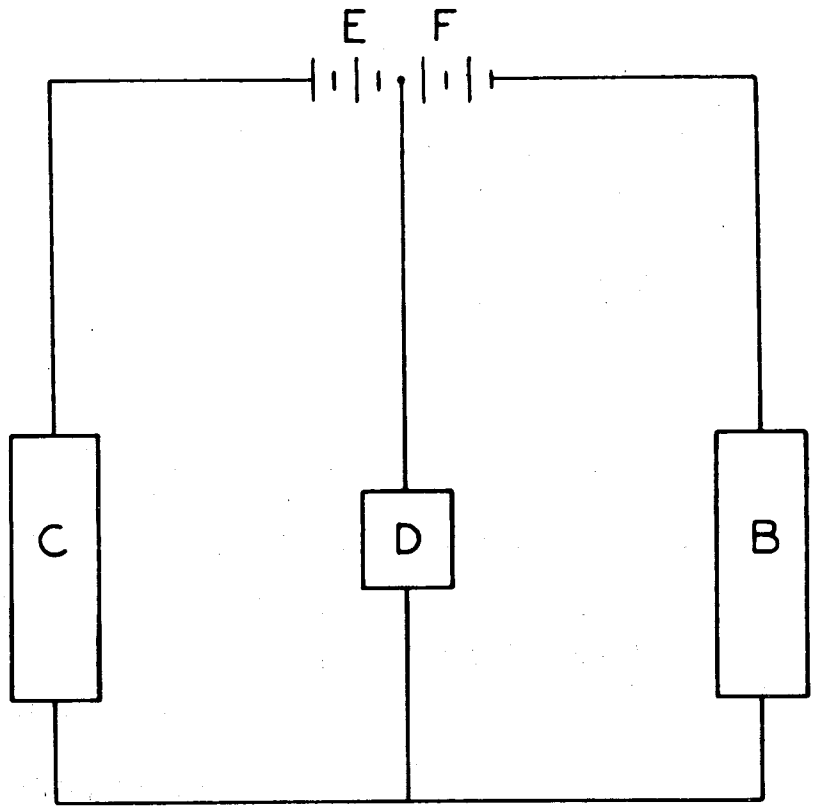


Fig. 2

DIAGRAMMATIC EL. CIRCUIT
FOR DIFFERENTIAL DETECTORS

PERFORMANCE EQUATION

$$\frac{S}{\sigma^1} = \frac{\sqrt{\Phi} \cdot \sqrt{\eta} \cdot A_c \cdot \sqrt{RC} \cdot (A)(B)}{2\sqrt{A_p}}$$

S = SIGNAL HEIGHT

σ^1 = NOISE RATE

Φ = FLUX (PHOTONS PER ROENTGEN AND cm^2)

η = DETECTOR EFFICIENCY

A_c = AREA OF THE DEFECT IN cm^2 PERPENDICULAR TO THE RADIATION BEAM

A_p = COLLIMATOR AREA AT THE DETECTOR IN cm^2

RC = TIME CONSTANT (RESISTANCE IN OHMS x CAPACITY IN FARADS)

$(A) = 1 - e^{-\mu x}$ = RADIATION ABSORBED IN THE DEFECT

e = BASE OF NATURAL LOGARITHM

u = X-RAY ABSORPTION COEFFICIENT OF THE PENETRATED MATERIAL IN cm^2 FOR MONOENERGETIC X-RAYS, EQUIVALENT TO PEAK PHOTON ENERGY.

x = RADICAL DEPTH OF THE DEFECT

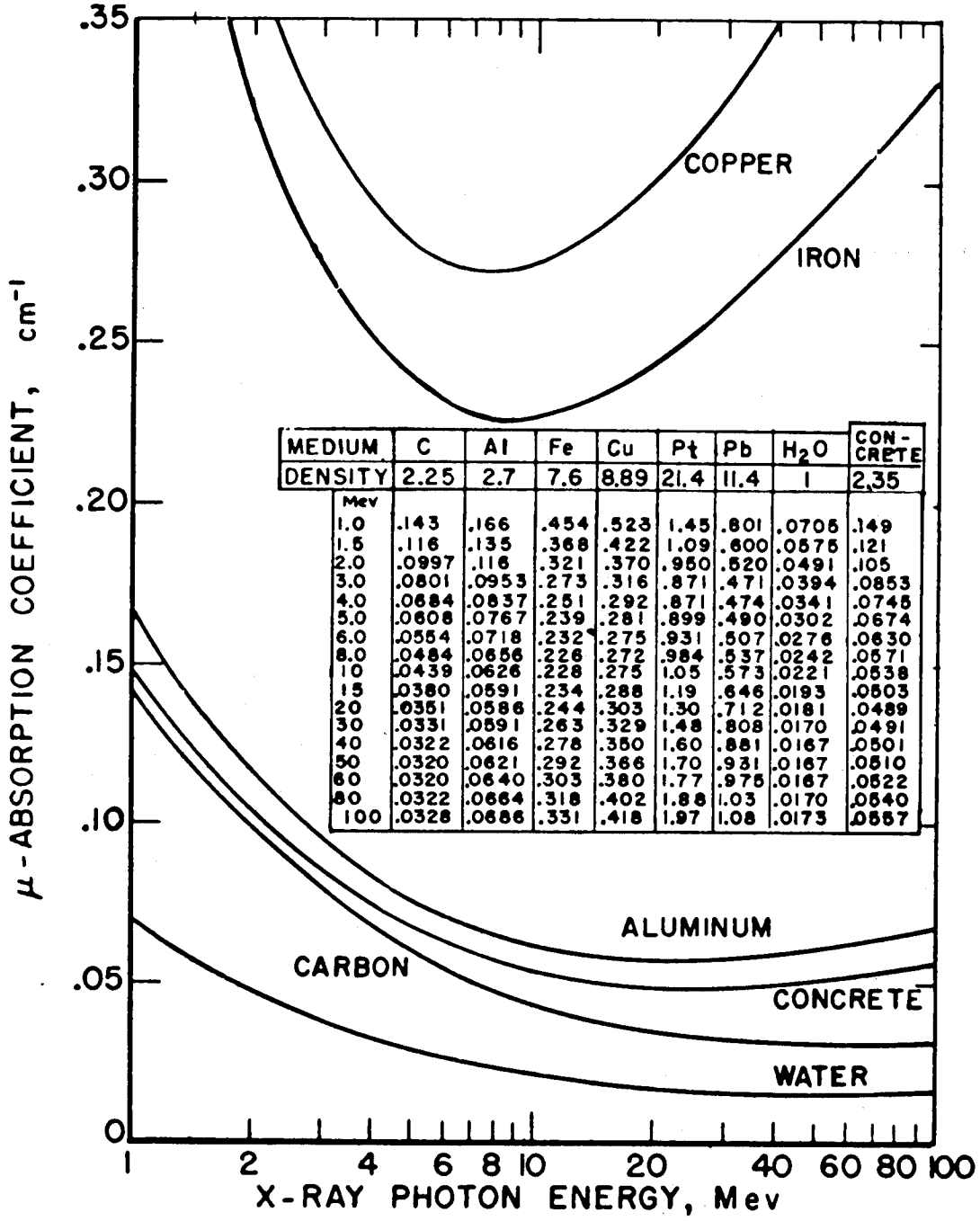
$(B) = 1 - e^{\frac{-t}{RC}}, t = \frac{l}{v}$

l = DETECTOR COLLIMATOR APERTURE IN THE SCANNING DIRECTION

v = SCANNING SPEED PER SEC.

Fig. 3

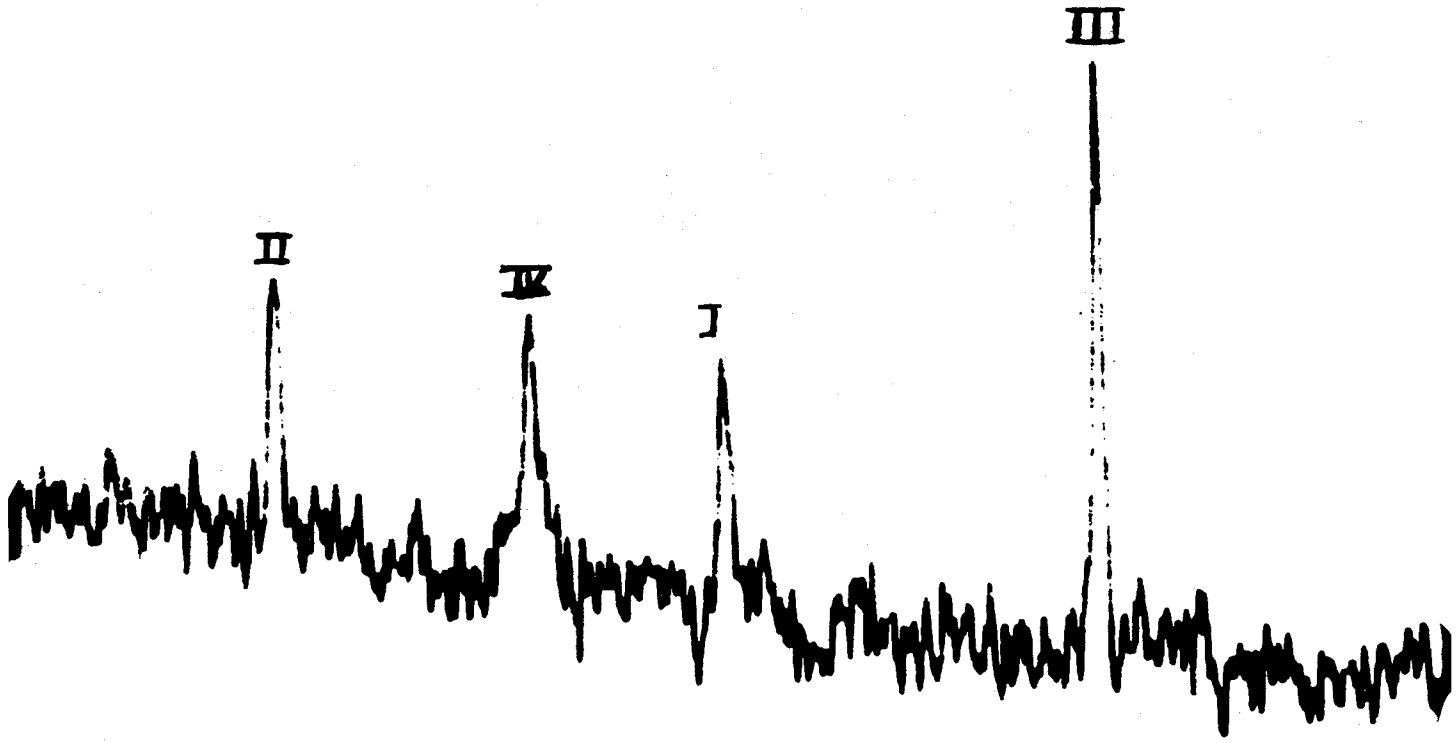
Appendix E. X-ray Absorption Coefficients for Monoenergetic X-rays



MEDIUM	C	Al	Fe	Cu	Pt	Pb	H_2O	CONCRETE
DENSITY	2.25	2.7	7.6	8.89	21.4	11.4	1	2.35
Mev								
1.0	.143	.166	.454	.523	1.45	.801	.0705	.149
1.5	.116	.135	.368	.422	1.09	.600	.0575	.121
2.0	.0997	.116	.321	.370	.950	.520	.0491	.105
3.0	.0801	.0953	.273	.316	.871	.471	.0394	.0853
4.0	.0684	.0837	.251	.292	.871	.474	.0341	.0748
5.0	.0608	.0767	.239	.281	.899	.490	.0302	.0674
6.0	.0554	.0718	.232	.275	.931	.507	.0276	.0630
8.0	.0484	.0656	.226	.272	.984	.537	.0242	.0571
10	.0439	.0626	.228	.275	1.05	.573	.0221	.0538
15	.0380	.0591	.234	.288	1.19	.646	.0193	.0503
20	.0351	.0586	.244	.303	1.30	.712	.0181	.0489
30	.0331	.0591	.263	.329	1.48	.808	.0170	.0491
40	.0322	.0616	.278	.350	1.60	.881	.0167	.0501
50	.0320	.0621	.292	.366	1.70	.931	.0167	.0510
60	.0320	.0640	.303	.380	1.77	.975	.0167	.0522
80	.0322	.0664	.318	.402	1.88	1.03	.0170	.0540
100	.0328	.0686	.331	.418	1.97	1.08	.0173	.0557

Fig. 4 *Absorption coefficient of X-rays in cm^{-1} plotted as a function of X-ray photon energy in million electron volts.*

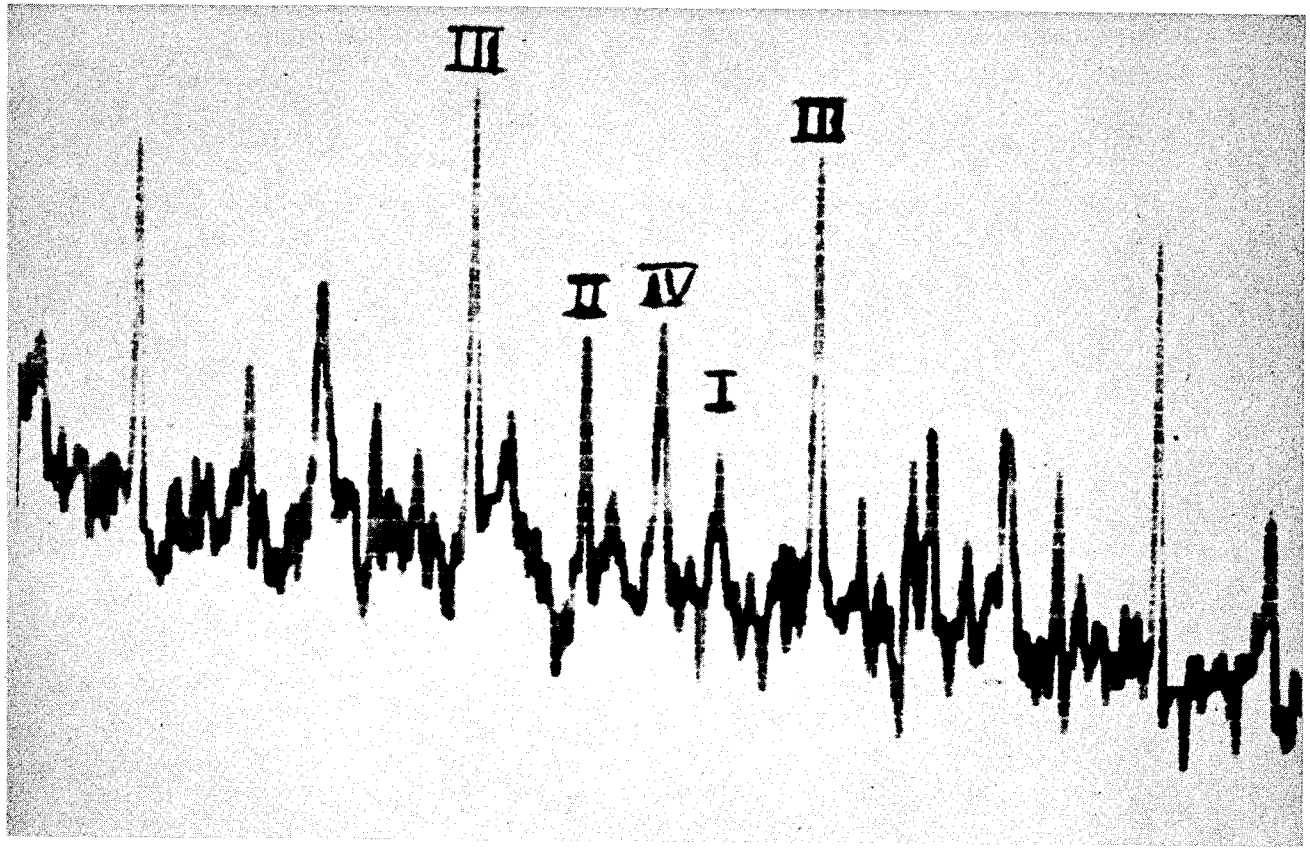
These data were taken from Gladys White, X-ray attenuation coefficients (unpublished results, 1952). It should be emphasized that the absorption coefficients given in the figure apply to a monoenergetic X-ray or X-rays and not to a continuous distribution of X-rays as obtained from an electron accelerator. The densities of the media given in the table are in units of grams per cubic centimeter.



Scintilator detector at 22 Mev betatron.
Absorber: max. propellant thickness of second-stage POLARIS.
scanning speed: .14 inch/second
Simulated cracks in the propellant:
1 inch long in the X-ray beam path:

I. .07 x 1 inch wide
II. .125 x 1 inch wide
III. .25 x 1 inch wide
IV. Lead .010 inch long in the radiation path, 1 x 1 inch wide

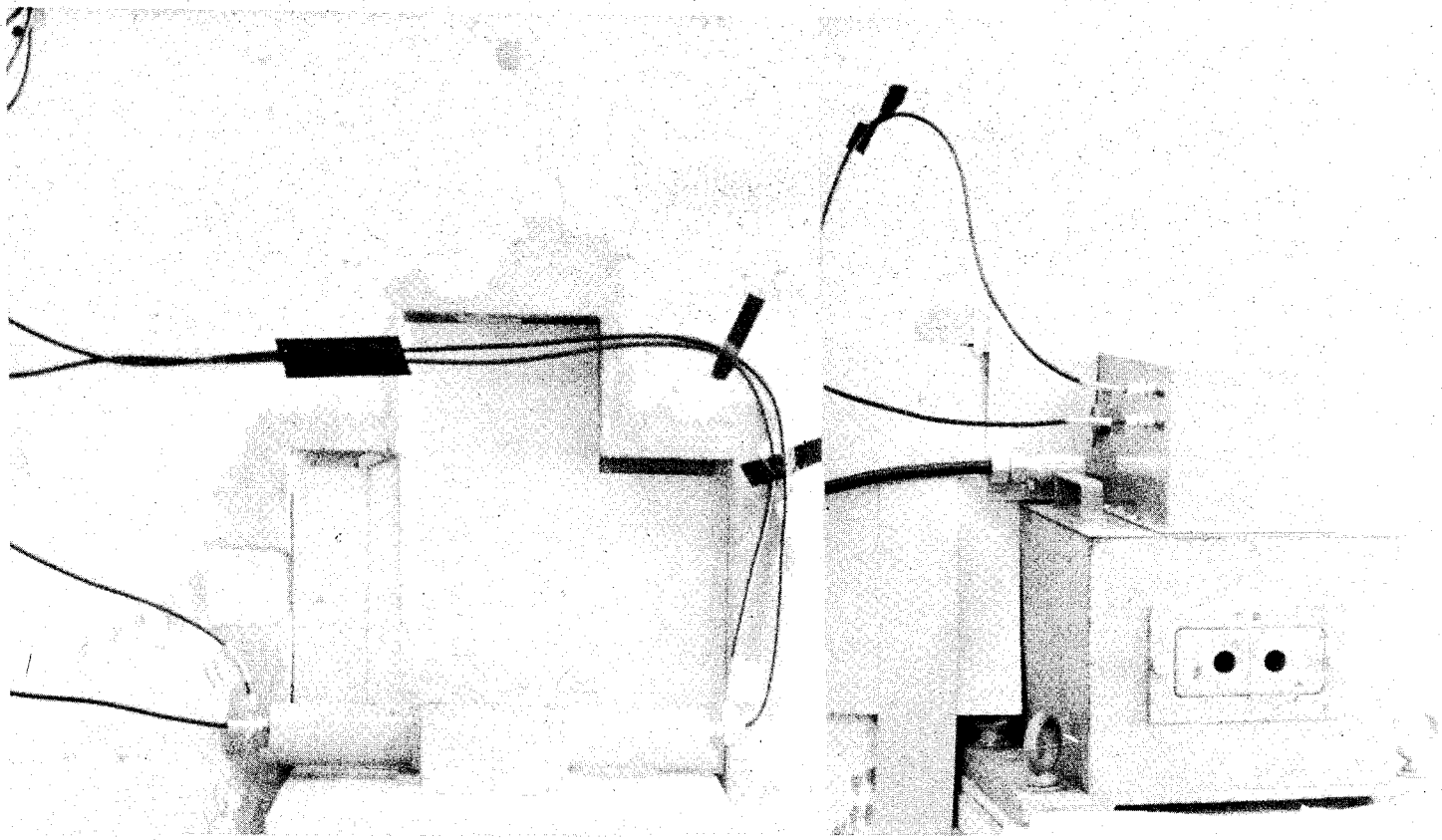
Fig. 5



Scintilator detector at 22 Mev betatron.
Absorber: max. propellant thickness of second-stage POLARIS.
Scanning speed: .68 inch/second
Simulated cracks in the propellant:
1 inch long in the X-ray beam path:

I. .07 x 1 inch wide
II. .125 x 1 inch wide
III. .25 x 1 inch wide
IV. Lead .010 inch long in the radiation path, 1 x 1 inch wide

Fig. 6



SCINTILLATOR ION CHAMBER

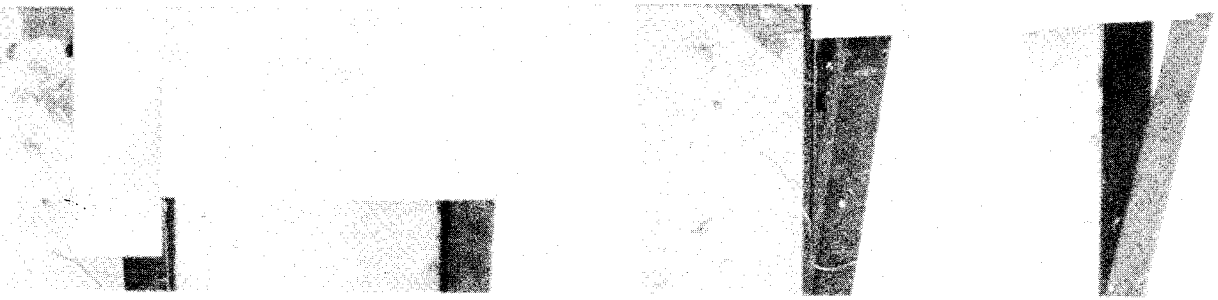


Fig 1

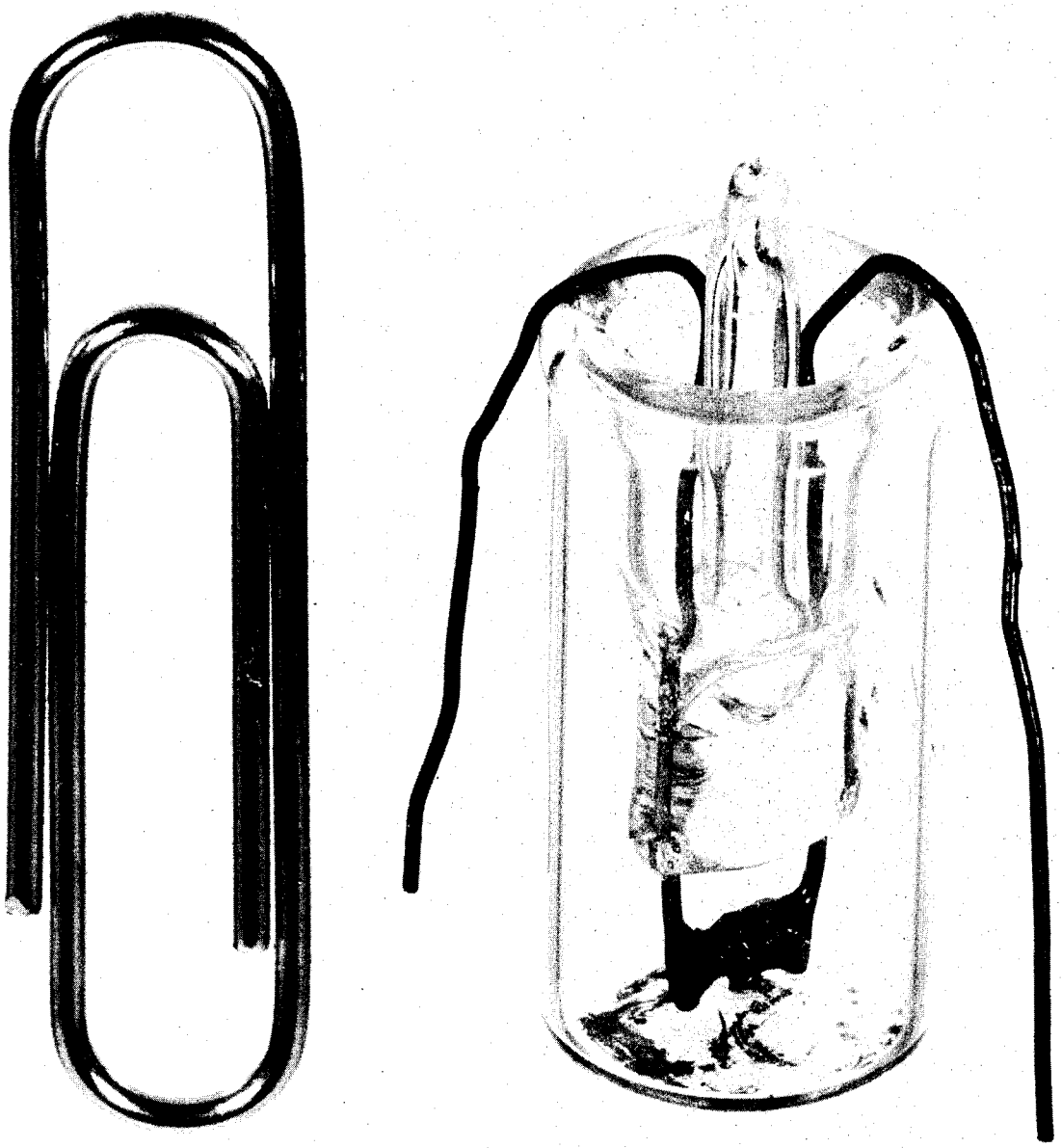
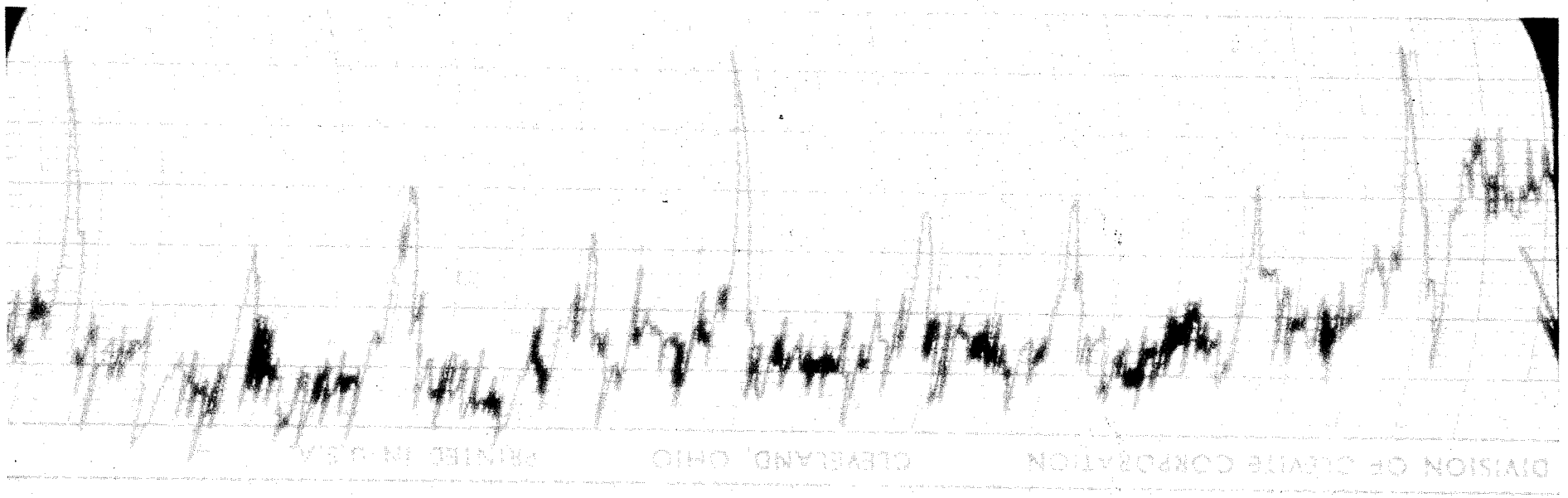


Fig. 8



Absorber, at the betatron, 26 in. Al. = 46 in. poly.
Recorder: Brush, electric burning stylus
"Defects": Lead 1/2" x .125", grain (1.64 density),
1" x .07, 1" x .125, 1" x .187
Speed: 7 seconds/inch - .0035r/sec

Fig. 9

NONDESTRUCTIVE TESTING APPLICATIONS OF X-RAY ANALYSIS 10969-06

by OTTO RENIUS
Physical Sciences Laboratory
U. S. Army Ordnance Tank-Automotive Command

In November 1895, Wilhelm Konrad Roentgen, a professor of Physics at the University of Wurtzburg, Germany made a discovery which laid the groundwork for the development of many nondestructive test methods. Professor Roentgen discovered X-radiation. This discovery came at a time when the greatest physicists of the world honestly believed that all the physical truths were known. Their complacency was completely shattered; for Roentgen's discovery led directly to the inexhaustible field called "Modern Physics."

Those in the nondestructive testing field are well aware of the importance of the discovery of X-rays whenever a radiographic or fluoroscopic examination is made. However, to avoid complacency in the use of X-rays, nondestructive test laboratories must examine the applications of X-ray analysis to many problems. Here is an important, truly nondestructive, tool which can be used for the examination of materials, particularly in the crystalline state.

THE NATURE OF X-RAY ANALYSIS

X-rays are short wavelength electromagnetic radiation lying between but overlapping portions of Gamma rays and ultraviolet rays. As seen in Figure 1, the wavelength range for X-radiation is approximately 0.06 Angstroms to 1019 Angstroms. Gamma rays and X-rays share a portion of the electromagnetic spectrum. They are identical in their interaction with matter and differ only in their method of generation. Also, X-rays and ultraviolet rays cover a joint portion of the overall electromagnetic spectrum which extends into visible light. As the wavelength of the electromagnetic radiation increases, it passes through the infrared portion and finally into radio waves. This radiation is produced by the sudden stopping of swiftly moving electrons. The necessary parts of an X-ray generator are then;

1. A source of electrons, proceeding from a cathode.

2. A target or anode in the electron beam.

3. A means of applying a potential difference between the cathode and the target to accelerate the electrons.

These briefly are the requirements for X-ray generators such as those used in radiography or fluoroscopy. In addition to the generator, the components necessary to complete an X-ray spectrometer are shown in Figure 2. (a) A specimen holder for positioning the specimen at a measured angle to the X-ray beam. (b) A radiation detector or camera for detecting the diffracted beam. (c) A goniometer for measuring the angle of the diffracted beam. (d) A ratemeter or counter for measuring the intensity of the diffracted beam. (e) A recorder for a graphic presentation of the diffraction angle and beam intensity. Figure 3 illustrates another type of commercial X-ray spectrometer. The components are essentially the same as shown in the previous slide; the principal difference

being the vertical rather than horizontal goniometer. The vertical goniometer and detector are seen mounted on the platform of the X-ray generator. A diffraction camera is seen positioned at another X-ray port. The X-ray tube which extends vertically from the center of the generator has four ports so that the counter and three cameras can be used simultaneously. The ratemeter-scaler, pulse height analyzer, and recorder are rack mounted adjacent to the spectrometer.

X-rays are emitted in a continuous spectrum whose short wavelength limit is determined by the voltage of the tube, and in a characteristic spectrum which is formed when sufficient voltage is applied to the tube. Figure 4 illustrates the spectrum of a tungsten target tube operating at 100 kilovolts. The shortest wavelength of a tube operating at 100KV is 0.124 Angstroms. As the tube voltage is increased the short wavelength limit of the tube is extended toward shorter and shorter wavelengths. The characteristic spectrum seen superimposed on the spectrum remains constant at its wavelength position. It is this characteristic spectrum which is of primary interest in X-ray diffraction. By the choice of a proper filter, enough of the unwanted characteristic and continuous spectra can be eliminated to make the beam nearly monochromatic. This monochromatic X-radiation is used as the basis for X-ray diffraction.

X-RAY FLUORESCENCE

If X-rays of sufficient energy strike a target, they cause secondary X-radiation in a characteristic spectrum to be emitted by the material. This characteristic spectrum called X-ray fluorescence is unique for each element and is used as the basis for a method of chemical analysis.

The various elements in the sample to be analyzed emit their unique spectra when bombarded by the primary X-ray beam. The elements are identified by detecting the wavelengths present in the emitted spectra. This analysis is carried out with an X-ray spectrometer arrangement similar to that shown in Figure 5. The X-ray tube normally used for fluorescence analysis has a tungsten target which allows the tube to be operated at high current for maximum output. The sample to be analyzed is placed directly under the port of the X-ray tube and tilted in such a way that the fluorescent X-radiation strikes an analyzing crystal of mica, lithuim fluoride, or other material. The analyzing crystal focuses the secondary X-rays on the goniometer circle. By traversing this circle with the detector, the angular position of the fluorescent X-rays can be measured. The insert of the slide illustrates a typical recorder chart tracing as the detector is driven around the goniometer circle. It is seen that the elements within the specimen being analyzed emit sharp characteristic peaks which are easily located and identified. The several lines for each element are caused by emission as electrons drop from outer shells to replace those knocked out of the inner shells of the specimen atoms. It is readily seen that the X-ray spectrum is much less complex than the spectrum of the same element as found in optical spectroscopy.

Under normal analysis condition, only the emission from the K shell is used for analysis since the emission from the L and M shells of the atom are of long-

er wavelength and therefore more easily absorbed in air.

A geiger counter or other suitable detector is used to measure the intensity of the X-ray beam. Since the emission intensity of each element is proportional to the concentration of that element in the material being analyzed, the method is also quantitative. When accurate standards are used as a reference, the method is much more rapid than conventional chemical analysis and has the advantage of being nondestructive.

A comparison with emission spectroscopy shows that, in general, the X-ray method is superior for elements in the concentration range 1 percent to 100 percent. Below 1 percent, emission spectroscopy is superior in most applications. Figure 6 is a typical working curve showing the concentration of nickel in a sample as a function of the number of counts per minute detected at the 2θ angle for nickel. The curve is based upon a series of chemically and spectrographically analyzed standards. The standards are placed in the sample holder of the spectrometer and counted several times to determine the number of counts per minute given by a specific characteristic line of the element. Some error is introduced if the matrix material does not remain constant. For example, 25 percent nickel in chromium may not intersect the curve at the same place as 25 percent nickel in iron. In determining the concentration of nickel in an unknown specimen, the intensity of the fluorescent emission is determined at the 2θ angle corresponding to the characteristic line used for calibration with the standards. The number of counts at that position is then referred to the working curve and the concentration is read directly. One disadvantage of the X-ray spectrographic method of chemical analysis is the limit imposed by the absorption of longer wavelength radiation in the air path of the spectrometer. This limits the analysis by fluorescence to those elements with atomic numbers greater than 22 (Titanium). The lower limit can be extended to atomic number 13 (Aluminum) if a helium path is supplied for the spectrometer, and to atomic number 5 (Boron) if the analysis path is a vacuum.

X-RAY DIFFRACTION

X-ray diffraction provides a tool for looking into the atomic arrangement of a material. It supplies information on crystal geometry and structure. The fundamental equation of X-ray diffraction was expressed by Bragg in 1914. He verified the simple equation $\lambda = 2d \sin\theta$ where: λ is the wavelength of X-radiation, d is the spacings of the atomic planes in the crystal being analyzed, and θ is one-half the angle between the transmitted beam and the diffracted beam. Figure 7 shows the arrangement for the transmission technique used in X-ray diffraction. In this technique, the specimen is placed with its surface normal to the incident X-ray beam. The monochromatic X-rays are transmitted through the specimen, with a portion being diffracted at the 2θ angle. Since the technique depends upon the X-rays passing through the specimen, it is limited to material thicknesses in the order of several mils. The detector scans the goniometer circle and detects the diffracted rays from the various atomic planes of the sample. The angular position and intensity of these rays are then recorded. As in fluorescence analysis, a geiger counter is ordinarily used for

detecting the diffraction angle. However, photographic film can also be used to record the diffraction pattern resulting in a photograph as illustrated by Figure 8. This figure illustrates the type of photograph obtained when a highly oriented specimen is placed normal to the X-ray beam in the transmission technique. The X-ray film is placed at a distance of several inches from the specimen surface and is also normal to the X-ray beam. The particular specimen illustrated is stretched rubber. It is interesting to note that in the unstretched condition the rubber is amorphous similar to a liquid or glass. However, when the rubber is stretched or frozen, a highly oriented crystal structure is seen on the diffraction photographs. One disadvantage of the photographic method is the long exposure time required. This figure required an exposure time of 30 minutes. However, for metallic specimens, exposure times of several hours are not uncommon. By measuring the distance of the diffracted ray from the transmitted beam, the θ-angle can be calculated; then, from Braggs equation the "d" spacings of the atomic planes can then be found. A reference to published tables of atomic spacings will allow the material to be identified. To reiterate, the diffraction information discloses the various compounds and phases present in the specimen while the fluorescence method gives a measure of the elements present, whether these elements are free, combined, or both.

OTAC PROGRAMS

Past and current applications of X-ray diffraction techniques in nondestructive testing at OTAC are:

a. Identification of compounds in fuel sediment.

b. Determination of preferred orientation in rolled armor.

c. Structure analysis of corrosion resistant films.

d. Determination of residual stress in gears.

Figure 9 illustrates the recorder chart tracing of a small sample of sediment found in gasoline which had been stored for a prolonged period of time. The sample material was obtained by collecting the residue after the gasoline had been passed through a fine filter. This residue adhered to the filter paper which was then placed in the X-ray spectrometer. Filtered radiation from a copper target X-ray tube irradiated the sample of residue. The detector was driven along the goniometer circle and the intensity and angle of the diffracted peaks was recorded. The detector travels along the goniometer circle at a constant rate of 2° 2θ per minute. An auxiliary pen places a mark on the recorder chart paper at every degree of travel. It is then a simple matter to determine the 2θ diffraction angle. By measuring the diffraction angles, computing the atomic spacings, and referring to catalogues, the sediment was found to be composed of diethyllead carbonate and lead carbonate. The specimens of gasoline which had been stored a greater length of time showed a greater concentration of lead carbonate than the more recent gasolines. It was therefore concluded that the tetraethyl lead was suffering a form of decomposition under some

storage conditions.

The direction and intensity of crystal orientation in a material such as steel has a great effect upon the physical properties of the metal. For example: when sheet steel is reduced in thickness by cold rolling, the crystals orient themselves in a preferred direction. This leads to some undesirable directional properties in the metal, and causes splitting or cracking in subsequent operations. For other applications such as transformer cores, highly preferred orientation is desirable and may be necessary for maximum operation.

The extent of this crystal orientation can be readily checked by X-ray diffraction. Figure 10 shows an integrating pole-figure goniometer used to hold a specimen for preferred orientation studies. This device is used to hold a thin metallic specimen about 1 1/8" diameter and up to approximately 3 mils thick. The rolling direction of the specimen is normally used as a reference mark. By removing the usual sample holder of the X-ray spectrometer and positioning the integrating goniometer in its place, the spectrometer can be readied for orientation determinations in a matter of minutes. The sample is rotated and tilted at various angles to the impinging X-ray beam in the manner shown in Figure 11. The specimen holder raises and lowers the sample vertically at a frequency of two seconds per cycle. The length of this vertical stroke is one inch, which is sufficient to integrate a number of crystals. As the sample raises and lowers, it is rotated about on axis normal to the specimen surface providing the beta angle. After one complete revolution, which requires 48 minutes, contacts placed on the specimen holder activate a switch which turns the specimen 5 degrees about a vertical axis providing the alpha angle rotation. The construction of the specimen holder is such that alpha angles up to approximately 60° can be attained. Recorder chart tracings as shown in Figure 12 are used to indicate the intensity of the diffracted beam as the specimen changes position. Contacts on the pole figure goniometer are wired to the recorder to activate an indexing pen when the specimen changes alpha angle. Since the sample rotates with constant angular velocity, a continuous chart record of intensity versus Beta angle for each Alpha angle can be obtained in approximately 4 hours with little attention from the operator. As each alpha angle is recorded it may be analyzed. The background or random intensity level is determined. Multiples of random intensity are then drawn, and the Beta angle is recorded at each point where the curve crosses an intensity level. This procedure is followed for each Alpha angle recorded. A pole figure is then constructed to give a quantitative record of the crystal orientation. The pole figure, as illustrated in Figure 13, is a quantitative map of the crystal distribution and is a convenient method of showing the crystal positions in the specimen. This illustration is a pole figure for rolled aluminum. It is readily seen that a high degree of preferred orientation exists for the atomic planes measured at this diffraction angle. If the pole densities of other atomic planes are plotted, the figure appears entirely different. The pole figure is used by the metallurgist to interpret preferred orientation in terms of the crystallographic planes parallel to the direction of rolling. This determination cannot be made metallographically, and only X-ray diffraction can provide the complete information.

An X-ray diffraction study of protective films is able to supply some

information on the structure necessary to provide the optimum corrosion resistance. Figure 14 illustrates several diffraction patterns of titanium filmed by a vacuum deposition process at OTAC. Photograph A, in the upper left corner illustrates the structure of the base stock after it has been subjected to a temperature of 1600°F during a degassing process. This illustrates that the base has been annealed during the process and as a result is nearly randomly oriented when the titanium is deposited on it. The thickness of this stock is 20 mils. Photograph B in the upper right illustrates the titanium as deposited on an amorphous base of glass. The film thickness is approximately .4 mil. The titanium film shows random orientation as deposited on the amorphous base. Photograph C in the lower left corner shows the diffraction pattern of a .4 mil titanium film as deposited on a degassed ferrous base. The specimen shows some preferred orientation which may be due to heating during the evaporation process. However, the specimen is essentially randomly oriented. Photograph D in the lower right corner illustrates the diffraction pattern of the same specimen as shown in C, after the specimen had been subjected to a vacuum diffusion treatment at 1750°F. This diffused specimen now exhibits extremely preferred orientation as a result of being subjected to the high temperature. It is interesting to note that titanium deposited on steel shows poor adhesion and corrosion resistance before diffusion. After diffusion however, when a fiber texture is formed, the film shows good adhesion if the specimen is subjected to a 180° bend. The oriented film also exhibits good corrosion resistance properties and will pass exposure to 600 hours salt fog.

If a metal component is subjected to some form of elastic deformation, X-ray diffraction techniques can give a measure of the strain present in the metal. The atomic spacings of the grains change from their stress-free values to new values proportional to the amount of stress applied. This change in the atomic spacings causes a shift in the 2θ diffraction angle which can be detected by X-ray diffraction. The stress is calculated from this shift by using a calibration procedure or by a mathematical computation involving the elastic constants of the metal.

The accuracy of the method is dependent upon the ability to determine the exact angular position of the diffracted peaks. Methods of curve fitting and symmetry corrections are employed to measure these peaks as precisely as possible. Although greater accuracies may be attained under ideal conditions, the error in typical measurements on steel specimens is approximately $\pm$ 4000 to 5000 psi.

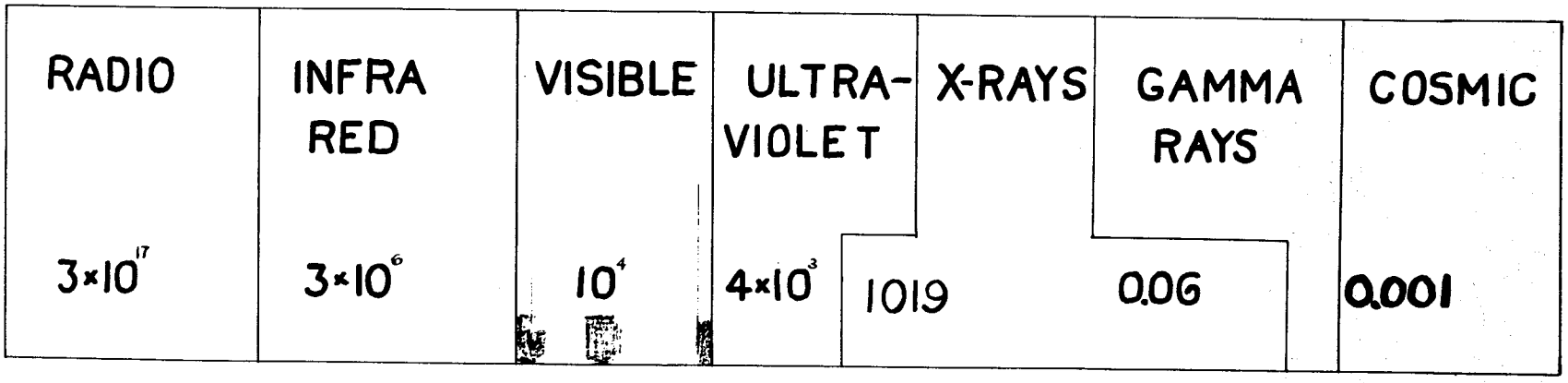
While these programs are typical of the nondestructive applications of X-ray analysis at OTAC, other procedures have been developed for specific tasks in other laboratories. For example, the thickness of thin films can be determined with a high degree of accuracy by determining the extent to which radiation diffracted from the base material is absorbed in the coating. The method is applicable to coatings in the thickness range .00004 in. to .004 in. and is useful for nondestructively measuring the thickness of electroplated films, evaporated coatings, and thin pigment layers.

The broadening of the X-ray diffraction lines is used as an accurate method

of determining the size of very small particles. The method compares favorably with the electron microscope for particles between 100 and 1000 Angstroms diameter. One disadvantage however, is that the method cannot be used for larger particles.

The techniques of X-ray analysis require skill in their application and evaluation but the results provide valuable information. From the outlined applications just shown, it must be concluded that X-ray fluorescence and diffraction are important industrial tools for nondestructive testing and their potentialities cannot be over looked as a means of quality assurance, particularly in defense materials.

ELECTROMAGNETIC SPECTRUM



WAVE LENGTH-ANGSTROM UNITS*

* 1 A = 10^{-8} CM

FIG 1

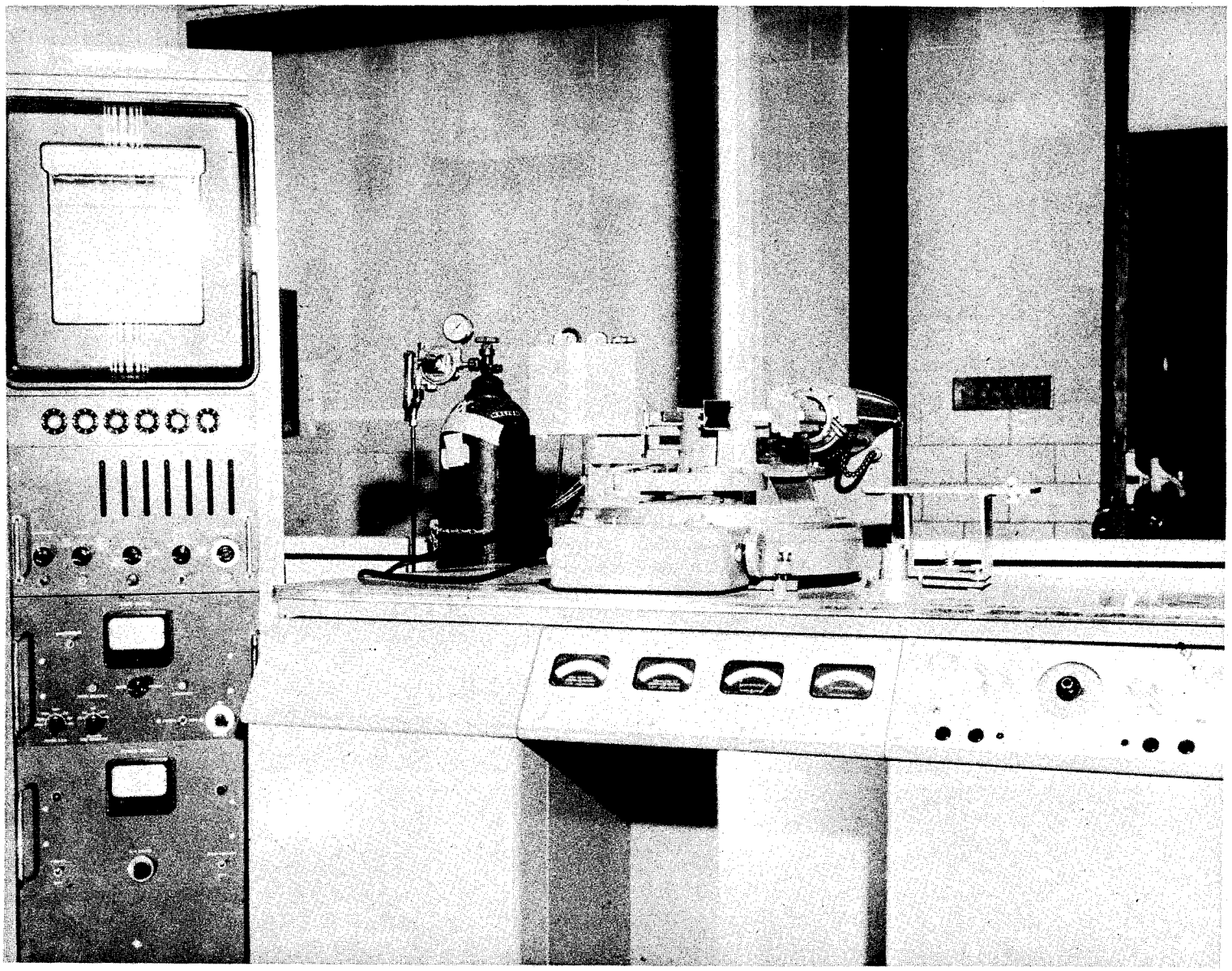
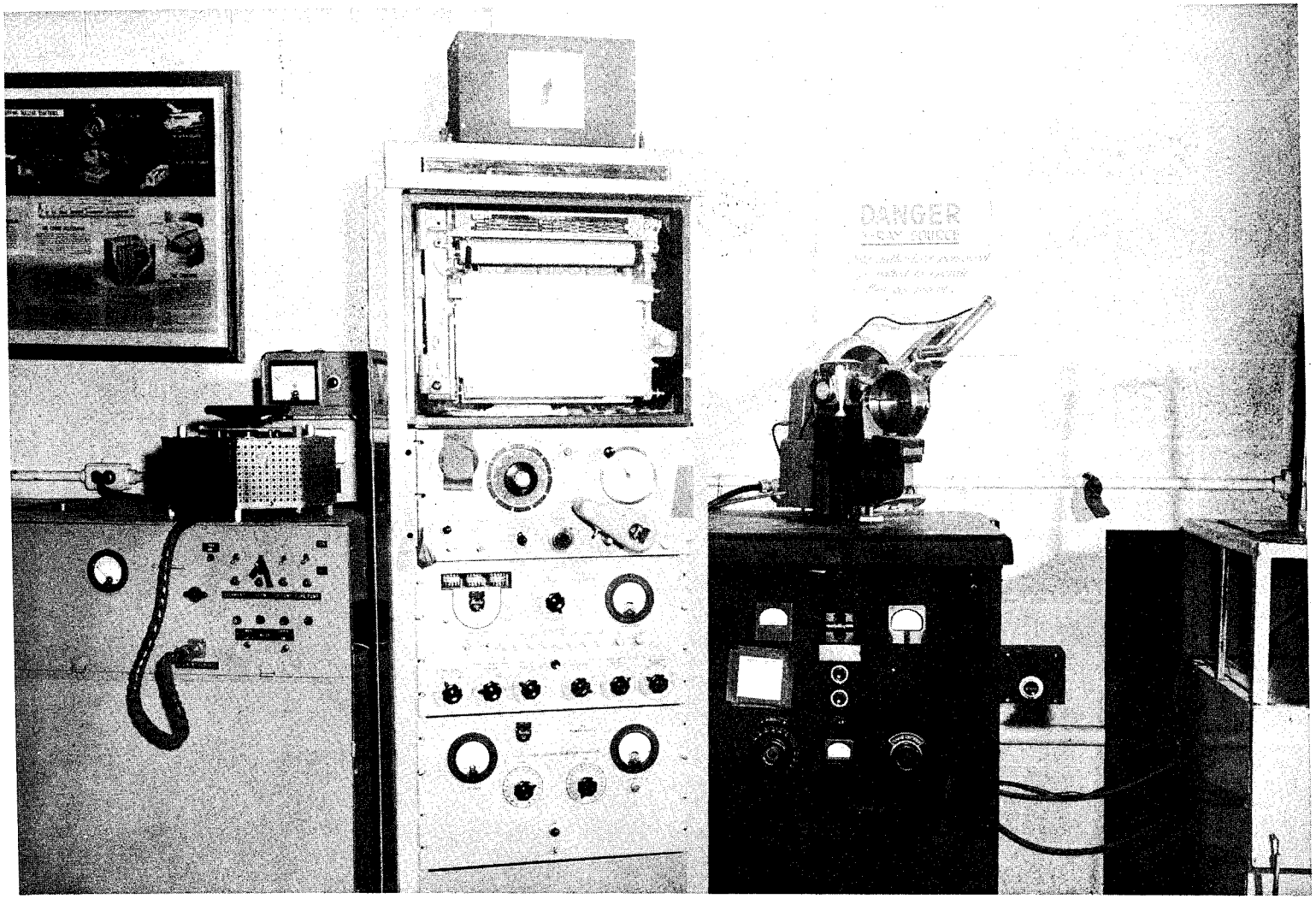
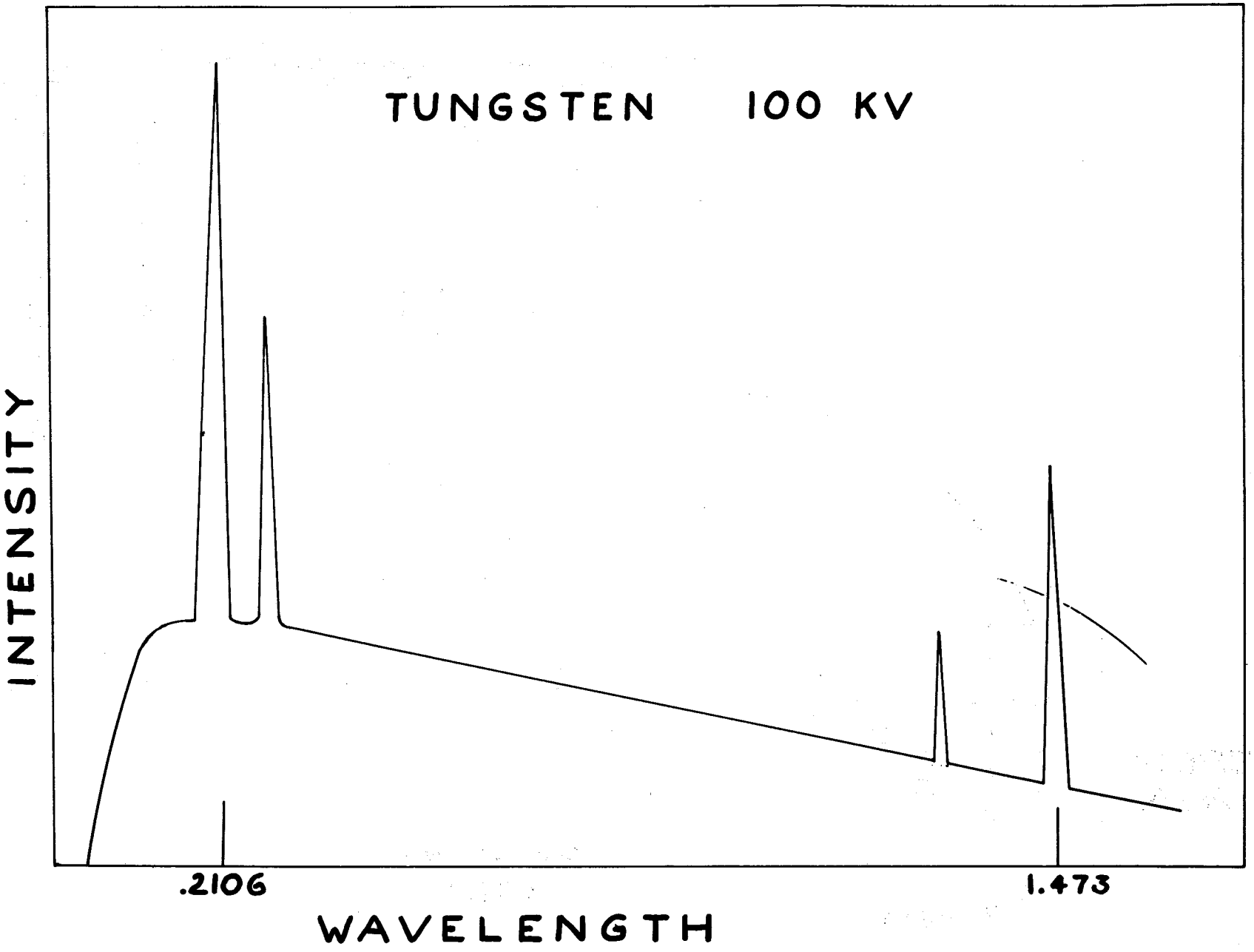


FIG 2



DETROIT ARSENAL NEG. NO. 66953 **DATE** 9 Aug 61
Applications of X-Ray Analysis. X-Ray diffraction unit vertical goniometer.

FIG 3


TUNGSTEN 100 KV
INTENSITY
.2106
1.473
WAVELENGTH

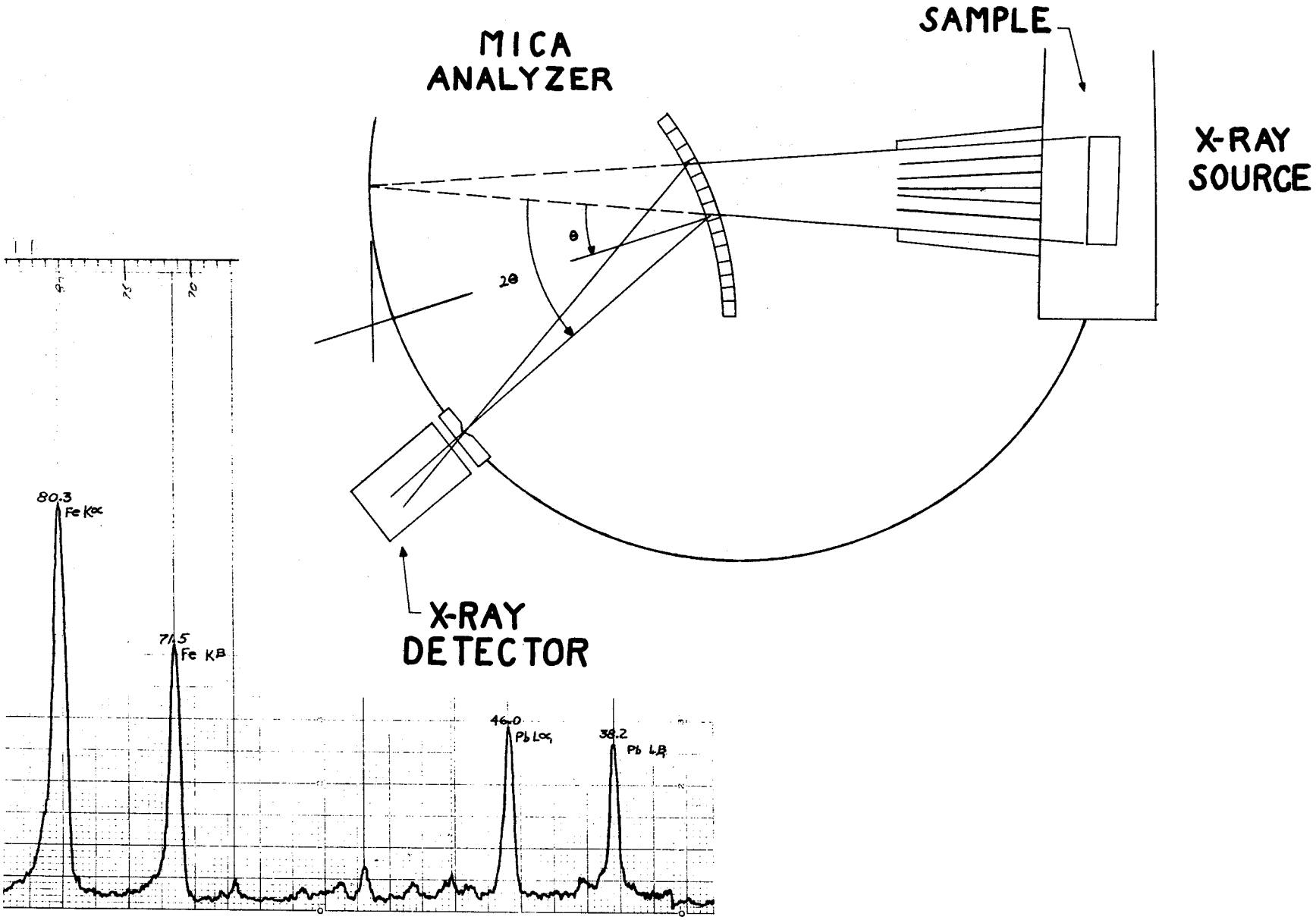

MICA
ANALYZER
SAMPLE
X-RAY
SOURCE
θ
2θ
X-RAY
DETECTOR
80.3
Fe Kα
71.5
Fe Kβ
46.0
Pb Lα
38.2
Pb Lβ

FIG 5

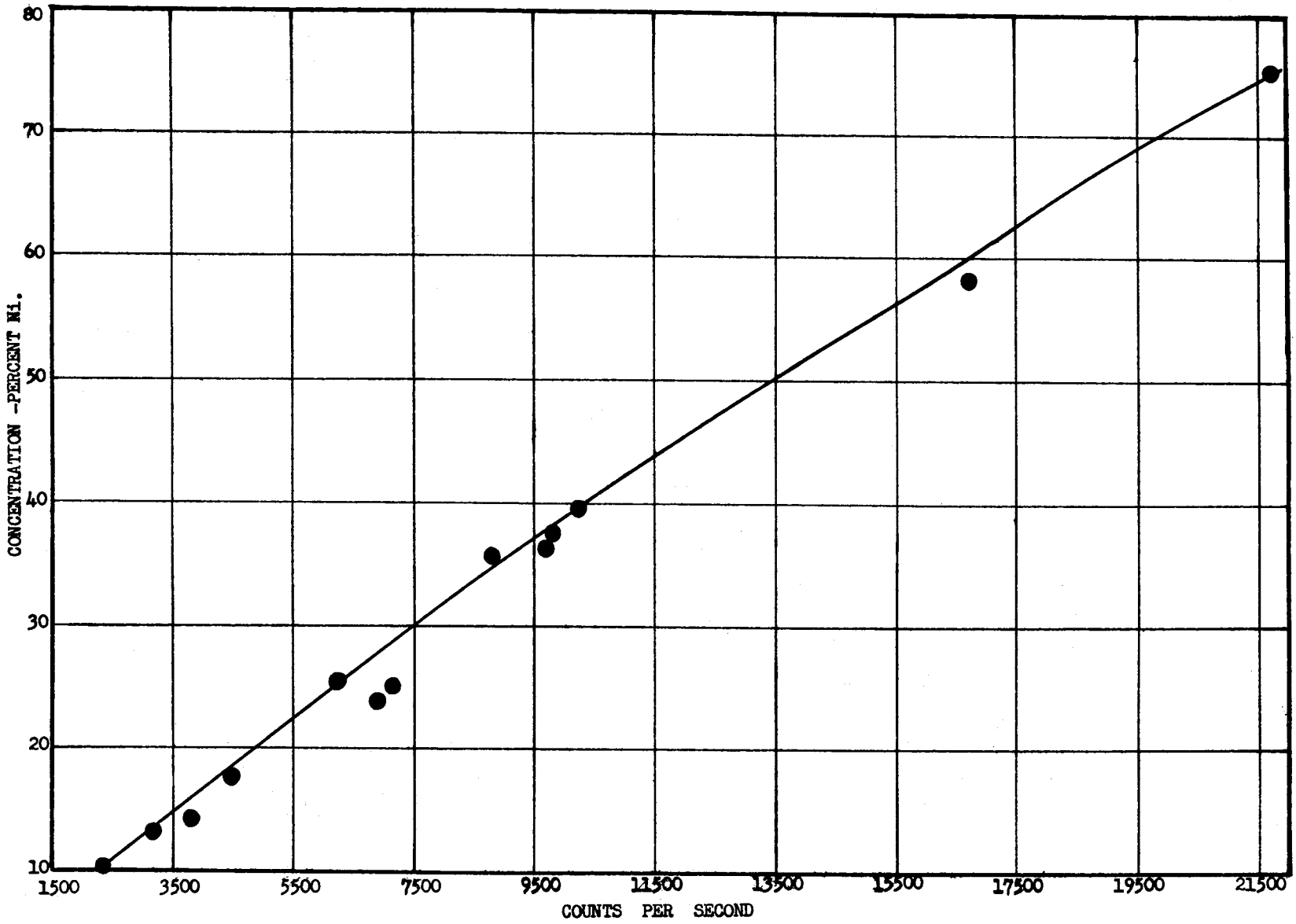
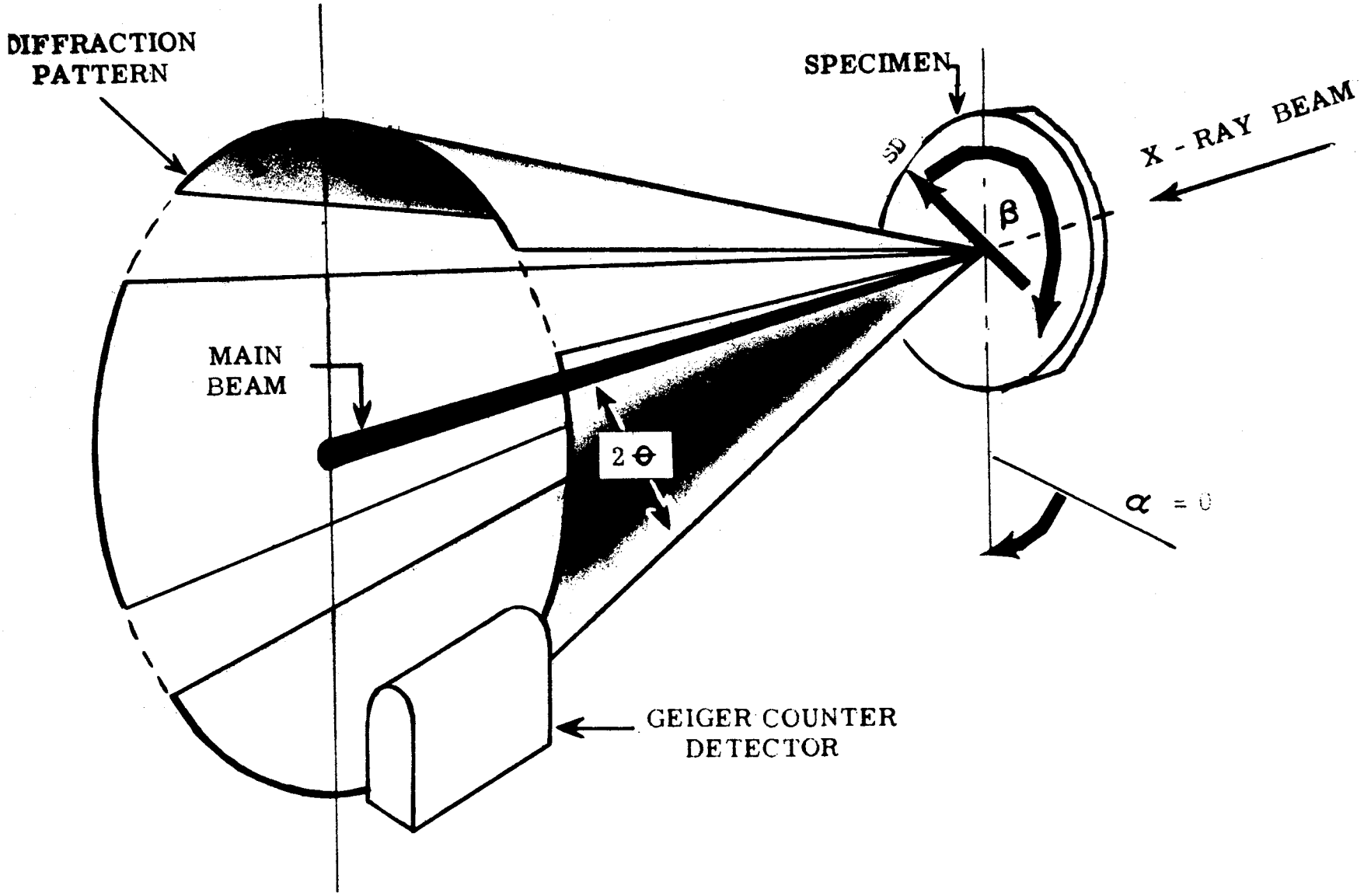

80
70
60
50
40
30
20
10
CONCENTRATION -PERCENT Ni.
1500
3500
5500
7500
9500
11500
13500
15500
17500
19500
21500
COUNTS PER SECOND

FIG 6



TRANSMISSION TECHNIQUE

FIG 8

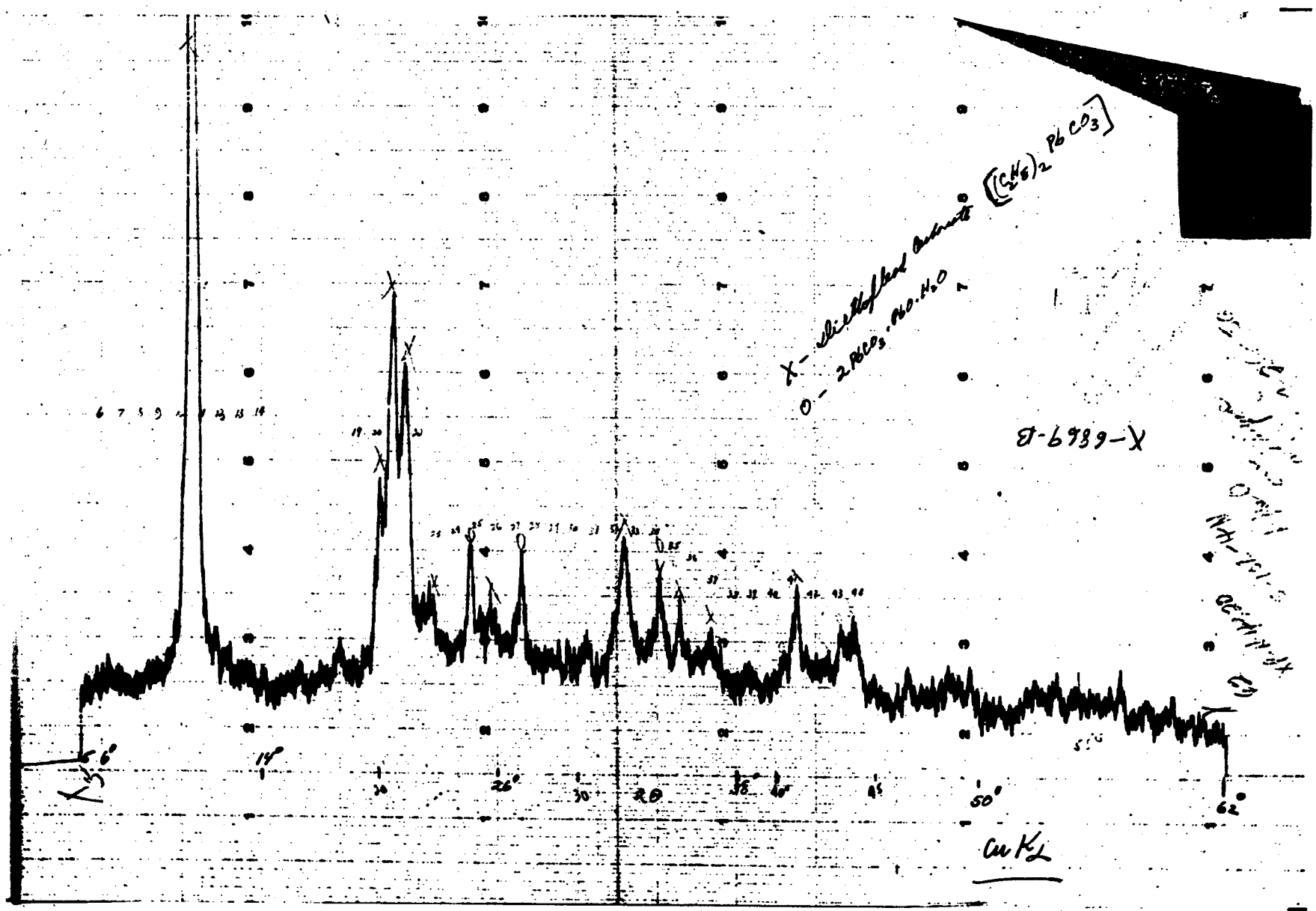

X - Lead Hydroxy Carbonate ((C4H6)2 Pb CO3)
O - 2 PbCO3 . Pb O . H2O
ET-6939-X
6°
14°
26°
30
20
36
50°
62°
Cu Kα

FIG 9

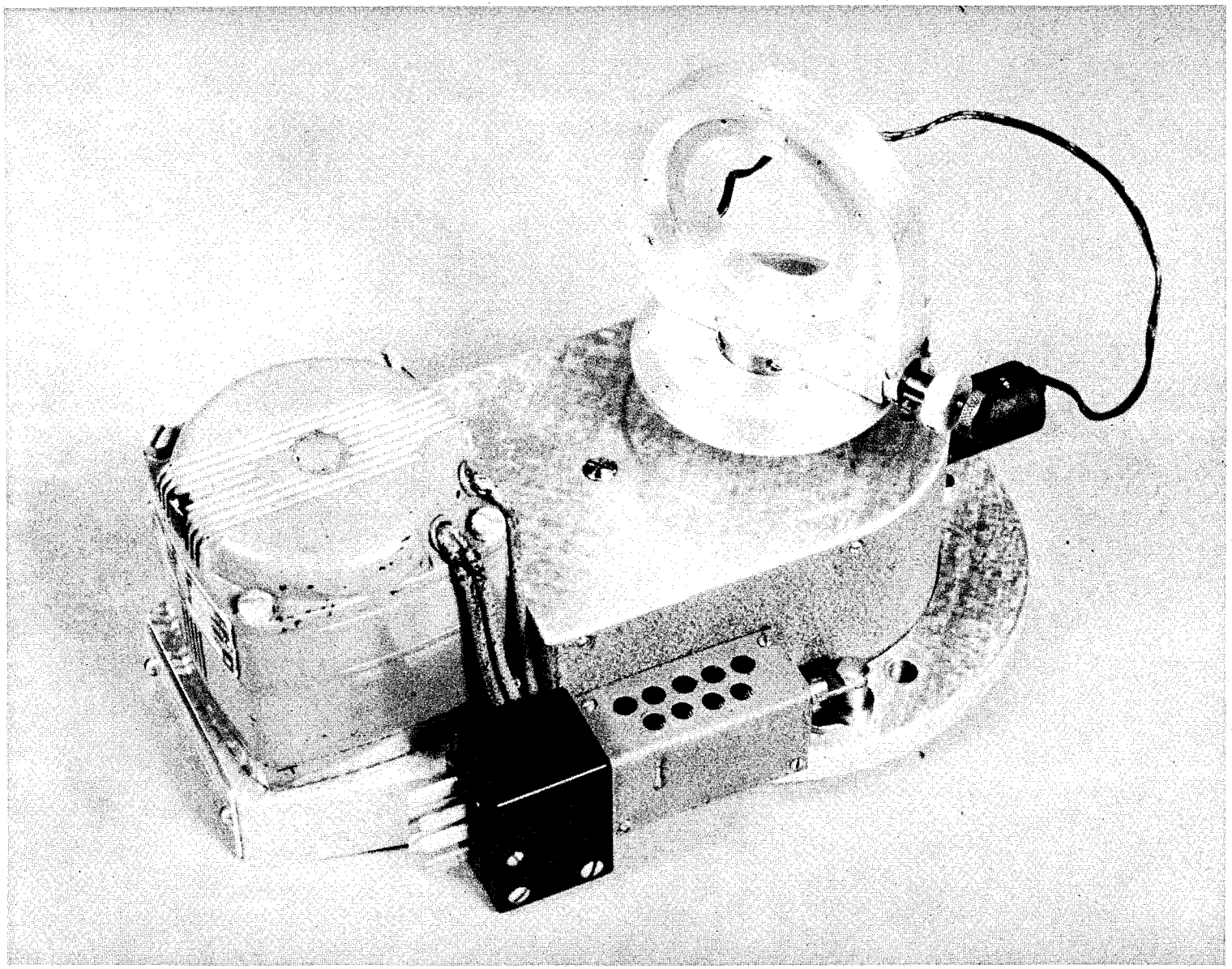
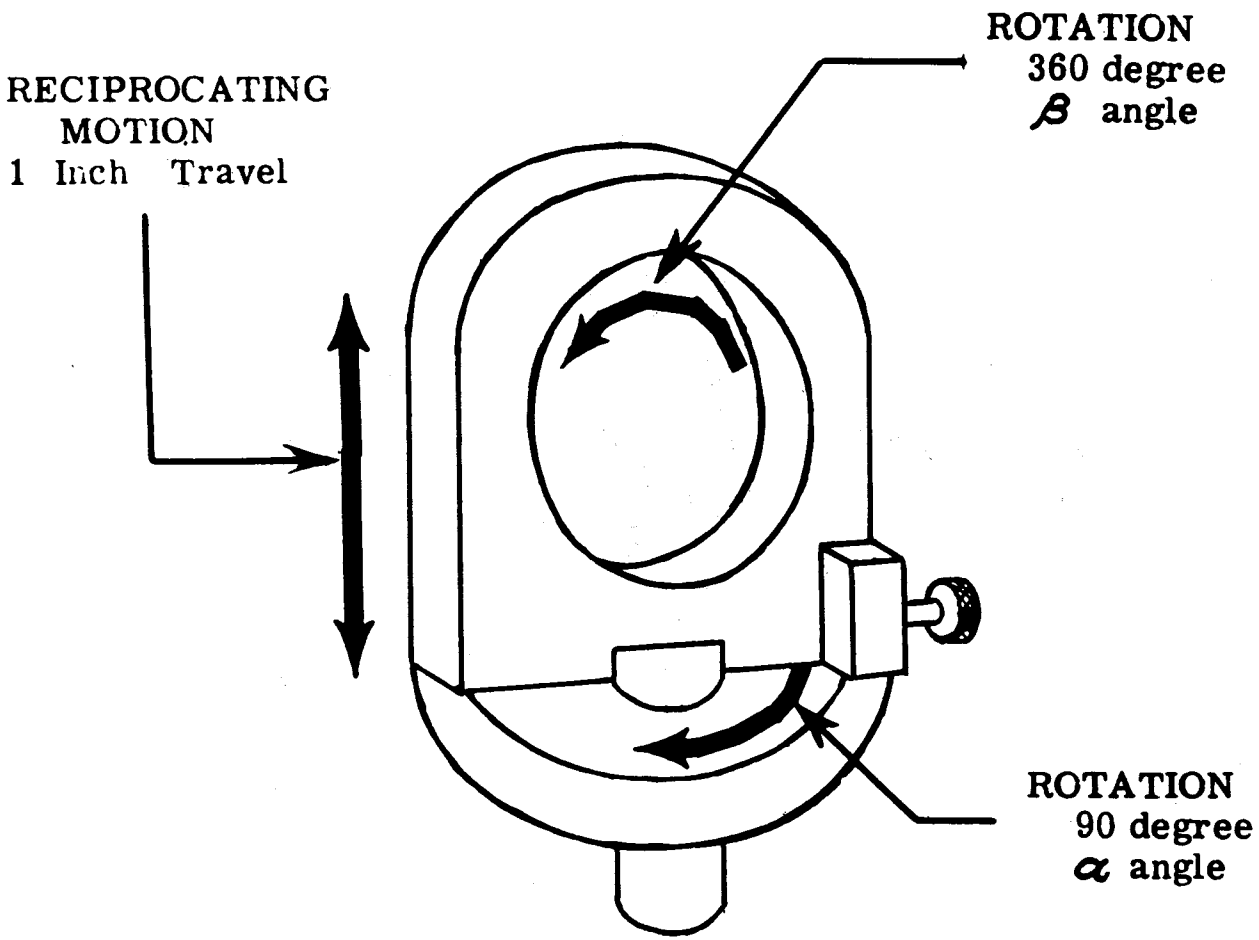


FIG 10



MOTIONS OF SPECIMEN IN THE INTEGRATING POLE-FIGURE GONIOMETER

FIG 11

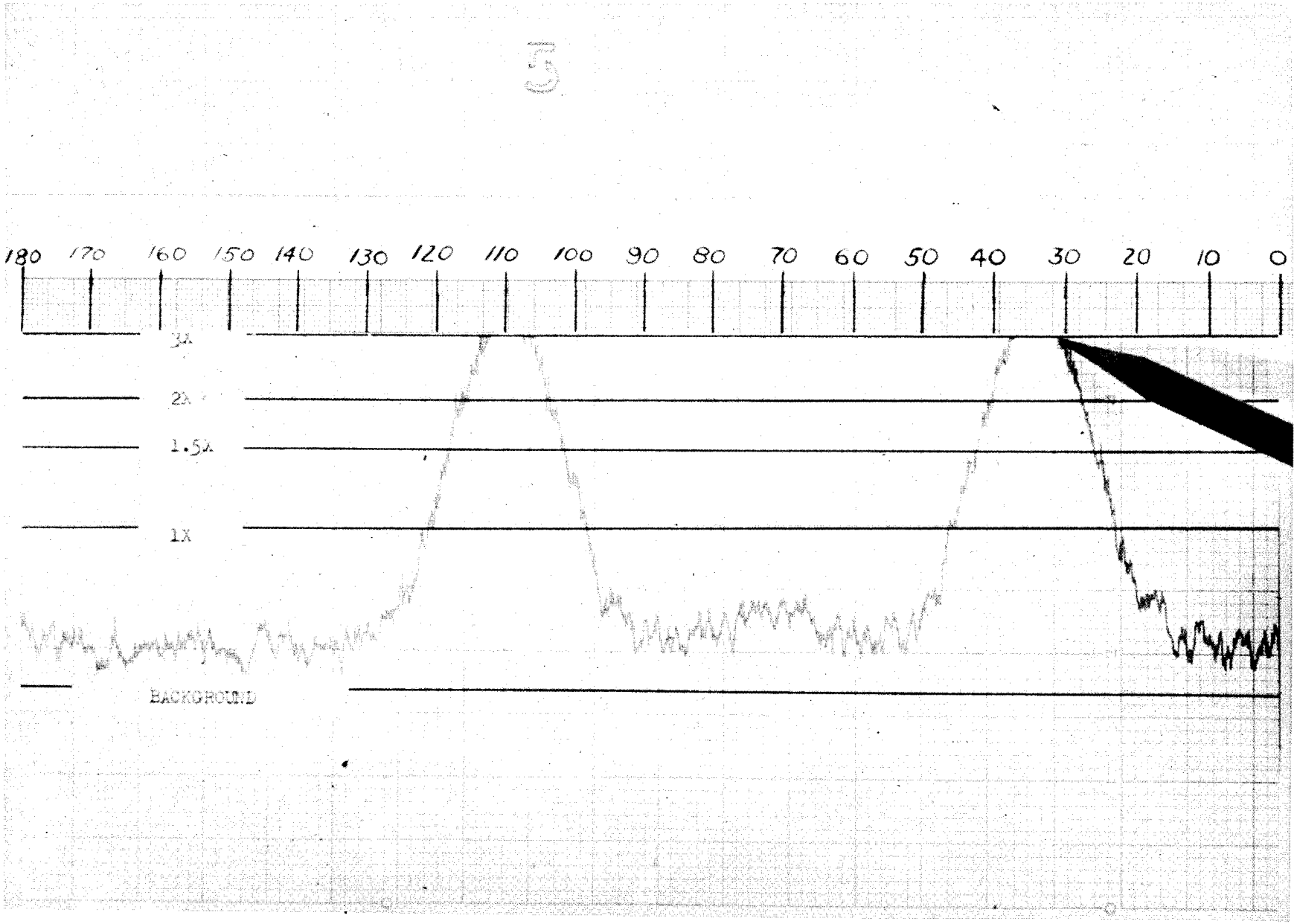
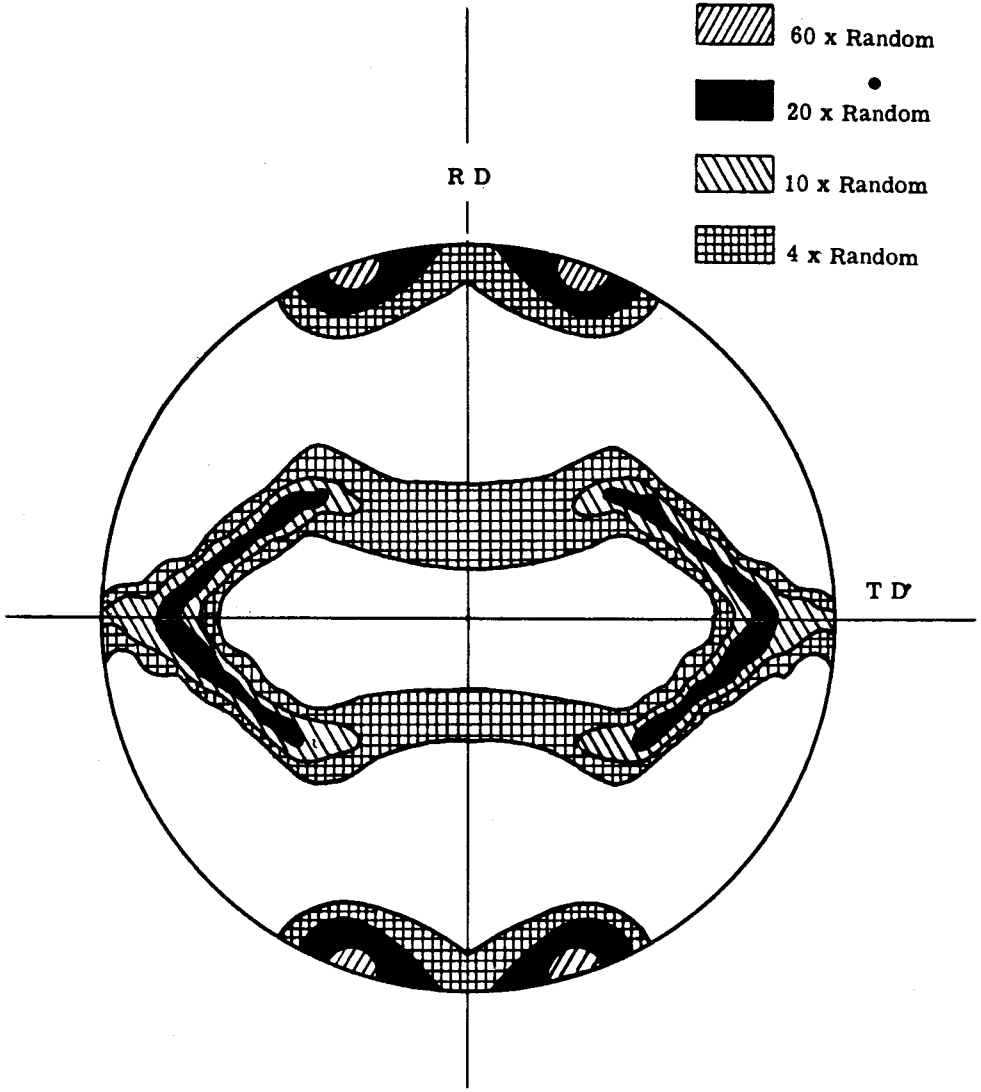
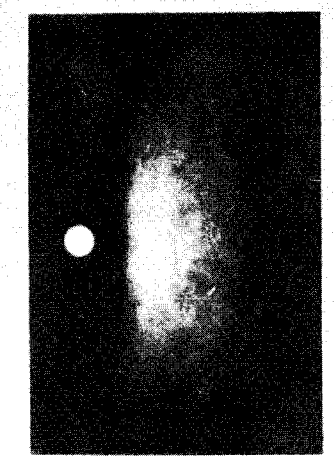

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
3X
2X
1.5X
1X
BACKGROUND

FIG 12



POLE FIGURE FOR (111) PLANE
OF ROLLED ALUMINUM

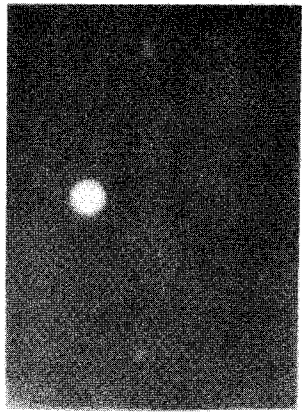
FIG 13



A. STEEL BASE MATERIAL AFTER VACUUM DEGAS AT 1600°F

B. TITANIUM COATED ON AMORPHOUS BASE

C. TITANIUM ON STEEL BASE AFTER DIFFUSION



D. TITANIUM ON STEEL BASE AFTER DIFFUSION AT 1700°F

FIG 14

1.

THE USE OF MAGNETIC RESONANCE IN NON-DESTRUCTIVE TESTING OF SOLID PROPELLANTS

10969-07

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I. Introduction

The purpose of this report is to give a brief review of magnetic resonance methods, to point out some applications which have been made in the field of chemical analysis and process control and to discuss some possible applications in the process control and inspection of explosives and solid propellants.

The initial discovery of Nuclear Magnetic Resonance was made independently by Purcell and Block in 1946. Over the past 15 years the major developments and investigations have been in the field of organic chemistry. Recently, some valuable applications have been developed in process control.

II. Theory

First let me review the basic phenomena of magnetic resonance.

Nuclear theory assumes that the nuclei of atoms are constantly spinning. Since these nuclei have electrical charges, this spinning should generate a magnetic field. The gyromagnetic properties of these spinning nuclei depend upon their shape and the distribution of the charge on their surfaces. Some nuclei behave as though they are non-spinning spheres with nuclear charge uniformly distributed over their surface. This type has no magnetic moment and are said to have a nuclear spin value of zero. They give no magnetic resonance signals. $Carbon^{12}$ and $Oxygen^{16}$ are included among these nuclei. Many nuclei behave as though they are spinning spheres with a uniform charge distribution over their surface. Based on certain quantum mechanics, considerations which will not be discussed here, these are assigned a nuclear spin value of 1/2. Included among these nuclei are $Hydrogen^{1}$ $Carbon^{13}$ $Nitrogen^{15}$ $Fluorine^{19}$ and $Phosphorus^{31}$. These spinning spheres behave in effect like spinning bar magnets.

If we apply a strong DC magnetic field to these spinning bar magnets at right angles to their spin axis, these spinning magnets will begin to wobble, or precess very much like a top. The rate at which this wobble occurs is a function of the strength of DC magnetic field, and the nature of the nucleus being measured.

If we now take a Radio-Frequency coil and place it at right angles to the DC field and apply an alternating field to this coil with the same frequency as the precession rate of these nuclei we will cause reinforcement of this precession and make the nuclei wobble in a much wider arc. If the strength of this RF Field is increased to sufficient intensity, the spinning bar magnets can be made to actually flip over. This phenomenon is called Nuclear Magnetic Resonance. It is possible to detect this flipping over of the nuclei if we place another coil at right angles to both the DC field and the RF Coil. A schematic diagram of what this Nuclear Magnetic Resonance spectrometer looks like is shown in Slide 1.

Note that during this operation, the nucleus has not undergone any change which will affect its chemical or physical properties. Since each nucleus has a specific precession frequency depending on its own nature and the surrounding chemical environment, it is possible by sweeping through a range of precession frequencies to obtain resonance signals from a series of different types of nuclei.

In actual practice, since it is very difficult to build an accurate RF oscillator of variable frequency, instead of varying the RF Oscillator to cause this sweep through the precession frequency range, we vary the DC magnetic field by means of a set of modulation coils on the large DC magnet. Since the precession frequency is a function of the strength of the DC magnetic field, this has the same net effect as varying the RF oscillator frequency.

The NMR spectrometer has a host of controls devices for stabilization and amplification, but these do not in any way change the basic principles stated here.

As was stated previously, all nuclei do not demonstrate this property of Nuclear resonance. Many of the most common elements such as Carbon^{12}, and Oxygen^{16} do not show any measurable resonance. This is because their nuclear charges are uniformly distributed and their nuclei seem to have zero spin. Slide 2 shows a list of atoms which respond to NMR and their precession frequencies.

Hydrogen^{1}	42.577
Fluorine^{19}	40.055
Phosphorus^{31}	17.235
Boron^{11}	13.66
Aluminum^{27}	11.094
Hydrogen^{2}	6.536

Another type of magnetic resonance phenomenon is called Electron Paramagnetic Resonance (EPR). This is an effect which is quite similar to Nuclear Magnetic Resonance except that it is attributed to certain orbital electrons

in the atom. Electron Paramagnetic Resonance can exist when an atom has an unpaired electron in one of its shells. Just as nuclei have charge, mass, angular momentum and magnetic moment, so do electrons. These results from not only the axial spin of the electron but also its orbital spin around the nucleus. When electrons are paired in an orbit, their gyromagnetic effects neutralize each other. However, when an unpaired electron exists, resonances can be detected. These unpaired electrons exist in elements with unfilled electron shells such as the transition elements (Vanadium, Chromium, Manganese, Iron, Cobalt, Nickel, Copper). They are also found in free radicals, radiation damage sites and free ions.

Since electrons are much lighter than protons and have much higher spin rates, their precession frequencies are much higher than protons. EPR measurements are in the microwave range (10,000 megacycles) and require Microwave oscillators, detectors and wave guides for transmission and detection of the signals. Otherwise the basic instrumentation is quite similar to NMR.

A third type of resonance that exists in a nucleus is called Nuclear Quadrupole resonance. If we go back to our spinning nuclei, we find that if we bring a unit electric charge near these spherical spinning protons with uniform charge distribution this charge will experience the same electrostatic field regardless of the direction of approach. This means that these nuclei have no quadrupole moment. However some nuclei behave as though the charge distribution is not uniform. If a unit charge were brought toward the spinning nucleus from different directions, it would encounter differences in the electrostatic field. These nuclei are said to have a quadrupole moment. They may show a concentration of charge at the equator, or at the poles and are called oblate, or prolate respectively.

The existence of this nucleus with a quadrupole moment, in the presence of the internal electrical field of a crystal gives rise to a precession phenomenon very much like the Nuclear precession which occurs in NMR. By subjecting the crystal to an RF field which sweeps back and forth in the precession range of the quadrupole, an absorption of energy occurs at each sweep through the precession frequency and resonance occurs. Chlorine^{35} exhibits this property of quadrupole resonance. Measurement of this quadrupole resonance may be of value in determining crystal latices and structures of crystal.

The method of measuring this quadrupole resonance utilizes an instrument similar to NMR but without the large DC magnet.

Up to this point the impression may have been given that every nucleus in a particular sample of material under test flips over when testing. This, of course, is not so. Actually, when a sample is subjected to NMR, the molecules in that sample are in a constant state of thermal agitation and their spin axes are in all conceivable orientations with respect to the magnetic fields. Actually, there are only a few nuclei precessing and flipping over at any one time. If we place a sample in NMR spectrometer and turn on the current in the DC Magnet, it will take a certain length of time for these oriented nuclei to start precessing. Also, thermal agitation will cause these precessing nuclei

to fall out of phase with the field and fall back to their original state while others will take their places. After a short time an equillibrium condition will be established in which essentially the number of precessing nuclei will become constant. If we now shut off the magnetic field the precessing nuclei will fall back to normal state and no new ones will take their places. The time it takes for the initial equillibrium to be established after application of the magnetic field and the time it takes to fall back to zero nuclei in the precessing state due to thermal agitation after turning off the magnet is referred to as the Relaxation time (T_1). This time can very from 10^{-5} to 10^5 seconds depending on nature of the nuclei.

Another type of relaxation at work in this system is the interaction of neighboring nuclei with their environment (i.e. the effects of the nuclei on each other due to the various attractive or repulsive forces at work in the system) (T_2). In the case of liquids where the nuclei do not remain in fixed positions relative to each other this coupling effect is small and T_2 will be long. For solids, and highly viscous liquids this T_2 value will be shorter, since the coupling or randoming effect will be greater.

The effect of the Relaxation time (T_1) is to control the rate at which we can drive the sweep rate of our spectrometer to obtain proper resolution of the signal and the effect of (T_2) is to establish the width or sharpness of the curve obtained from the spectrometer.

Slide 3, shows the difference in the type of signals obtained for solids and for liquids.

One more effect which must be considered here is one called saturation. The strength of the RF field must be selected in induce transitions in large enough numbers to be observed. However, if too much energy is used, transitions will be in such large numbers that the thermal equillibrium of the sample will be disturbed and resonance effects will cancel out. The RF saturation level therefore is very much dependent therefore on T_1 and T_2. This RF saturation effect can be utilized to separate two materials having close resonance frequencies but different T_1 and T_2 values and therefore different saturation levels.

III. Applications

By now the question in everyone's mind is how can all this theory be applied to process control and non-destructive testing?

Let me take these resonance phenomena, one at a time and show how they have been used or might be used for these purposes.

1. Control of Continuous Mixing of Composite Propellant

We have a continuous mixer for solid propellant formulation for a large missile motor in which we feed a steady stream of premixed fuels, binders and oxidizers. This continuous mixer produces mixed propellant at the rate of 100 lbs per minute. In order to get proper performance from this propellant the

proportions of the ingredients must be maintained constant and within a specified range of 1/2 percent. We subject this mixture of propellant to NMR at the outflow of the mixer after careful experimentation to determine the conditions under which we can obtain a usable NMR signal. In this particular case we are looking at the signals produced by the Hydrogen Protons in the Liquid Binder & Fuel and in the Solid Ammonium Perchlorate oxidizer.

The short relaxation time of the solid will produce a wide NMR signal while the longer relaxation times of the liquid portion of the mixture will give us a sharper resonance signal. Slide 4 shows an actual curve obtained for a solid propellant mix. Since the area under the NMR curve is proportional to the number of protons being counted, the ratio of the areas under the sharp portion and the broad portions can be determined. This will give the ratio of solid to liquid components. This curve can be scanned at a rate as high as 20 cycles per second. Actually a slower scanning rate is used.

The aluminum content of this mixture can also be determined by the proper selection of components. The precession frequency of aluminum is quite different from Hydrogen and can be detected separately. The amount of aluminum present can be determined from the height of the absorption curve.

To automate these signals to a control system is a comparatively easy job. The areas under the curve can be analyzed electronically and the data fed back to control centers which adjust the rates of flow of the feeding streams to make the necessary corrections.

Slide 5 shows the basic system design described here. This was developed by Southwest Research Institute under sub-contract from Thiokol-Longhorn. It is now actually being constructed by SWRI under an Air Force Contract for the control of a continuous mixer at the Thiokol Brigham City Plant where the Minuteman first stage motor is being loaded.

2. Proposed Application of NMR to Control of Solids to Liquid Ratio of TNT for Shell Loading in SPCC Process

In the loading of shell with TNT, in order to reduce the voids developed in the casting of the explosive and to expedite the cooling of the melt a method has been developed in which a continuous crystallization of TNT is permitted until a certain solids to liquid ratio is obtained before casting of the shell filling. Slide 6 shows the processing system. In this operation, the TNT is melted in the melt unit and molten material drops into the cooling and holding kettle where the melt is agitated, the melt cooling process started, and the air removal process which began in the melt unit is completed. When the melt reaches the pouring temperature, the air-removing vacuum is removed and the melt passes through a valve to a small reservoir. The melt is maintained at a constant level in the small reservoir so that a constant flow rate is obtained from the reservoir to a crystallizer, such as a Votator. The Votator crystallizer is used to form the nuclei crystals in the melt without introducing air into the system. When the amount of crystals formed reaches 12 + 1 percent, the shell should be loaded. It is therefore desired to measure the solids content of the

material coming out of the crystallizer for quality control both in a batch operation and in a continuous process.

At the present time, the control of this Single Pour Controlled Cool (SPCC) process is in the hands of an "expert" who from experience can divine the adequacy of the mixture flowing into the shell loading kettles. The need for objective process control is obvious.

Since there are relatively large differences in line width in the NMR signals from solid and liquid TNT it should be possible using techniques similar to those used in the previous case to determine the ratio of the areas under these curves and feed back the information obtained to control the temperature of the crystallizer and thus the ratio of solid to liquid phase.

3. Characterization of Raw Materials used in Manufacture of Composite Propellants

The fabrication of rocket motors with uniform performance characteristics required control not only of the ratios of the ingredients used in the propellant composition, but also a careful control of the individual ingredients used in the mixture. The physical properties of a composite solid propellant has a marked effect on the functioning of a motor in terms of temperature effects, handling and long term storage. These physical properties are in a great measure dependent on the high polymer used as binder and fuel.

Because of the complicated nature of these polymers it is difficult to accurately characterize them by wet chemical methods. Infra-red Spectroscopy has had some success, but frequently does not show up subtle structural deviations. High Resolution NMR can frequently give much information about high polymers in terms of functional groups and impurities. The term High Resolution NMR means exactly that. Instead of obtaining a single absorption line for the Hydrogen protons in a compound, it is possible, by some special techniques, to resolve this absorption line into a series of absorption lines. This series of absorption lines is the result of the effects of different functional groups in a molecule on the hydrogen protons in that molecule. What this actually means is that different hydrogen protons in a molecule will have slightly different precession frequencies because of the effects of adjacent nuclei. These changes in precession frequency are called "Chemical Shifts" and can be measured. In the next series of slides are given some examples of analysis of polymer by NMR which can be used to characterize these materials.

a. Polysulfide Resin (LP3 and LP33)

LP3 and LP33 polysulfide materials manufactured by the Thiokol Company and used in a series of Solid Propellant Motors have a structure which is considered to be as follows:

$$HS\ (C_2H_4\text{-}O\text{-}CH_2\text{-}O\text{-}C_2H_4\text{-}S\text{-}S)_n - C_2H_4\text{-}O\text{-}CH_2\text{-}O\text{-}C_2H_4\text{-}SH$$

It should be possible from examination of this compound under High resolution NMR to determine the ratio of the different functional groups present in the polymer and thus determine its molecular weight.

Slide 8 shows duplicate typical spectrogram obtained from a sample of LP33 resin which was subjected to NMR analysis. The areas of the spectrograms which are believed to represent the different functional groups are noted. Slide 7 gave assignment of spectral lines to the different functional group.

TABLE 3

Assignment of Line Groups in LP33 Spectra

Spectral Position of + Line Group	Functional Group
0.0	C_6H_6 Reference
+2.6	$-O-CH_2-O$
+3.5	$-O-CH_2-C-$
+4.4	$-C-CH_2-S-$
+5.8	-SH

This assignment is based on theoretical considerations and experience.

Quantitative treatment of these spectra consists of measurement of the areas under the line groups since the area under a line group is proportional to number of protons giving rise to the line group. The data obtained for a series of samples of LP33 resin are given in Slide 9.

TABLE 4

Areas Under Line Groups in LP33 Spectra (Normalized to arbitrary value of 250 for the Areas under the $-O-CH_2-C-$ and $-C-CH_2-S-$ groups

LP33 Sample	$-O-CH_2-O-$	$O-CH_2-C-$ and $-C-CH_2-S$ Average	-SH
45M	124	250	14.7
	130	250	15.9
68M	127	250	13.4
	127	250	14.7
79M	132	250	15.2
	139	250	15.6

TABLE 4 (CONT'd)

LP33 Sample	$-O-CH_2-O-$	$O-CH_2-C-$ and $C-CH_2-S$ Average of	-SH
81M	137	250	15.1
	130	250	16.9
83M	138	250	19.6
	139	250	19.6
85M	128	250	13.2
	133	250	15.4
87M	124	250	15.6
	127	250	15.4

From the data in Slide 9 and the structural formula it is possible to calculate the average molecular weight of the polymer. This is shown in Slide 10.

The relative number of protons for each functional group is as follows:

-SH	2
$-O-CH_2-C-$	$4N+4$
$-C-CH_2-S-$	$4N+4$
$-O-CH_2-O-$	$2N+2$

Where N+1 is the number of monomer units in the chain. Since the area under each portion of the curve represents the total number of protons from that functional group in the molecule, the average molecular weight can be calculated as follows:

$$\frac{\text{-SH (Area)}}{\text{Total -C-CH}_2\text{-S (Area)}} = \frac{2}{4N+4} \quad (1)$$

where (N+1) is the number of monomeric units and the average molecular weight is

$$166.25\ (N+1) + 2 = \text{molecular - wt.} \quad (2)$$

Substituting the areas given in Table 4 into equation (1) and solving for the molecular weight from (2) we can get the data shown in Slide 11.

TABLE 5

LP33 Polymer Lot No.	Number (N+1) of Monomer Units	Average Molecular Wt.
45M	8.16	1360
68M	8.90	1480
79M	8.11	1350
81M	7.81	1300
83M	7.53	1250
35M	7.88	1450
87M	8.06	1340

In this particular series of tests, the molecular weight is quite uniform. A lot of material could be readily picked out if the molecular weight were markedly different. Any unusual line showing the presence of an impurity might also be detected. For example an appreciable amount of water in the sample would show up as an increase in the size of the "bump" marked H_2O in Slide 8.

(2) Characterization of Other Ingredients of Polysulfide-Perchlorate Propellant

In addition to LP33, a number of other materials used in the manufacture of Polysulfide propellants have been investigated. Figures 16 thru 20 show high resolution NMR Spectra of Liquid Polymers LP-3MV, LP-205, Venzyl Mercaptan, Butyl Carbitol Formal (TP90B) and GMF (a mixture of Quinone dioxime and Monoxime).

These spectra are marked with the functional groups which are believed to be represented by corresponding spectral lines. Analysis of these materials can be done from these spectra using the approach used for LP33.

Proposed Application of NMR to Continuous Process Control of Spent Acids in Nitrocellulose

In propellant and explosives manufacture, nitration is a major process. In most nitration plants, the spent acid from the reaction is fortified with nitric and sulfuric acids to bring the mixed acid back to the level required for recycling. Before this can be done, chemical analysis is performed before adjustments can be made.

It is believed that this time consuming and inaccurate process can be automated and the accuracy improved by application of NMR. Slide 17, shows a flow diagram of the acid system in a nitration plant. The dotted boxes show the points where NMR control instruments could be inserted to analyze the composition of the spent acids, compute the corrections to be made and meter in the correct amounts of fortifying acids.

An analysis of the system for NMR analysis is shown in Slide 18.

TABLE 2

Nuclear Spin Resonance
Frequencies at 10,000 Gauss.

	Resonant Frequency	Relative Sensitivity
$Hydrogen^{1}$	42.577 MC	100%
$Nitrogen^{14}$	3.076	0.1%
$Sulfur^{32}$	0.0	--
$Sulfur^{33}$	3.266	0.23%

Since S^{33} has a natural abundance of only .74% the signal level will be extremely low and probably lost in the noise of the system. The wide separation in resonant frequency between $Hydrogen^{1}$ and $Nitrogen^{14}$ makes it appear feasible to perform this analysis on these two components.

A good deal of experimental work will have to be done to work out the details of this application since there are many interfering substances present in the spent acids resulting from partial nitration and breakdown products.

The Use of Nuclear Quadrupole Resonance for Particle Size Control and Concentration of NH_4ClO_4

As I pointed out before, when a crystalline material which contains a nucleus having an electric quadrupole moment is placed in an RF field a resonant frequency can be found which is a function of the spin of the nucleus and the internal electral field of the crystal. If a measurement is made on a large single crystal a certain specific resonant frequency will be obtained. If this large crystal is broken up in to very small crystals, there will be a change in resonant frequency due to the change in the internal electrical field strength of the crystal. Although these changes are small, extremely accurate frequency measurements can be made which should be able to detect these changes in resonant frequency due to the different sizes of particles present. If a measurement were made of frequency VS amplitude for a ground crystalline sample, then a curve of the type shown on blackboard would be expected. The frequency would be proportional to the particle size of the crystal while the amplitude at that frequency should be a measure of the number of particles of that size in the sample. These values could be standardized by means of samples of sharp cuts of particle size measured optically. The total area under the curve would

represent the total amount of material present.

Since the sweep of the frequencies and measurement can be made rapidly this technique might be applied to a moving stream of processed propellant or airveyed stream of oxidizer coming from a grinder. The area under the curve would represent the amount of ammonium perchlorate in a sample while the magnitudes at any points or the shape of the curve would represent the particle size distribution.

Interference would be expected only from those elements which have an electric quadrupole moment. Very few of the common elements exhibit this property and therefore there should be very little interference.

A program is now underway at Southwest Research Institute to obtain the basic data required to evaluate the practicality of this application.

Applications of Electron Paramagnetic Resonance (EPR)

A. Most promising areas for application of EPR to propellant processing is in its ability to detect very small quantities of the transition elements and for determination of free radicals. The high intensity of the signal from EPR makes it possible to detect extremely small quantities of the active materials.

A. Free Radicals

As stated previously, the possibility of obtaining an EPR signal in a molecule requires the presence of an unpaired electron. In normal organic compounds, the valence electrons are always paired. However in some compounds, such as diphenyl picryl hydrazyl, one of the bonds is unused, although the material is quite stable under ordinary conditions. Materials of this type are called "free radicals" and exhibit quite intense, narrow EPR absorbtion curves.

1. Application to Raw Material Characterization

In the examination of Polybutediene Acrylic Acid (PBAA) raw polymer it was found that five of six samples tested showed the presence of a natural EPR signal. The strength of the signals measured indicated the presence of a free radical which when calculated as the amount of diphenyl picryl hydrazyl required to give the same signal area gave the results in Slide 20.

TABLE 3

PBAA Lot No.	Free Radical (DPPH Equivalent) u gm per gm	Cavity Q with Sample in Place
RU-6	mil	600
RU-9	.068	820
RU-16*	.22	410*
TH-999	.21	580
TH-1071	.16	750
RM-90	.11	820

*After Vacuum Drying

B. Tracer Elements

Unpaired electrons may also occur in the inner (electron shells) energy levels of atom. The first of these in the periodic table is Scandium 21. In the same manner, Titanium, vanadium, chromium, manganese, iron, cobalt, nickel and copper each possess at least one valence that leaves an odd number of inner electrons in the 3rd Shell. There are also four more transition series of elements: yttrium through palladium (unfilled 4d Shell), the rare earths (unfilled 4f shell), Hafnium through platinum (unfilled 5d Shell) and the Uranium series (unfilled 5f Shell). All these compounds may give an observable EPR signal.

The mere possession of an unpaired electron does not necessarily mean that an EPR signal can be observed. Other factors in a compound may wash out this effect. For example, it may be observable only at very low temperatures.

In any proposed application of this type, therefore, it is necessary to study not only the EPR absorption signal of the particular metal compound but also its reaction with the envireonment in which it is to be made a tracer as well as the effect of that medium on the microwave frequencies to be used.

1. An Attempted Application

An example of the problems which arise in attempting a tracer application to propellant process control is given here.

In an effort to develop a quick method for determining the concentration of Polybutadiene Acrylic Acid (PBAA) resin in a composite propellant, it was found that a copper chelate, disodium cupric sequestrene (a derivative of ethylenediamine tetra acetic acid), gave a very striking EPR signal when 0.1% was dispersed in PBAA resin. The signal was unchanged after a prolonged period

of standing. These tests indicated that the CU-EDTA Chelate would be suitable as a tracer material for incorporation in PBAA for detection and measurement by EPR, if this compound could be shown to have no effect on the performance of the final material. Unfortunately, when the cross linking polymerizing agent was added to the PBAA resin it was found that this cross linking resin was so polar that almost none of the RF energy could get through the sample and it was impossible to make any measurements on the tracer material. This line of investigation had to be abandoned as far as application of EPR to this polymer system was concerned. However, the investigation did show the feasibility of using EPR to monitor a PBAA resin before addition of the cross linking agent.

There is no reason to believe that the tracer method could not be applicable to other polymer systems.

MISCELLANEOUS AREAS OF APPLICATION

Effects of Radiation on Propellants

Radiation effects on solid propellants result in the formation of free radicals or ionized particles in the propellant. The presence of these can be readily detected by EPR.

NON DESTRUCTIVE EXAMINATION OF PROPELLANTS

While most of the methods and applications discussed thus far are non-destructive, they are not the types of tests which are normally thought of when the term Non-Destructive Testing is used. From a consideration of the overall picture of magnetic resonance presented here, however, it is easy to conceive of numerous applications of the more prosaic type which might be applied as non-destructive techniques. Let me give a brief discussion of these.

(1) the Effects of a changing viscosity of a propellant material would have an effect on the relaxation time T_2. This would be reflected in a broadening of the NMR signal obtained from this propellant. This broadening of the signal might then be used to determining such things as (a) degree of cure of a propellant composition while it is still in process, (b) the amount of post-cure embrittlement which has occured in a sample kept in storage for a period of time. The possibility that crystallization of a polymer or embrittlement may have occured during storage or (c) the effects of temperature and humidity on the elastic constant of a material. The same sample could be studied over a long period of time to determine such effects as crystallization of a polymer after long time storage at a low temperature. This is an actual problem which has come up recently with one polysulfide unit.

(2) Since the area under the curve is a function of the amount of material under test, it might be possible to monitor a stream of propellant for voids or porosity using an integrating technique to detect the changes in the weight of propellant passing through a section of casing hose at any moment.

(3) The high resolution technique could be used to detect decomposition or side reactions which might occur in a propellant under severe or standard storage conditions. This would be demonstrated by changes in the shape of the spectrum or the quantitative evaluation of functional groups present.

(4) One application recently proposed is the detection of Hydrogen embrittlement in metals subjected to plating operations. The presence of absorbed hydrogen has a morbid embrittleing effect on all types of metals. A spring which has been plated will frequently fail prematurely because of this effect. Although at this point it does not appear feasible to study ferromagnetic materials, application to non ferrous metals may be possible.

If one lets his imagination run wild many more applications will come up. With a superficial understanding of what magnetic resonance is and a knowledge of some of the things it can do all sorts of ideas will present themselves. Leave the details to the experts to work out. Just tell them what you want to do with NMR, EPR or NQR and they will tell you how. And just as important, keep in touch with the latest applications in this rapidly expanding field. Improvements in instrumentation and technique are constantly being made. What was impossible yesterday, may become routine tomorrow.

Let me leave you with this one last thought -- in spite of what I have said here, there are many things you cannot do with Nuclear Magnetic Resonance.

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X-RAY DIFFRACTION

AS A FUNCTIONAL MEASUREMENT TOOL

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The purpose of this paper is to present an old technique and laboratory test method, that of X-Ray Diffraction as it is applied to the production inspection or measurement of spin compensation of copper liners. These liners are a component part of the shaped charge utilized with types of chemical energy, armor defeating projectiles. For this paper, the terminology of cones will be used rather than liners, since it will better describe the manufacturing process by which the cones or liners are made. It will also more adequately describe this inspection procedure.

(ILLUSTRATION #1 - Neg. No. S1330)

The instrumentation used consists of a standard x-ray diffraction unit with a rotating Geiger Mueller detector making one revolution every 12-1/2 minutes.

This inspection procedure was developed by Aberdeen Proving Ground under the technical cognizance of Frankford Arsenal.

This presentation divides into two parts. The first part is the fundamental theory and engineering correlation as related to this application. Only a small amount of the engineering test is described to indicate the completeness of the engineering correlation established. The second part covers the fixture design and its application.

This application is the result of extensive engineering function, destructive and non-destructive tests correlating performance with basic theory.

This inspection method inspects and measures grain orientation relating backward through a series of engineering characteristics to functional performance. The correlating factors are:

Rotational Frequency to Jet Degradation

Jet Degradation to Cone Detonation Loading

Cone Detonation Loading to Planes of Weakness (Crystal Slip Planes (III))

Crystal Slip Plane (111) to Crystal Diagonal Plane (110)

Crystal Diagonal Plane to Grain Orientation

Grain Orientation to X-Ray Diffraction Pattern

This X-Ray Diffraction pattern measured by the goniometer gives the $(I_{\alpha 1} - I_{\alpha 2})$ value or a numerical value which is representative of grain orientation.

This phenominal procedure is a tribute to engineering technology. Thus, after a step-by-step series of correlating engineering factors, sufficient data exists that functional performance of the shear formed cone can be directly measured by the X-Ray Diffraction technique.

In all laboratory experiments, inaccuracies occur. In this case, however, due to instrumentation operation, background radiation, selection of spot for microdensitometer measurements, control of film development and fixing, the remaining data is usable.

In the description of the manufacturing process and seven correlation factors, repetition of the basic material structure is intentional to provide for careful explanation and defining of the process throughout the correlation stages.

(ILLUSTRATION #2 - Neg. No. S1333)

In the manufacture of rotary extruded cones, a lightly cupped disk of electrolytic copper is held firmly against a rotating mandrel and formed into a cone. This manufacturing process known as "Rotary-Extrusion", or "shear forming", is a mechanical modification of the standard metal spinning technique.

After the cupped copper disk is gripped between the live center and the rotating mandrel, a circular friction-driven, carboloy tool moves in against the metal blank and travels down the side of the mandrel, maintaining a preset distance between the mandrel and the tool edge. The pressure exerted by the tool forces the metal to conform to shape of the mandrel at a controlled thickness equal to the required cone wall thickness. After the cone is formed, the excess blank material is trimmed from the cone base by a shearing operation.

The critical manufacturing parameters in this process that will have the greatest effect on end item performance are:

Shape of the tool edge

Velocity of mandrel rotation (speed)

Velocity of tool travel down mandrel (feed)

Direction of mandrel rotation

Hardness of the copper blank

(ILLUSTRATION #3 - Neg. No. S1326)

Temperature of the finished cone

Why such critical control of these process characteristics? A control of the metal anisotropies is necessary in order to manufacture into the cones a pre-established preferred grain orientation or spin compensation. What is meant by anisotropies is the control of different properties of the cones when testing

along different axes. Originally these factors were monitored only by measurement of the twist angle measured from a prescribed line of the copper disk as it was deformed during the manufacturing process. This improved method measures the grain orientation by means of the intensity difference of a refracted x-ray beam.

(ILLUSTRATION #4 - Neg. No. S1510)

The term "spin compensation" should be defined for this discussion. It is the mechanical deformation manufactured into a shear formed cone to compensate for the rotation of the round. It indirectly includes all the correlating characteristics necessary for functional performance and it relates directly to ballistic performance. Spin compensation is found only in conical shaped charge liners manufactured by the rotary extrusion process. What does spin compensation do? It counteracts the degradation of the jet which occurs when the round is experiencing external rotation and thereby improves penetration performance.

During detonation loading of the cone, failure occurs or starts along the planes of weaknesses or slippage planes of the crystals. The control of these slippage planes is the preferred orientation manufactured into the cone. Direct correlation between the preferred orientation of the crystal planes and spin compensation has been established.

(ILLUSTRATION #5 - Neg. No. S1332)

Preferred grain orientation is the controlled result of the metal flow during the rotary extrusion forming process. This alters the orientation of the grains in the copper. When this happens the relative movements of the material near the two cone surfaces are in opposite directions. Plastic flow during the formation of the cone carries the material down the mandrel. The material

between the two surfaces is under the influence of a controllable shearing force which produces the grain or crystal orientation.

(ILLUSTRATION #6 - Neg. No. S1334)

In addition, engineering tests have demonstrated that grain shape, residual stress, etc., do not influence the grain orientation and thus the spin compensation rate. As an example as to why these characteristics do not influence spin compensation, the residual stress which is created during the rotary-extrusion process is almost instantaneously relieved due to the surface temperature 300°F. produced during the process. Actual internal temperature reaches approx. 500°F.

(ILLUSTRATION #7 - Neg. No. S1335)

What determines the preferred crystal orientation necessary for the angular measurement of spin compensation and how is it inspected? Ballistic functional tests determine the range of grain orientation which is acceptable and it is measured by x-ray diffraction. Copper is a face centered cubic material with no low or high temperature allotropic transformation. By the use of X-Ray Diffraction techniques, the type and degree of grain orientation can be determined. This method depends upon precise measurements of the intensities of the diffracted x-ray beam. This in turn is based on the change in the crystal structure under examination.

As is shown, the diffracted ray from one plane at one point in the x-ray beam, maintaining an angle θ will cause one spot on the x-ray film.

(ILLUSTRATION #8 - Neg. No. S1336)

If there is random orientation of the grains in the metal covered by the area of the x-ray beam, a cone of diffracted rays coming from all the possible

orientations of a given plane and making an angle θ with the x-ray beam, will cut the x-ray film in a circle. This diffracted ring will have uniform intensity for random grain orientation, but if the atomic planes tend to be oriented in one direction, the intensity of the diffracted beam will become greater in one part of the ring and less at others. Therefore, the variation of the intensity around the diffracted ring is a suitable measure of departure from randomness in the distribution of the crystal orientation in the metal.

(ILLUSTRATION #9 - Neg. No. S1328)

The difference or control of grain orientation is represented by the thick area or the deep density of $1\sigma_1$ at σ_1 angle. Notice that at the 0 and 180° location, the intensity of the refracted beam due to interference from the internal crystal planes is zero. For random grain orientation the intensity would be zero around the entire circle of rotation.

(ILLUSTRATION #10 - Neg. No. S1331)

When the film is replaced by the Geiger Mueller tube and the same intensity difference recorded, the peaks and difference in intensity are easily recorded and the same data is obtained without the need for film development.

(ILLUSTRATION #11 - Neg. No. S1327)

Normally the atoms in a crystalline substance are arranged in a symmetrical, repeating, three-dimensional pattern and the distance between the planes of the atoms are of the magnitude of x-ray wave lengths. Hence, the crystals act as a three dimensional grating for x-rays in a manner analogous to the diffraction of visible light by a ruled one dimensional grating. Under appropriate conditions the electrons associated with each atom scatter the incident x-ray beam in a coherent manner. Braggs Equation illustrates this relationship.

$n\lambda = 2d \sin\theta$

λ = wave length of the x-ray

d = spacing between the atomic planes

θ = angle of diffraction between the x-ray beam and the atomic plane

n = an integer, generally 1, in this work

thus $\sin\theta = \frac{1\lambda}{2d}$

A control on the angle thus is related to the distance between the atomic planes of the crystal and so controls the orientation of the grains.

(ILLUSTRATION #12 & #13 - Neg. No. S2354 and Neg. No. S2353)

X-Ray Diffraction. How does the x-ray beam differ from quantitative practices in determining the preferred grain orientation of these cones? The ideal plane to work with would be the crystal slip plane (111) or the path of least resistance to loading failures. However, this would require special thin or special backed specimens. Therefore the 110 or 220 plane is used and the complete cone is used as the specimen under examination.

When the complete cone is used, the curved surfaces of these cones causes a broadening of the diffracted x-ray beam. In the development of this technique, the following procedure was used.

1. Collimated radiation created through .0075 inch thickness of cromium was used in the back reflection techniques. (Cr K_a) measurements of intensity and angles were made with a microdensitometer on the 220 plane, hereafter referred to as the 110 plane.

2. The specimen holder holds the entire cone and allows for three circle adjustment, example: rotation of the cone about three axes. The

principal axes provided for longitudinal and lateral rotation of the specimen about mutually perpendicular axes through the point where the x-ray beam hit the cone. These two axes form an orthogonal, triaxial system with the x-ray beam forming the third axis.

3. Cones from different lots were x-rayed at various points on the surface of each cone. The material was then etched from the outside of the cone and then re-x-rayed, etc.

4. Pole figures (plots of the texture pattern or preferred orientation, as it varies around the axis previously described) were plotted for selected points.

5. All variables, such as development of x-ray film, etc., were maintained constant from experiment to experiment.

6. Intensity measurements were made on the film to determine if a numerical correlation between preferred orientation and spin frequency existed.

7. Intensity absorption correction data was determined from x-ray diffraction pictures taken at various angles of incidence between the x-ray beam and the specimen.

X-Ray Diffraction pictures were obtained every 60° around the cone and the patterns obtained were similar in all cases. Cones from other lots were also checked at 60° spacing and again uniformity of pattern was obtained.

From this study, a point one-inch from the base of the cone was chosen as the standard region or circumference to be inspected.

Additional engineering tests and studies were made of the normal incident x-ray patterns at different depth from the outer surface. The following observations were made.

1. The region of strongly preferred orientation changes from one

side of the diffracted ring to the other as the x-ray patterns are taken from the outer to the inner wall of the cone, reaching a point where no further change takes place in the inner portion of the wall.

2. Cones showing considerable spin compensation also have regions of randomly oriented metal in the outer half of the cone wall.

3. Cones showing little spin compensation have no region of random orientation.

Following this work with the beam normal at 90° to the cone surfaces, the cone was rotated in 5° steps about each of two axes through the point where the x-ray beam strikes the cone. One axis in the specimen allows rotation right and left of normal while the other allows rotation above and below normal. Normal is the spot 1" above the base selected as a typical position.

On rotating the cone above and below normal no intensity change was noted. Rotating to the right or left of normal changed the intensity until the distribution between the two maxima become equal.

a. The points where $(I\delta_1 - I\delta_2)$ are greatest occur 55° to 60° apart in all cases.

b. The maximum value of $I\delta_1 - I\delta_2$ occur approximately 30° to the right or left of the angle at which $I\delta_1 = I\delta_2$.

In surveying different cone lots, the angle of equal intensity $I\delta_1 - I\delta_2$ was found and one value either the right or left maxima was checked.

Conclusions:

The spin-compensation frequency of rotary-extruded cones is a function of the concentration of (110) planes oriented at a given angle to the surface.

The metal flow in the cones during the formation process is equal and

opposite in direction at the inside and outside surfaces of the cone. The difference in flow between the two surfaces causes a difference in orientation of the intensity maxima from the inside to outside surface.

Since the ratio of $I\alpha_1 / I\alpha_2$ maximum and the angle at which $I\alpha_1 = I\alpha_2$ vary linearly with spin compensation, it would appear that the more nearly the (110) plane lie to the plane of the surface, and the heavier the concentration of planes at a given angle near the surface, the higher the spin compensation.

X-RAY DIFFRACTION APPARATUS

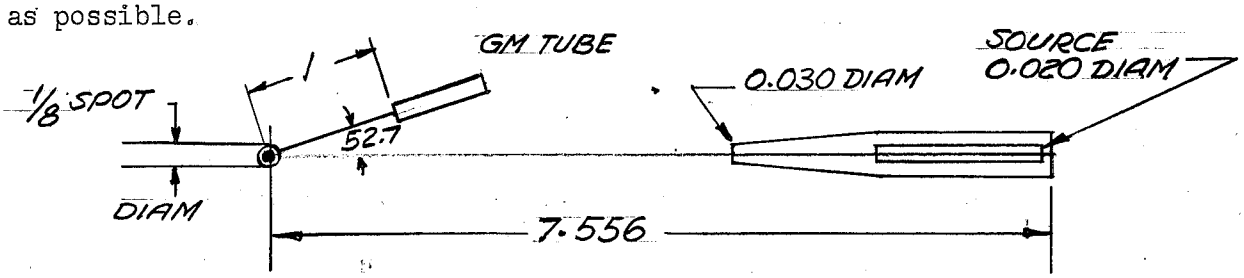
The direct correlation between the preferred orientation of the planes and the spin compensation frequency has been established.

(ILLUSTRATION #14, #15 and #16 - Neg. Nos. S1338, S1339 and S1337)

Any X-Ray Diffraction unit with 50kv, $10M_a$ and having a Cr tube is suitable for this inspection application. Actually this fixture has been used with a Norelco X-Ray Generator Model No. 12045 and an RCA X-Ray Generator Model No. 15385-15440. The detection system consists of Nuclear Chicago thin window GM Model D35 tube and analytical rate meter Model 1620B. The output of the rate-meter is recorded by a Raot-riter rectilinear record. A large variety of detection systems exist which will do an equivalent job and which are equally acceptable.

This fixture has been simplified. During the development of this application, three axes of rotation were employed. By pre-establishment of practical engineering parameters and determination of fixed operating conditions only one axis of rotation is used. This is the rotation of the GM tube at a fixed angle of 52.7° around the axis of the collimated x-ray beam.

The x-ray beam collimator is adjusted for maximum intensity by alignment adjustment screws. The beam collimator is placed as close to the x-ray source as possible.



Originally a vanadium filter was used at source of the x-ray beam to obtain Cr K_a radiation. The filter was removed, however, when it was discovered that the magnitude of beam intensity at the maxima points was more important than the purity of the beam.

After obtaining the maximum intensity of the collimated beam the cone which is mounted in a fixed position normal to the beam is moved to a position one-inch from the plane of rotation of the geiger tube as represented by the location of the collimator for the geiger-mueller tube.

The alignment of the x-ray beam is checked by placing a fluorescent screen Radelin FG#3122 at the surface of the cone. The spot size 1/8 to 3/16 is controlled by the collimator design and its location.

Positioning of the geiger-mueller tube is accomplished by removing it and its collimator and replacing them with a light and a corresponding orifice. The resulting light beam must then intersect on the corresponding spot of the x-ray beam.

Originally the single cycle time of the goniometer was approximately 30 minutes and the rate meter values for a time constant of 20 seconds. The approximate count was 30,000 C/M. The single cycle time has been decreased to 12.5 minutes. The rate meter values were also changed to a time constant of 10

seconds with an approximate count value of 15,000 C/M.

The x-ray diffraction units are standard. They consist of stable, shock-proof, full-wave rectified generator provided with suitable controls and mounted in a substantial metal enclosure bearing an equipment table. Four automatic, gravity-operated, beam-safety shutters are attached to the x-ray tube housing one for each beam port. The tube housing accomodates a series of quickly interchangeable four window diffraction tubes, available with a variety of target materials. In this application a chronimm target is used which supplies K_a 1 2.2896; K_a 2 = 2.2935 and KB 1 = 2.0848 originally a Vanadium filter of .00075 inch thickness was used and then discarded.

The tubes are end grounded, equipped with low-absorption mica windows and are arranged to establish or break both electrical and water connections in the tube housing by simple insertion of the tube.

Cooling water as required by the x-ray head is approximately 2.5/qts./ minute at a pressure of approximately 35 psi. A minimum and maximum pressure regulator provides overload protection for the tube. Minimum pressure cut-off is provided with a 20 second delay, and automatically protects the tube.

(ILLUSTRATION #17 - Neg. No. S1329)

Here again is a production line application in the field of non-destructive testing. Standard methods and existing equipment are used with a specially engineered and designed mounting fixture. As in most non-destructive applications, it is the extensive engineering correlation tests which have made this possible. If it is possible in this summary to philosophize on such successful inspection applications, it is from the viewpoint of the increased reliability which such methods give to both the manufacturer and consumer. It is particularly important to the manufacturing element since it provides methods which were

not in existence for eliminating waste and saving costs.

(Gentlemen) It is the application of such modern inspection methods that keep inspection and quality assurance on a par with the rapidly expanding ocean of technical knowledge of this the 20th Century.

Definitions:

an-i'so-trop-ic	Exhibiting different properties when tested along axes in different directions, as doubly refracting crystals.
bi'fur-cate	To divide into two branches, to fork.
deg"ra-da'tion	or arrest of development.
al"lo'trop'ic	The phenomenon of the existence of an element or compound in two or more different forms.
trans-ver'sal	A transverse line or fiber. Running or laying across.
a-nal'o-gous	Corresponding to something else, bearing some resemblance or proportion to.
cor"re-la'tion	The act or process of correlating, the relation of phenomena as a universal accomplishments whether casually connected or not.
col'li-mat"ed	A device consisting of a tube used for producing a beam of parallel rays.

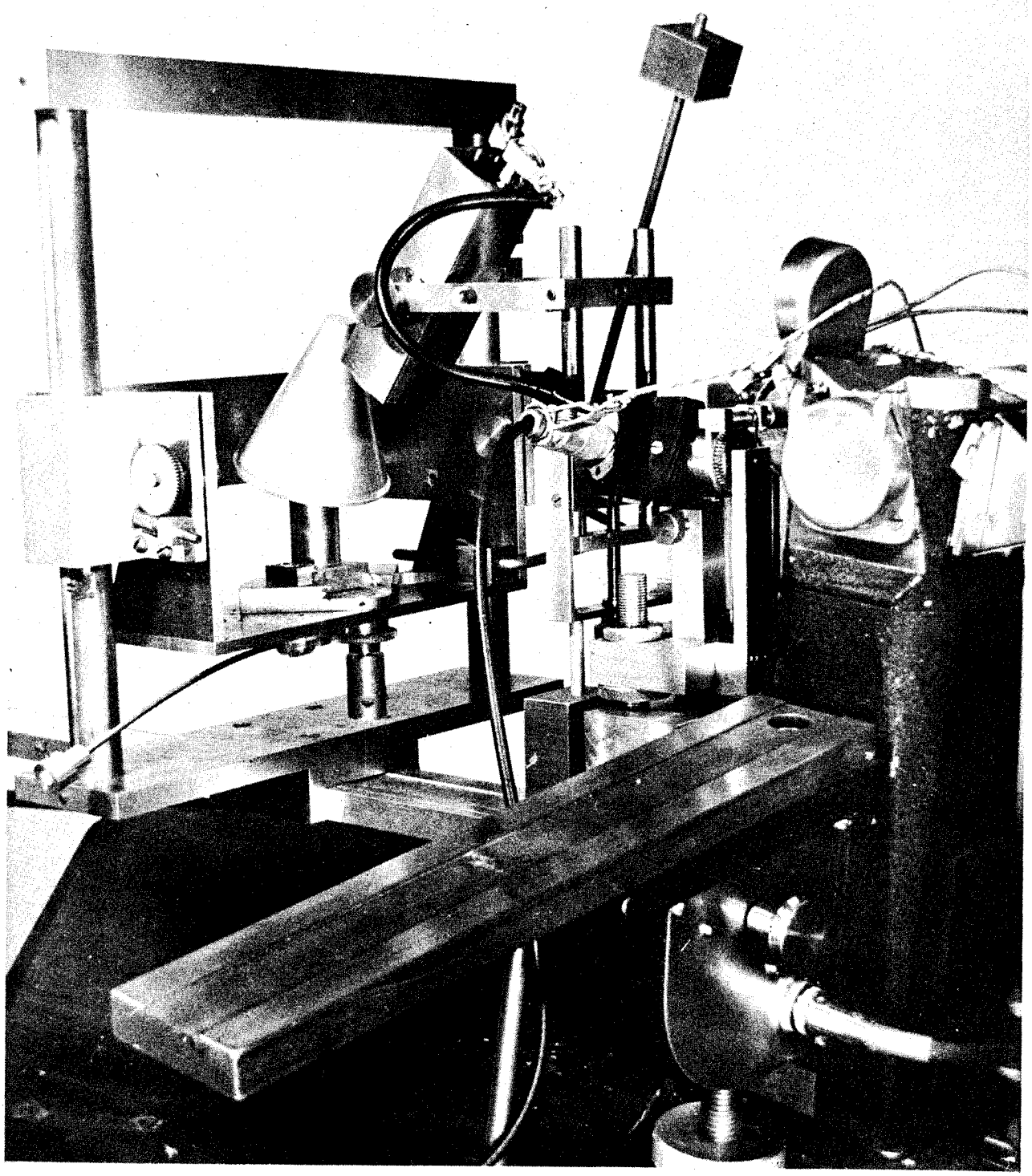
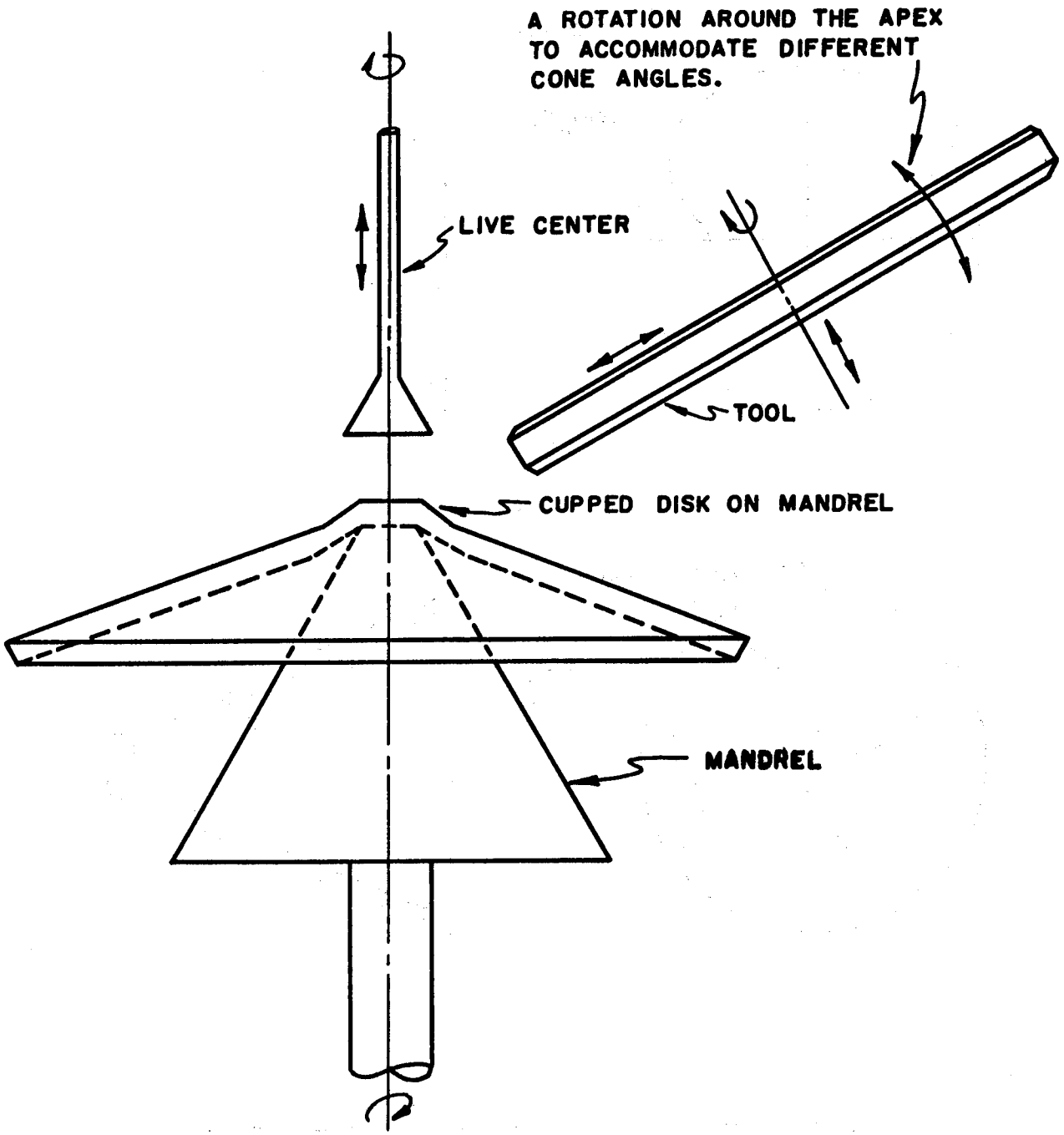


FIG. 1



SCHEMATIC DIAGRAM OF THE ROTARY EXTRUSION PROCESS.

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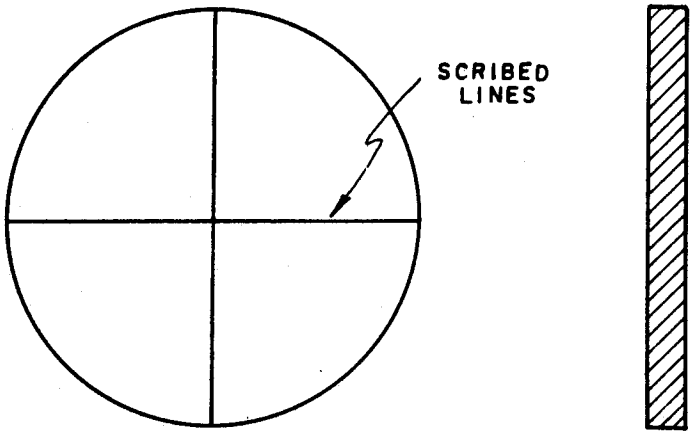
FIG. 2

BLANK



CONE

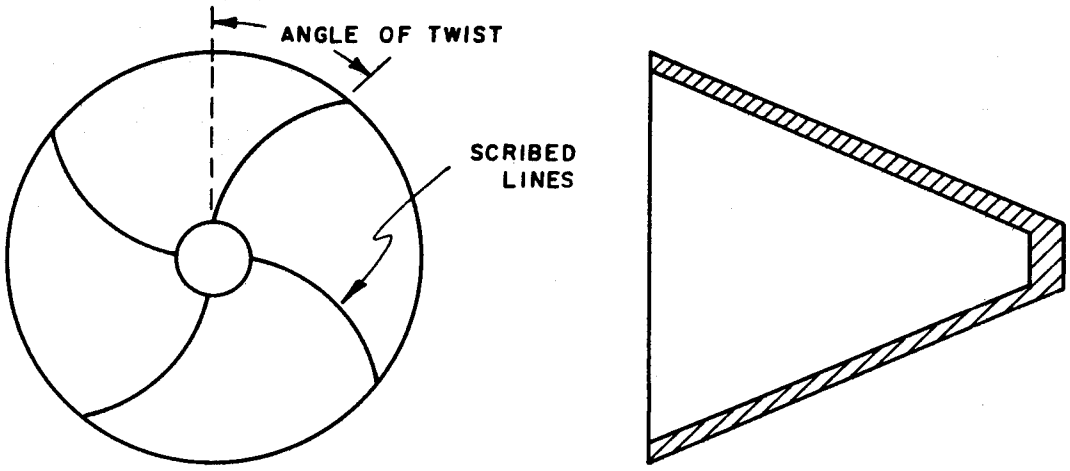
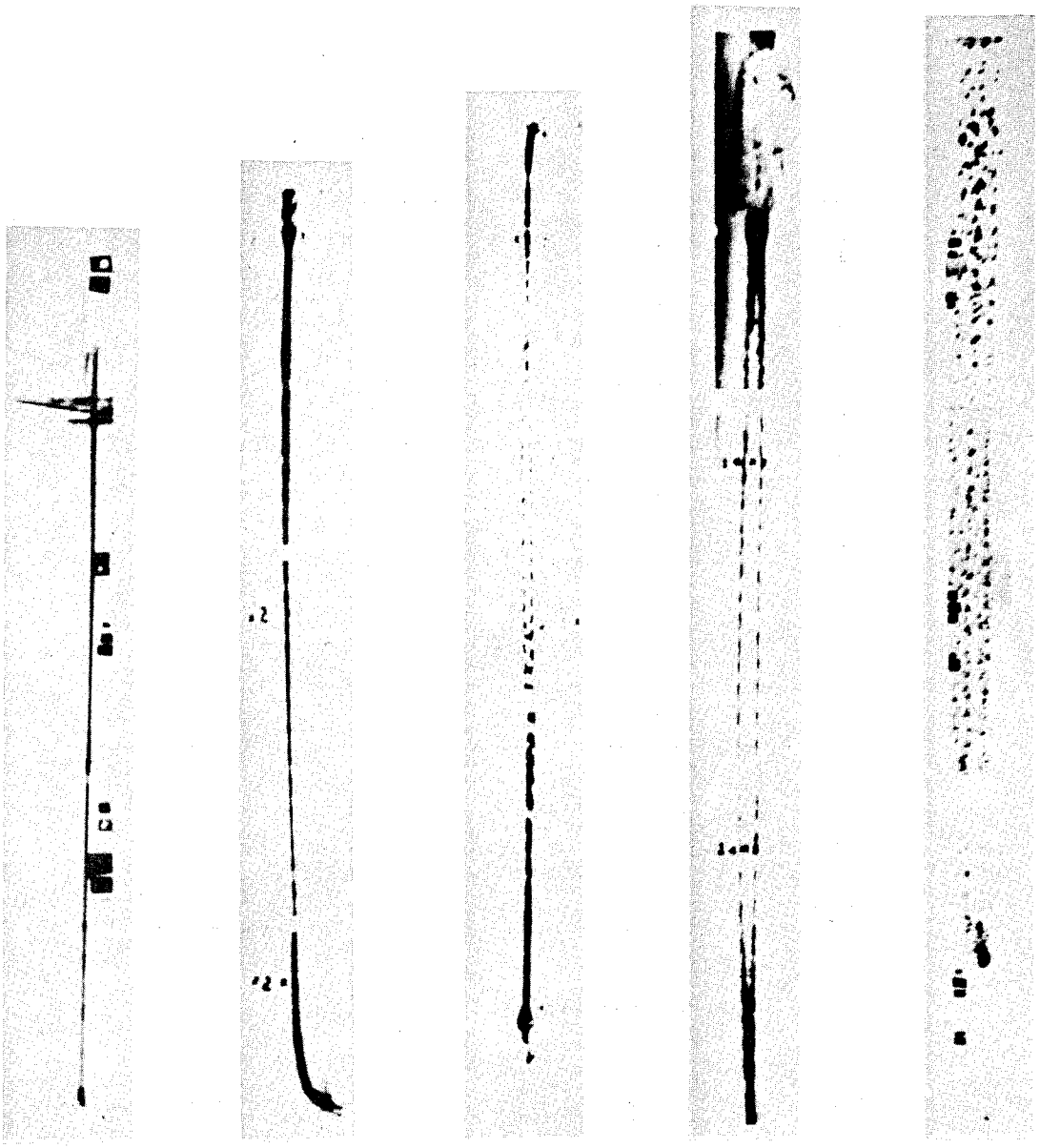


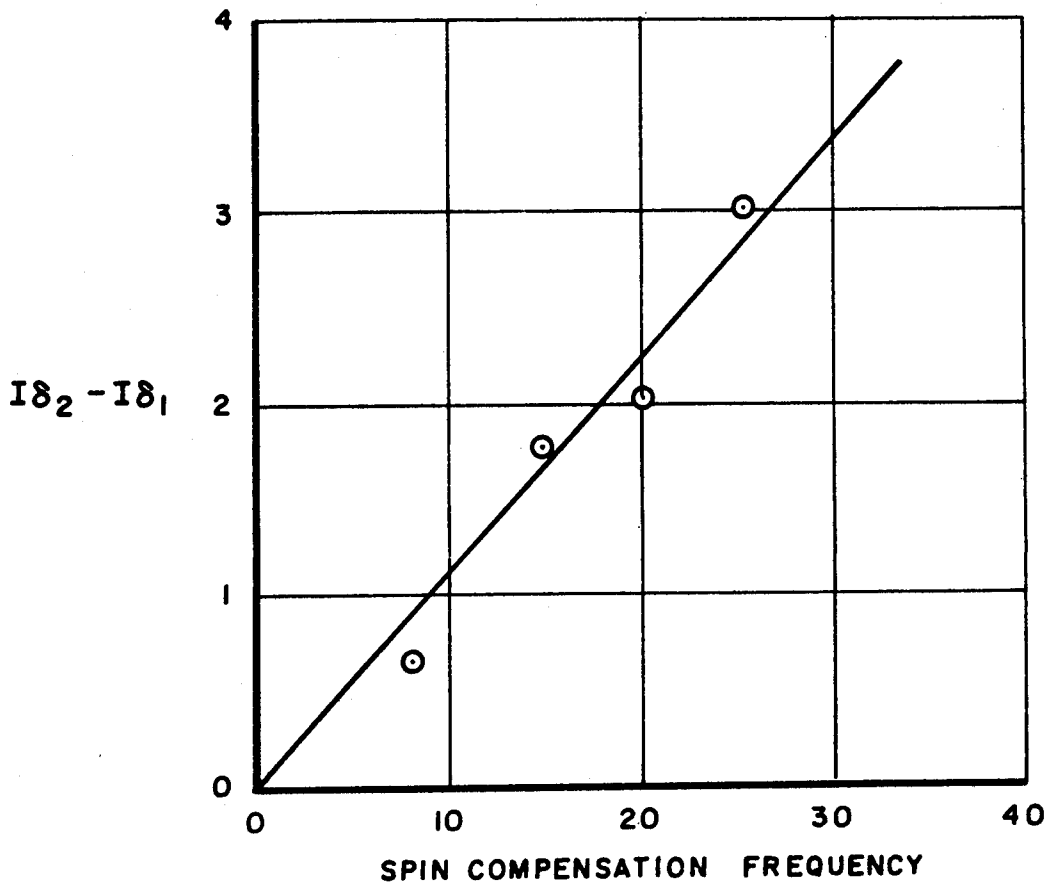
FIG. 3



(a) (b) (c) (d) (e)

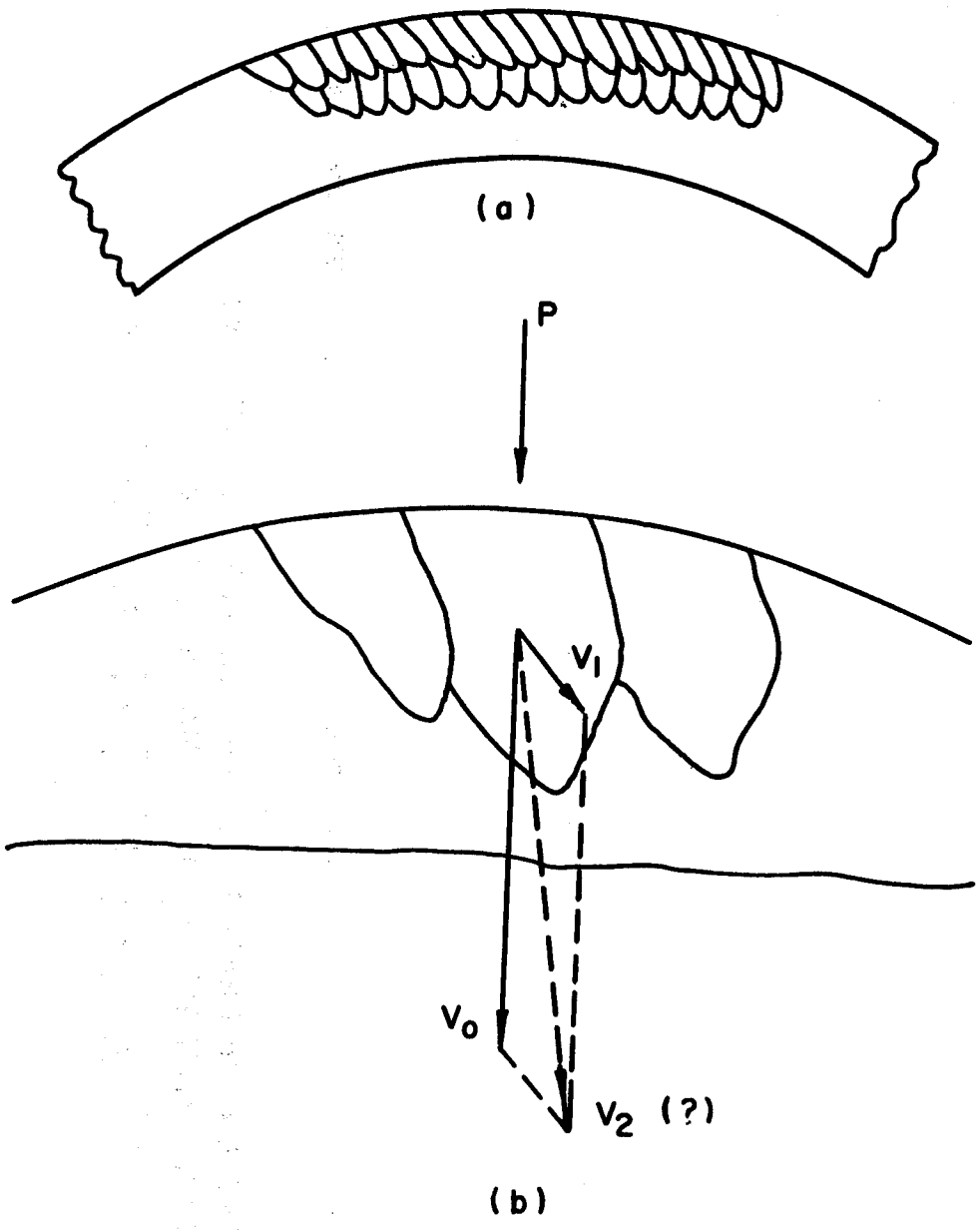
THE JET OF A SMOOTH 105 MM LINER IS SHOWN TO SPREAD WHEN THE SHAPED CHARGE IS ROTATED. ROTATIONAL FREQUENCIES FOR THESE JETS ARE (a) 0 RPS, (b) 15 RPS, (c) 30 RPS, (d) 45 RPS, AND (e) 90.

FIG. 4



$(I\delta_2 - I\delta_1)$ vs. SPIN COMPENSATION FREQUENCY
LINERS X-RAYED AT THE OUTSIDE
SURFACE.

FIG. 5

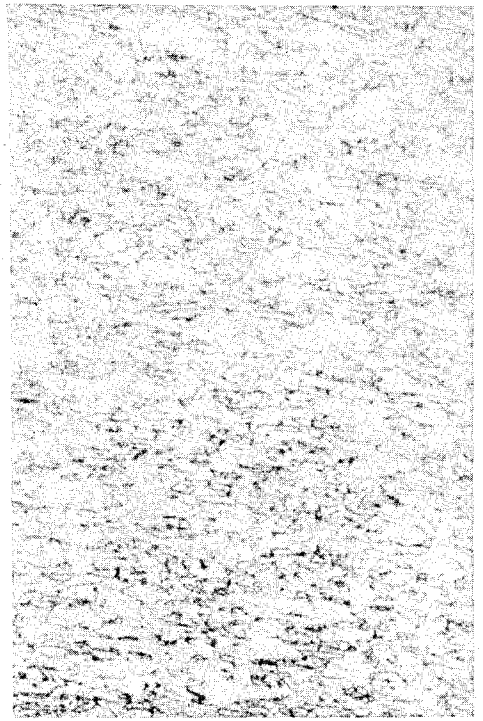


SCHEMATIC OF ORIENTED GRAIN STRUCTURE

FIG. 6

a.—BLANK MATERIAL

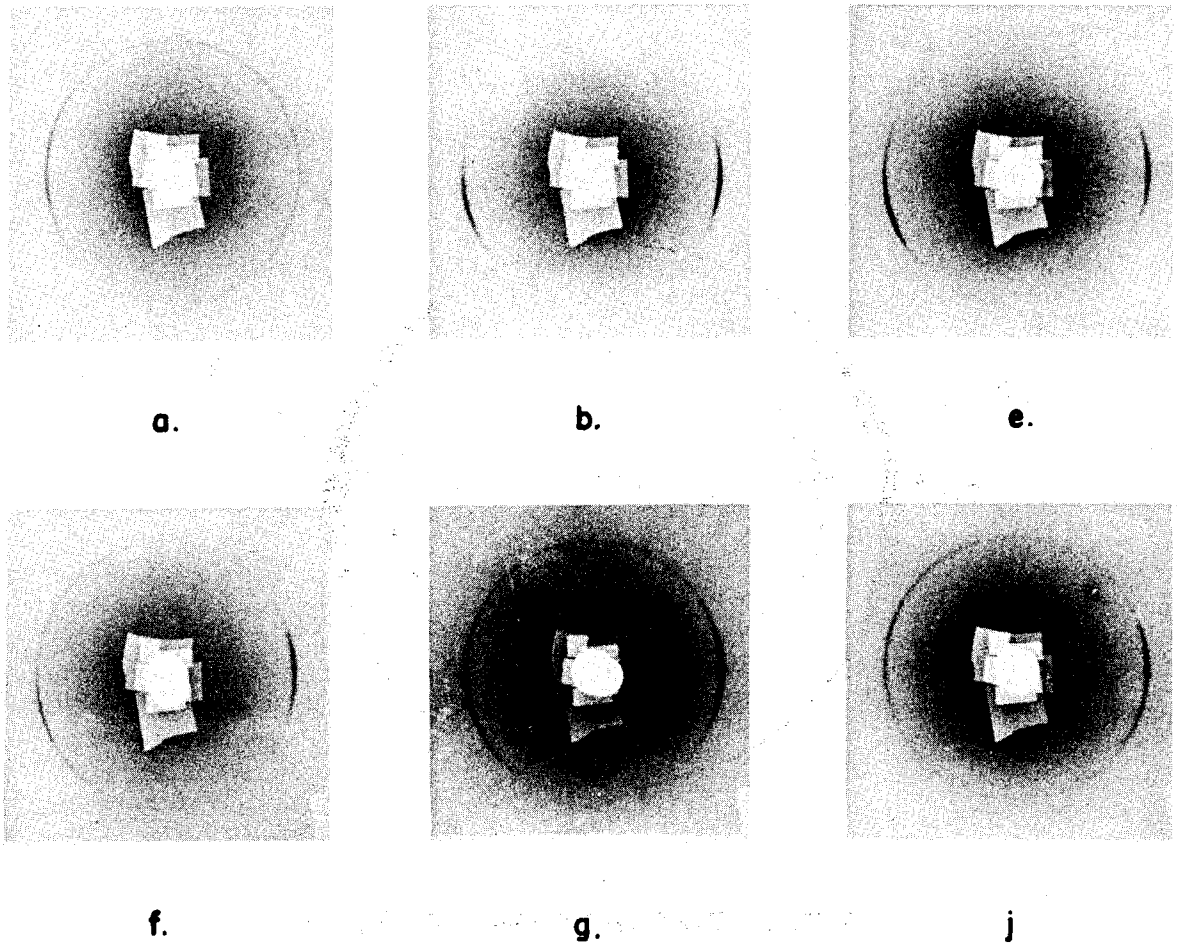
b.-OUTER CONE WALL



c.—INNER CONE WALL

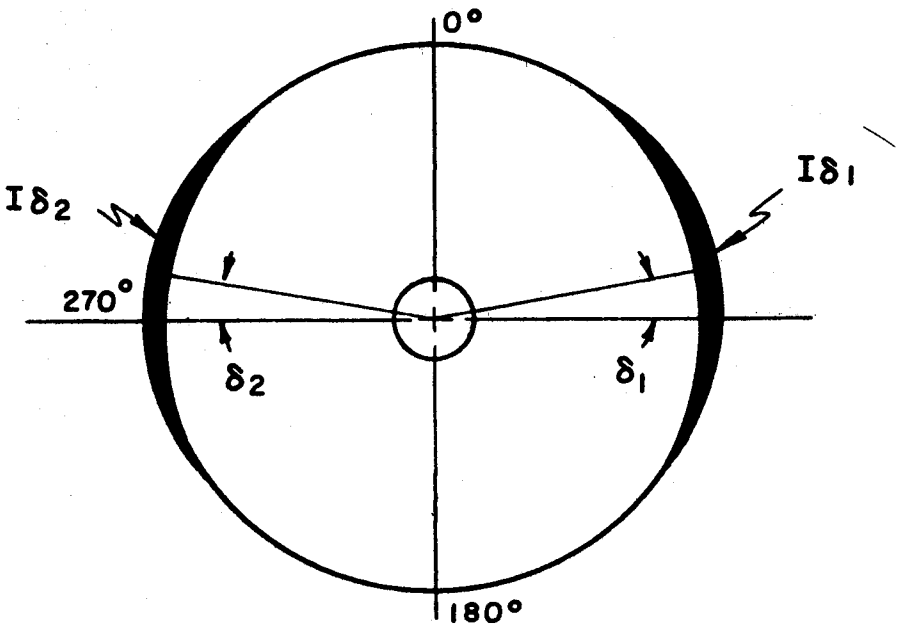
PHOTOMICROGRAPHS OF THE COPPER BLANK AND CONE MATERIAL — 200 X MAGNIFICATION.

FIG. 7



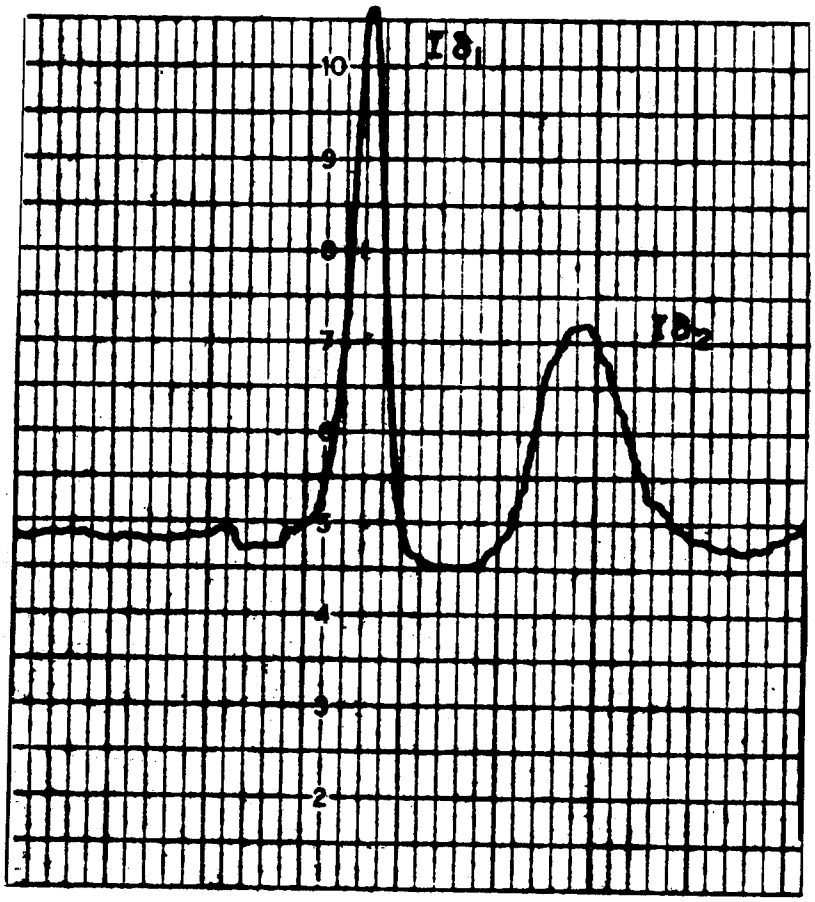
NORMAL INCIDENCE X-RAY DIFFRACTION PATTERNS OF DIFFERENT CONE LOTS.

FIG. 8



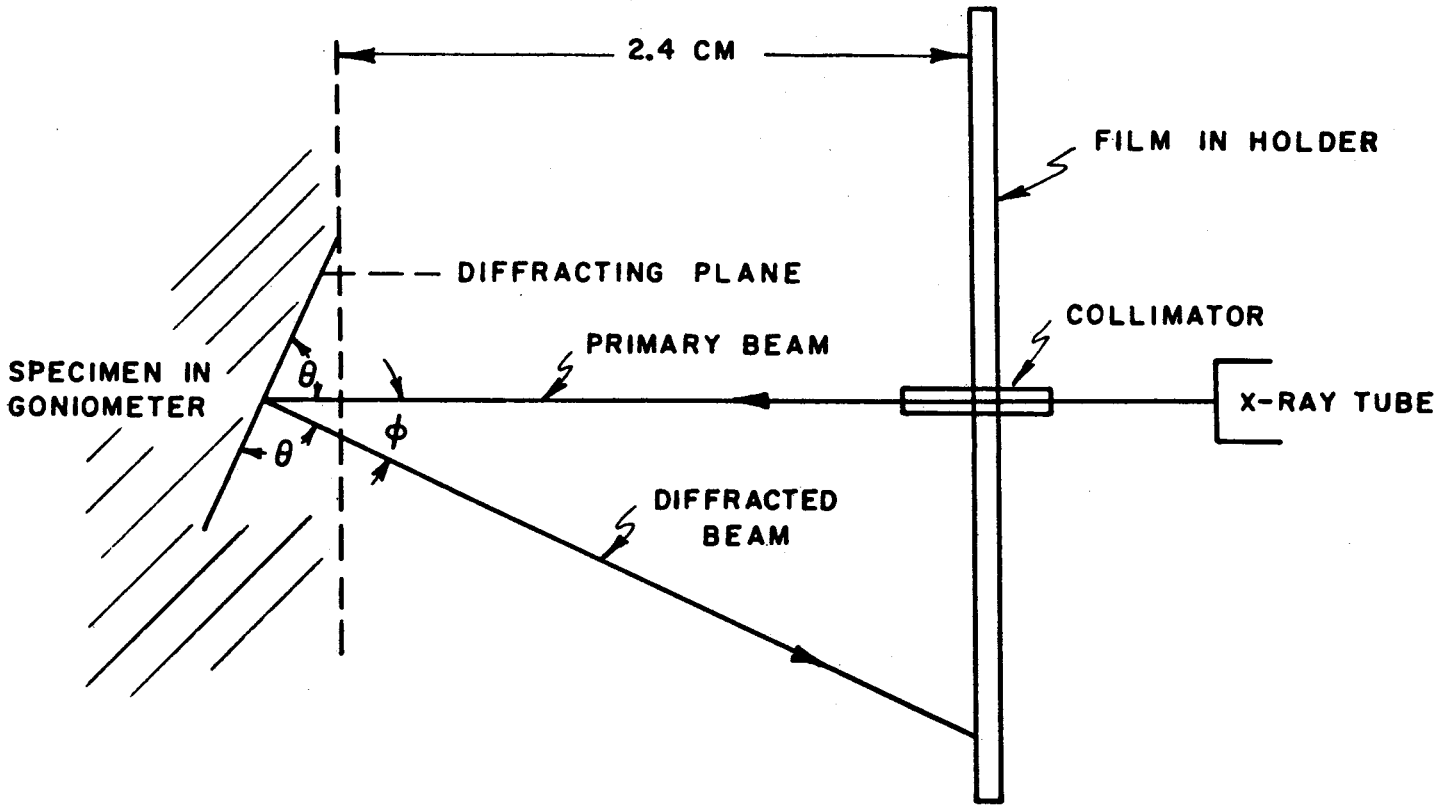
TYPICAL DIFFRACTION PATTERN

FIG. 9



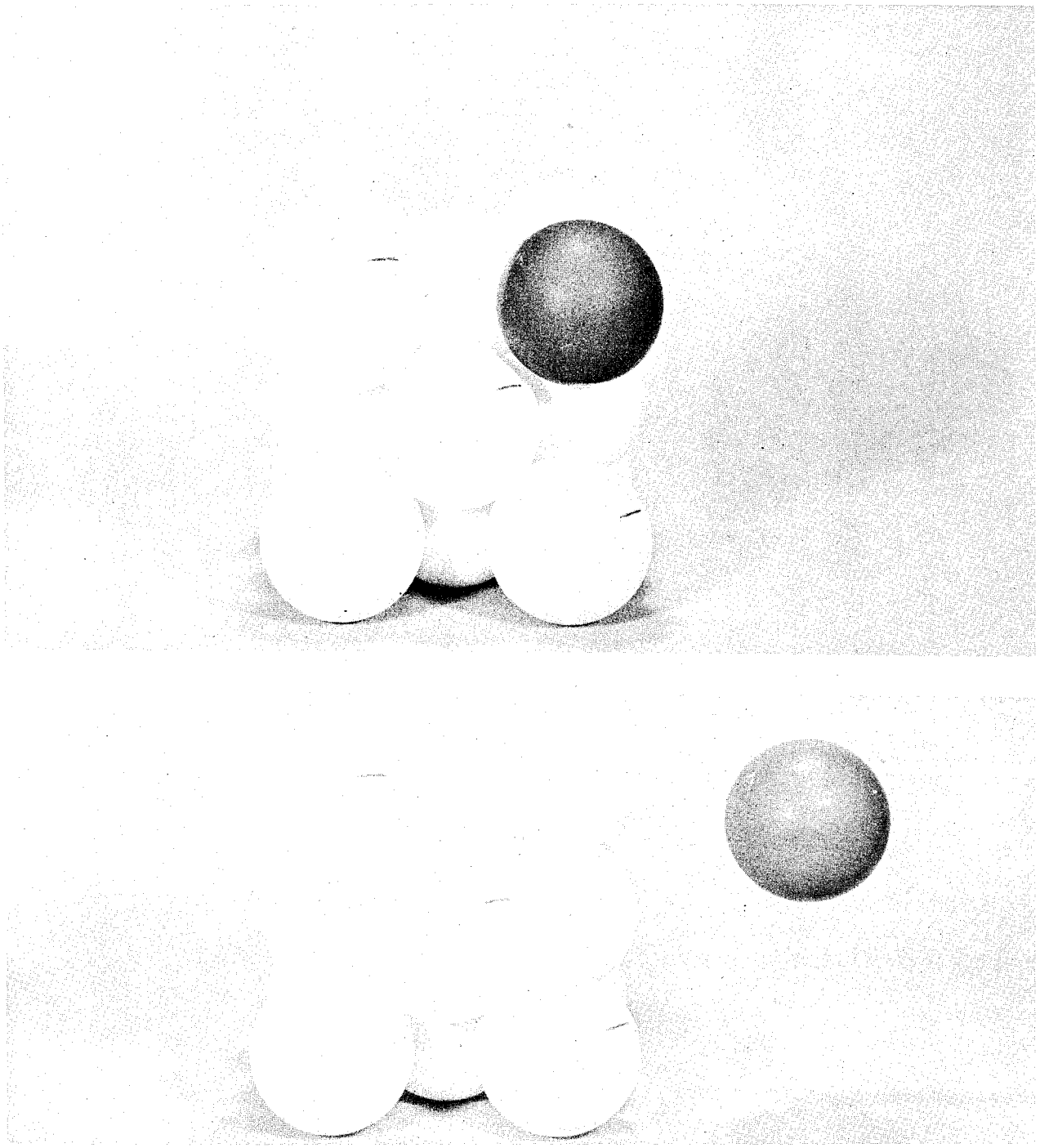
X-RAY DIFFRACTION CURVE FOR
FIRESTONE TYPE M LINER X-RAYED
AT .030" FROM THE OUTSIDE SURFACE

FIG. 10



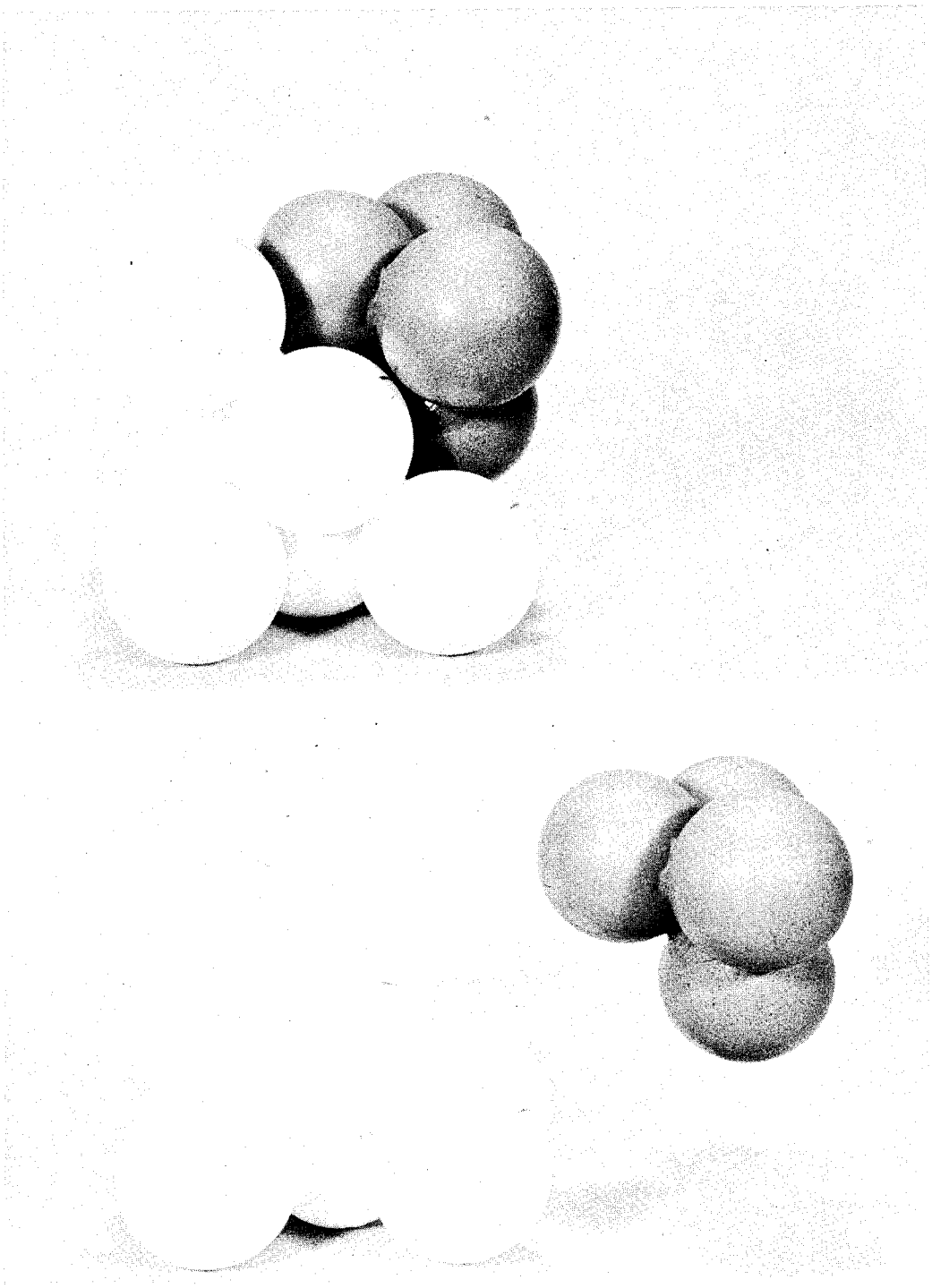
ARRANGEMENT FOR TAKING A BACK REFLECTION X-RAY PATTERN

FIG. 11



Copper Face Centered Cubic Illustrating 1-1-1 Plane

FIG. 12



Copper Face Centered Cubic Illustrating 1-1-0 Plane

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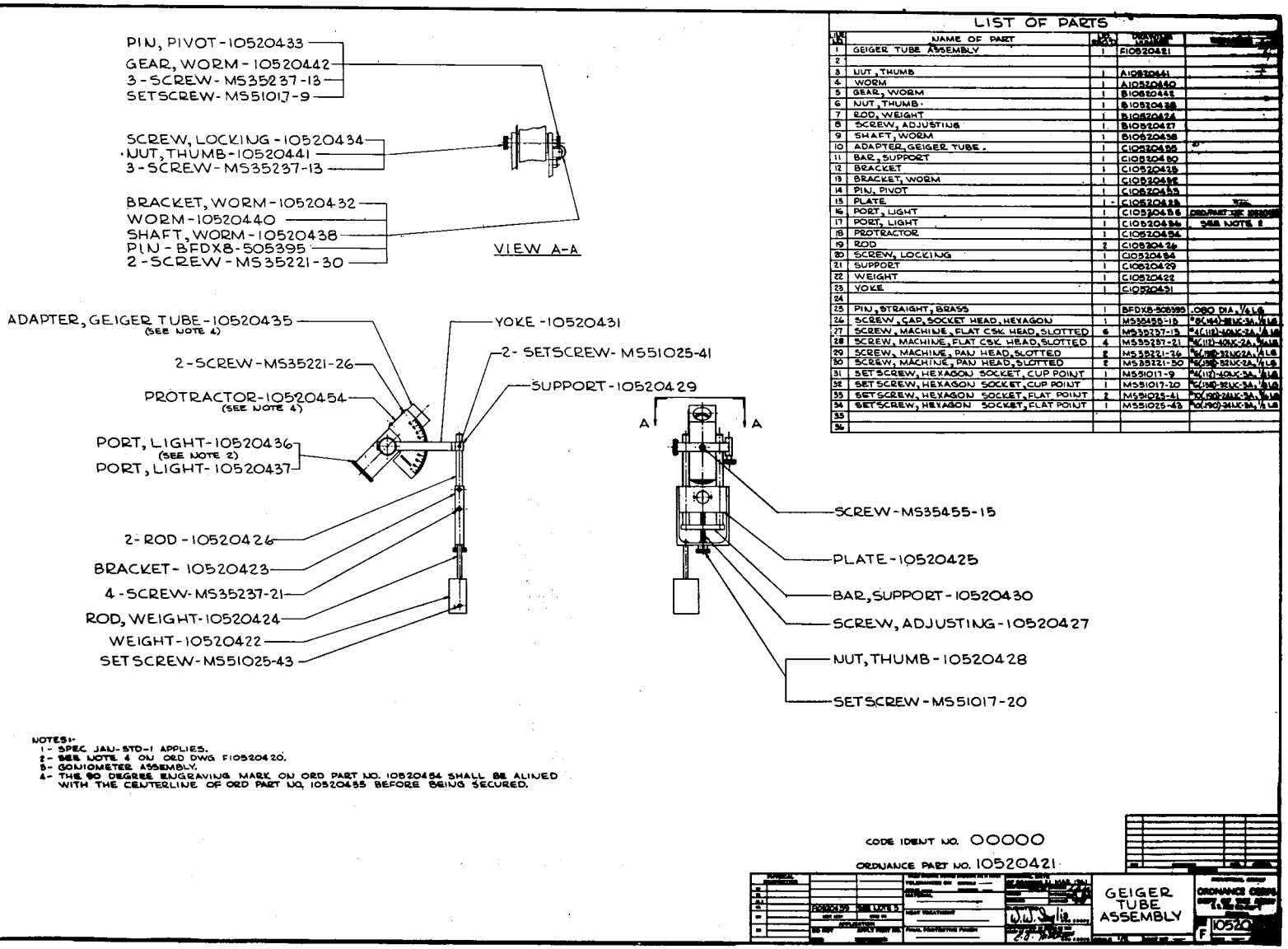
FIG. 13



LIST OF PARTS

LIC NO.	NAME OF PART	QTY REQ'D	ORDNANCE NUMBER	
1	GEIGER TUBE ASSEMBLY	1	F10520421	
2				
3	NUT, THUMB	1	A10520441	
4	WORM	1	A10520440	
5	GEAR, WORM	1	B10520442	
6	NUT, THUMB	1	B10520428	
7	ROD, WEIGHT	1	B10520424	
8	SCREW, ADJUSTING	1	B10520427	
9	SHAFT, WORM	1	B10520438	
10	ADAPTER, GEIGER TUBE	1	C10520435	
11	BAR, SUPPORT	1	C10520430	
12	BRACKET	1	C10520423	
13	BRACKET, WORM	1	C10520432	
14	PIN, PIVOT	1	C10520433	
15	PLATE	1	C10520425	
16	PORT, LIGHT	1	C10520436	
17	PORT, LIGHT	1	C10520437	SEE NOTE 2
18	PROTRACTOR	1	C10520454	
19	ROD	2	C10520426	
20	SCREW, LOCKING	1	C10520434	
21	SUPPORT	1	C10520429	
22	WEIGHT	1	C10520422	
23	YOKE	1	C10520431	
24				
25	PIN, STRAIGHT, BRASS	1	BFDX8-505395	.080 DIA, 1/4 LG
26	SCREW, CAP, SOCKET HEAD, HEXAGON	1	MS35455-15	#8(.164)-32UNC-3A, 1/2 LG
27	SCREW, MACHINE, FLAT CSK HEAD, SLOTTED	6	MS35237-13	#4(.112)-40UNC-2A, 1/4 LG
28	SCREW, MACHINE, FLAT CSK HEAD, SLOTTED	4	MS35237-21	#4(.112)-40UNC-2A, 3/4 LG
29	SCREW, MACHINE, PAN HEAD, SLOTTED	2	MS35221-26	#6(.138)-32UNC-2A, 1/4 LG
30	SCREW, MACHINE, PAN HEAD, SLOTTED	2	MS35221-30	#6(.138)-32UNC-2A, 3/4 LG
31	SETSCREW, HEXAGON SOCKET, CUP POINT	1	MS51017-9	#4(.112)-40UNC-3A, 1/8 LG
32	SETSCREW, HEXAGON SOCKET, CUP POINT	1	MS51017-20	#6(.138)-32UNC-3A, 1/4 LG
33	SETSCREW, HEXAGON SOCKET, FLAT POINT	2	MS51025-41	#10(.190)-24UNC-3A, 3/16 LG
34	SETSCREW, HEXAGON SOCKET, FLAT POINT	1	MS51025-43	#10(.190)-24UNC-3A, 1/2 LG
35				
36				

NOTES:
1 - SPEC JAN-STD-1 APPLIES.
2 - SEE NOTE 4 ON ORD DWG F10520420.
3 - GONIOMETER ASSEMBLY.
4 - THE 90 DEGREE ENGRAVING MARK ON ORD PART NO. 10520454 SHALL BE ALINED WITH THE CENTERLINE OF ORD PART NO. 10520455 BEFORE BEING SECURED.

CODE IDENT NO. 00000

ORDNANCE PART NO. 10520421

GEIGER TUBE ASSEMBLY

ORDNANCE CORPS

10520

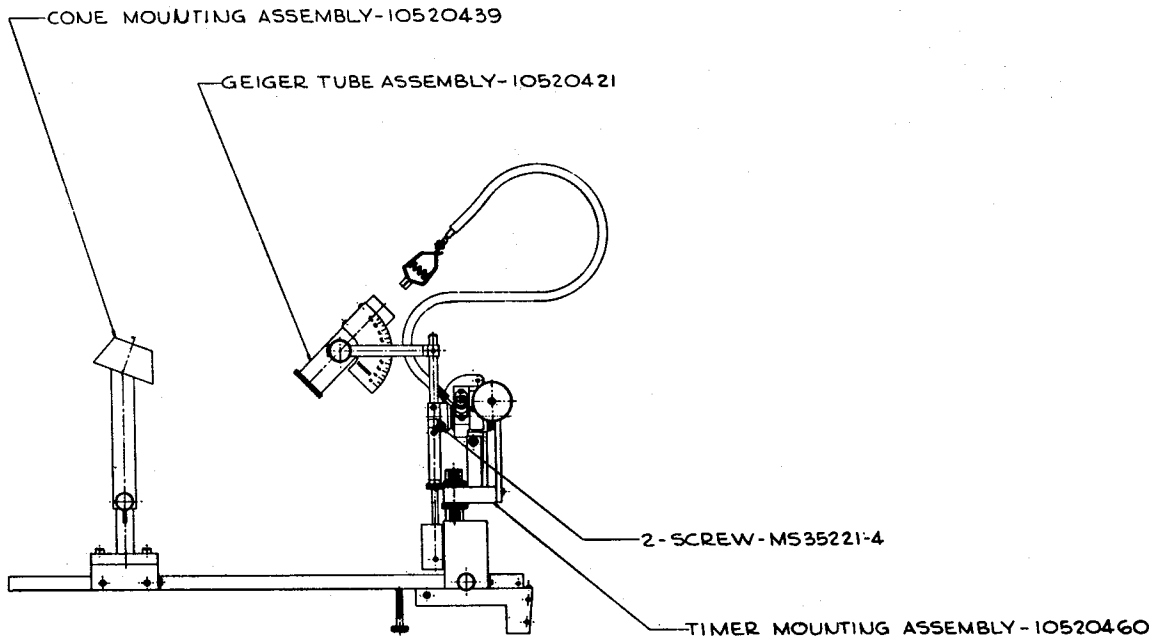
F

36-231S 1339-ORD-61

FIG. 14

LIST OF PARTS

ITEM NO.	NAME OF PART	QTY REQ'D	DRAWING NUMBER	REMARKS
1	GONIOMETER ASSEMBLY	1	F10520459	
2				
3	CONE MOUNTING ASSEMBLY	1	F10520439	
4	GEIGER TUBE ASSEMBLY	1	F10520421	
5	TIMER MOUNTING ASSEMBLY	1	F10520460	
6				
7	SCREW, MACHINE, PAN HEAD, SLOTTED	2	MS35221-4	#2(.086)-56UNC-2A, ¼ LG
8				



NOTES:-
1- SPEC. JAN-STD-1 APPLIES.
2- X-RAY DIFFRACTION SYSTEM ASSEMBLY.

CODE IDENT NO. 00000
ORDNANCE PART NO. 10520459

				GONIOMETER ASSEMBLY	ORDNANCE CORPS DEPT. OF THE ARMY
					F 10520459

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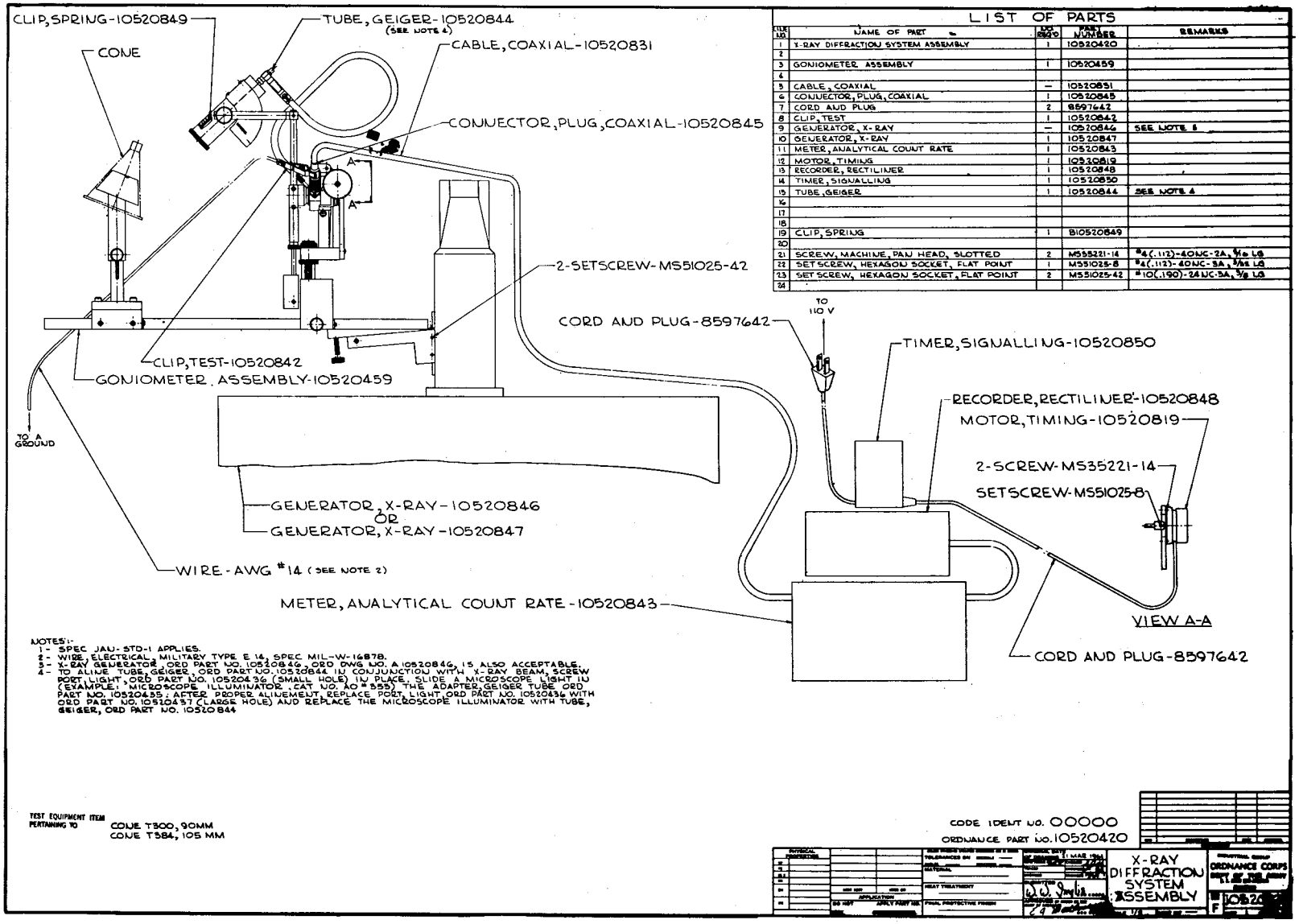
FIG. 15



LIST OF PARTS

ITEM NO.	NAME OF PART	NO. REQD	PART NUMBER	REMARKS
1	X-RAY DIFFRACTION SYSTEM ASSEMBLY	1	10520420	
2				
3	GONIOMETER ASSEMBLY	1	10520459	
4				
5	CABLE, COAXIAL	—	10520831	
6	CONNECTOR, PLUG, COAXIAL	1	10520845	
7	CORD AND PLUG	2	8597642	
8	CLIP, TEST	1	10520842	
9	GENERATOR, X-RAY	—	10520846	SEE NOTE 5
10	GENERATOR, X-RAY	1	10520847	
11	METER, ANALYTICAL COUNT RATE	1	10520843	
12	MOTOR, TIMING	1	10520819	
13	RECORDER, RECTILINER	1	10520848	
14	TIMER, SIGNALLING	1	10520850	
15	TUBE, GEIGER	1	10520844	SEE NOTE 4
16				
17				
18				
19	CLIP, SPRING	1	B10520849	
20				
21	SCREW, MACHINE, PAN HEAD, SLOTTED	2	MS35221-14	#4(.112)-40UNC-2A, 5/16 LG
22	SETSCREW, HEXAGON SOCKET, FLAT POINT	1	MS51025-8	#4(.112)-40NC-3A, 1/8 LG
23	SETSCREW, HEXAGON SOCKET, FLAT POINT	2	MS51025-42	#10(.190)-24UNC-3A, 3/8 LG
24				

NOTES:-
1- SPEC JAU-STD-1 APPLIES.
2- WIRE, ELECTRICAL, MILITARY TYPE E 14, SPEC MIL-W-16878.
3- X-RAY GENERATOR, ORD PART NO. 10520846, ORD DWG NO. A10520846, IS ALSO ACCEPTABLE.
4- TO ALIGN TUBE, GEIGER, ORD PART NO. 10520844 IN CONJUNCTION WITH X-RAY BEAM, SCREW PORT LIGHT, ORD PART NO. 10520436 (SMALL HOLE) IN PLACE SLIDE A MICROSCOPE LIGHT IN (EXAMPLE: MICROSCOPE ILLUMINATOR CAT NO. AO #555) THE ADAPTER, GEIGER TUBE, ORD PART NO. 10520435; AFTER PROPER ALIGNMENT REPLACE PORT LIGHT, ORD PART NO. 10520436 WITH ORD PART NO. 10520437 (LARGE HOLE) AND REPLACE THE MICROSCOPE ILLUMINATOR WITH TUBE, GEIGER, ORD PART NO. 10520844

TEST EQUIPMENT ITEM PERTAINING TO: CONE T300, 90MM; CONE T384, 105 MM

CODE IDENT NO. 00000
ORDNANCE PART NO. 10520420

X-RAY DIFFRACTION SYSTEM ASSEMBLY

ORDNANCE CORPS

26·231·S1337·ORD·61

FIG. 16

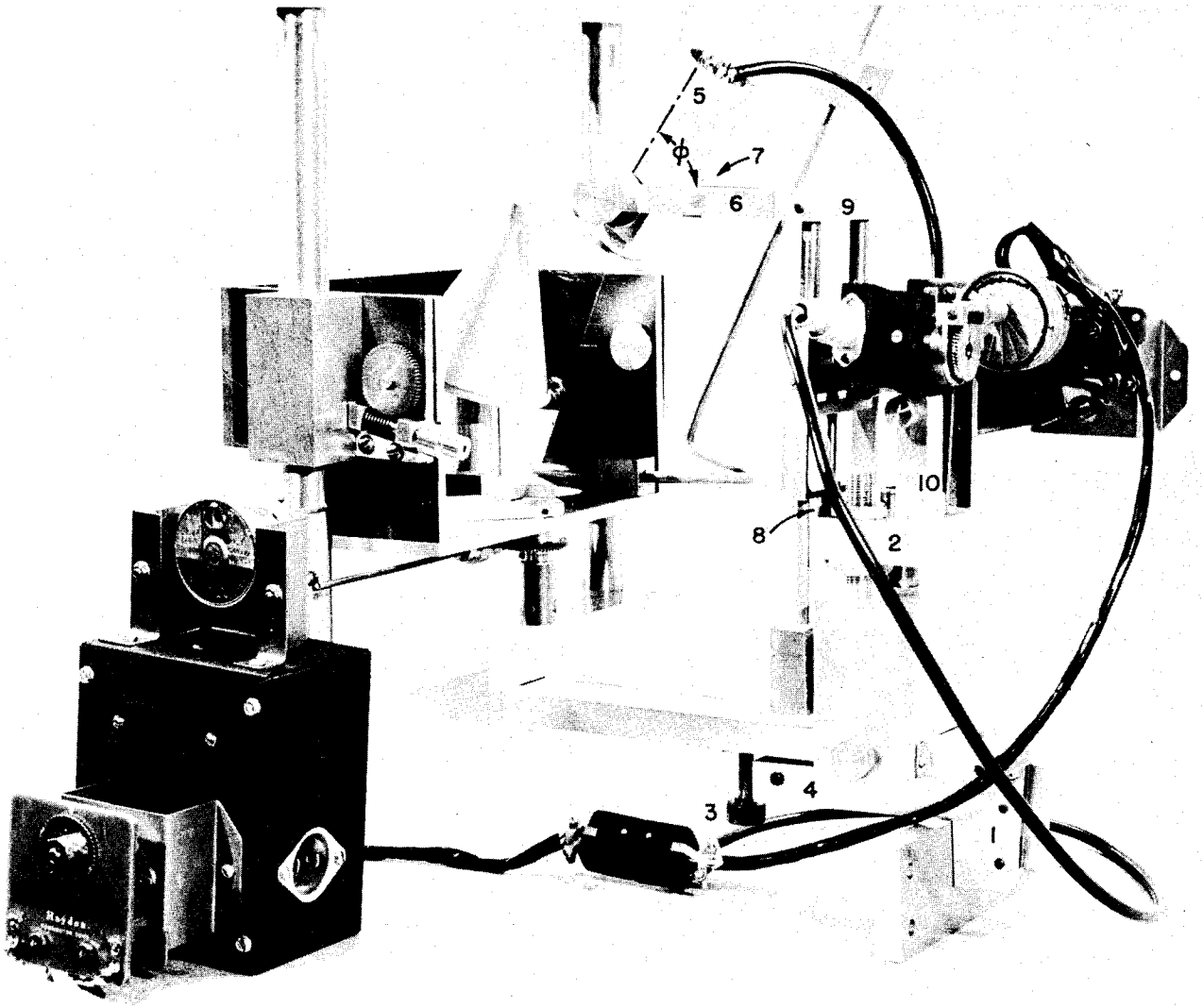


FIG. 17

SUMMARY OF FEEDBACK DATA

J. A. HOLLOWAY
U. S. Naval Ordnance Laboratory

A questionnaire had been sent to each presenter of formal and informal problems at the Ninth, Tenth, and Eleventh Defense Conference on Nondestructive Testing in July 1961. The purpose of this questionnaire was to determine the status of the problem solution at the present time. Following is a summary of the replies. A complete history of the problem is in the minutes of the particular conference.

ELEVENTH CONFERENCE

1. (Formal) - NDT of the Aviation Medical Acceleration Laboratory Human Centrifuge, by R. L. Snyder, AMAL, U. S. Naval Air Development Center, Johnsville, Penna.

Mr. Snyder reports that stress monitoring of the welded joints on the present centrifuge is still a problem. This problem will be transferred to the new centrifuge when it is installed in January 1962. Mr. Snyder is looking for a stress analysis program that can be incorporated into the plans and design of the new centrifuge.

2. (Formal) - Inspection for Soundness of Wall in Rocket Motor Tubes, by Richard Istot, U. S. Army Chemical Corps Materiel Command, Army Chemical Center, Md.

This problem still exists. Testing of motor tubes has been limited to a hydrostatic test in accordance with the applicable specifications. Other methods will be investigated during FY 1962.

3. (Formal) - Bond Inspection in Solid Propellant Rocket Motors, by J. A. Holloway, U. S. Naval Ordnance Laboratory, White Oak, Md.

The problem of detecting separations between the propellant and the liner of rocket motors has been solved to a limited degree. The method still employs tangential radiography but a density ratio technique is used to interpret the film. A description of the density ratio technique is contained in an unclassified Aerojet-General Report No. SRP-202 (Special), entitled "Theoretical Density Ratios as a Standard for Interpretation of Co60-1000 curie Gammagraphs" by R. J. Mascis, Development Engineer, Reliability Department and issued March 1960.

4. (Formal) - Nondestructive Test for Corrosion Determination in Inaccessible Areas, by Capt. John E. Metzger, WRAMA, Robins AFB, Georgia.

This problem is being actively pursued but a solution has not been found. Capt. Metzger indicates that some corrosion problems have become so severe that early detection of corrosion has changed their concepts of the term "excessive costs."

5. (Informal) - Inspection of Shaped Charge Cones, Ordnance Corps, Picatinny Arsenal, Dover, N. J.

This problem has been solved. A description of the method of solution was presented before the 12th Annual Conference by Mr. Warren W. Inglis, Frankford Arsenal, in a paper entitled "X-ray Diffraction as a Functional Tool".

6. (Informal) - Brazed Joint Soundness Evaluation, Indianapolis Air Procurement District, USAF, Indianapolis, Indiana.

This problem is being actively pursued but no solution has been found. Either ultrasonics or eddy currents offer the greatest promise of possible solution.

7. (Informal) - Inspection of Critical Structural Parts Without Removal, by Capt. John E. Metzger, WRAMA, Robins AFB, Georgia

This problem is being actively pursued at the present time. Capt. Metzger, indicates WRAMA has embarked on a rather extensive program of creating radiographic inspection manuals for most aircraft but no method has been found to be more than partially satisfactory.

8. (Informal) - Improvement of Aluminum Nuts and Screws that are presently in service, HQ. San Antonio Air Materiel Area, SAMQCM, Kelly AFB, Texas

Zytel 103 seals are being used between the tube flare and the aluminum "B" nut. This solution to the problem is undergoing service tests on the F-102 chase plane.

9. (Informal) - Detecting Lack of Bond in Adhesive Bonded Materials, Hq. AF Flight Test Center, Air R&D Command, FTFSP, Edwards Air Force Base, California.

This problem has been solved using especially designed ultrasonic resonance type equipment. Due to the estimated high cost of the proposed test equipment and the small number of items to be inspected, it was deemed uneconomical to procure such limited use equipment.

10. (Informal) - Detection of Incipient Cracks in Cylinder Head of Reciprocating Engine, by Mr. F. G. Schrader, O&R Dept. Code 341, Naval Air Station, Alameda, California

The solution to this problem consists of caustic etching of the cylinder head followed by low power magnification examination. The part is neutralized by nitric acid following the etching. The caustic blackens the surface of the aluminum alloys and the nitric acid removes the discoloration. A crack is indicated by a black line where the caustic seeps from the discontinuity.

11. (Informal) - Fillet-type Welding of Aluminum Tubing, Hq. Flight Test Center, Air R&D Command, FTFSP, Edwards AFB, California

X-ray is being used to inspect the welds. The root area of the weld is then viewed for voids with a high intensity viewer coupled with a pocket comparator having a calibrated eye piece. Lack of military specifications applicable to voids in aluminum tube welds continues to be a problem.

12. (Informal) - Standardization of Penetrameters for Radiographic Inspection of Solid Propellant, by Wallce K. Thomas, Army Guided Missile Agency, ORDXR-IQIP Redstone Arsenal, Huntsville, Alabama.

A solution to this problem is still being sought. It is proposed that MIL-STD-286, entitled "Propellant, Solid: Sampling, Examination and Testing," be expanded to include methods for nondestructive testing. A method of fabrication and requirements for the use of penetrameters may be included.

TENTH CONFERENCE

1. Detection of Water in Honeycomb Panels, by Lt. Finkelstein, Oklahoma Air Material Area, Tinker AFB, Oklahoma City, Okla.

The problem of inspection of honeycomb panels for water has been solved using a 400-1200 cps sonic generator called the Coindoscope. X-ray with paper film is also used with reduction of inspection costs. The details presented to the 11th Conference by Lt. George Mayer of OCAMA indicated a savings of $250,000 per year in salvage of good panels and lower inspection costs.

2. Fluorescent Penetrant Operational Problems, by F. G. Schrader, O&R Dept. Code 341, Naval Air Station, Alameda California.

Mr. Schrader states that one of the penetrate problems involving excessive seepage of the penetrant from close-fitting surfaces has been solved by using a lower viscosity emulsifier. This emulsifier penetrates faster without reducing the effectiveness of the method, and conforms to Specs MIL-I-25135, Type 1, Class 2, Control Standard MB-3.

Another associated problem of applying the dust developer effectively has been solved by local manufacture of a cabinet. A ten-minute cycle is employed. The powder is blown in the cabinet for 1 minute; 4 minutes are allowed for settling of the powders, and the dust is then exhausted from the cabinet for 5 minutes. The parts are then removed from the cabinet and inspected under black light.

3. Nondestructive Testing of Aircraft Control Cables, by Lt. George Mayer, OCAMA, Tinker Air Force Base

This problem has been solved using eddy current but the details are not available.

4. Reliable Screening of Mixed Soft and Hard Cylinders, by A. H. Getzel, Frankford Arsenal, Philadelphia, Penna.

Screening of these cylinders has been accompanied by coating annealed cylinders with a black chemical finish. The finish meets MIL-SPEC F-495, "Finish, Chemical, black for Copper Alloys".

NINTH CONFERENCE

1. Munitions and Containers Filled with Lethal Chemical and Biological Agents, Mr. Carl A. Martin, U. S. Army Chemical Corps Engineering Command, Army Chemical Center, Maryland

Leak testing of munitions and containers filled with lethal chemical and biological agents has been solved with an automatic helium leak detector system. The munition is evacuated, filled to proper percent void, back filled with helium to atmospheric pressure and sealed. This technique can be applied to testing container closures of any size providing it is practical to integrate the filling, closing and testing operations. Design of the associated high vacuum system is critical and precise programming is required for automatic operation. Filling, closing, and testing cycles varying from 30 seconds to 2 minutes have been used.

REPORT ON NONDESTRUCTIVE TESTING ACTIVITIES AT RESEARCH AND DEVELOPMENT GROUP, FRANKFORD ARSENAL

B. HOFFMAN
Frankford Arsenal
Philadelphia, Pennsylvania

Nondestructive test research and development activities at the Frankford Arsenal during the past year have resulted in the development of new equipments, techniques and applications employing electro-magnetic and ultrasonic principles. The following summary is representative of the most important of these endeavors:

I- Investigation of Hall Effect Devices for Flaw Detection in Ferrous Materials

Under Project OMS 5010-11-80600-51-01 authorized by the Ordnance Materials Research Office, evaluation studies of Hall Effect semiconductor devices were conducted to determine their merits as detectors of surface flaws in ferrous material. During the first year's effort, major emphasis was placed on the evaluation of flaw detection capabilities and probe design criteria required for the detection and measurement of magnetic leakage fields. Experimental work was also conducted to determine the effectiveness of DC, AC and pulsed type control currents. Although all three control currents used were capable of detecting surface flaws, it was found that the pulse power control current provided a significant increase in sensitivity over the other techniques.

Results of tests to date indicate that Hall Effect semiconductor devices are excellent surface flaw detectors when applied to ferro-magnetic materials doing away with the requirement of using magnetic particles to outline a flaw. Because the Hall generator is electrical in nature it can easily be applied to automatic inspection equipment.

Next year's effort will be devoted to the determination of flaw depth in steel. The possibility of correlating the tangential or vertical components of the leakage field with the geometry of a crack will also be investigated. A final research report R-1581 dated February 1961 was published describing all work done under this program.

II- Ultrasonic Tire Test System

Under Project AOS 2210.4500.061.01 authorized by the Army Ordnance Corps Tank Automotive Command, a prototype ultrasonic tire test system was designed and developed which automatically scans a tire tread area for the presence of flaws while simultaneously recording results obtained. This nondestructive test technique was developed to replace one or more of the present destructive means (plunger, drum and/or road test) referred to in existing military specifications for new or recapped tires.

A description of the system follows: Located within a tank containing an ultrasonic couplant is a rotating platform on which is affixed the tire to be tested. This platform is driven by a variable speed drive which permits testing at various scanning rates. An ultrasonic transmitting transducer projects

ultrasonic energy through the tire which is picked up by a receiving transducer. Any discontinuity in the path of the beam (such as a ply separation) will cause a reduction in the amount of energy passing through the test area. This energy is converted to an electrical signal. By making use of a system of mechanical couplings with appropriate gear reductions, the tire is scanned spirally throughout the tread area while simultaneously the electrical flaw detection signal is recorded permitting the presence and location of flaw to be ascertained.

A final report dated February 1961 was published describing all work done under this program.

III- Inspection of Propellant Actuated Device Aluminum Catapult Tubes by Submerged Ultrasonic Technique

Upon request of the Frankford Arsenal Industrial Group, a study was made to determine the applicability of utilizing ultrasonic immersion techniques to detect laminar flaws in PAD aluminum catapult tubes. A sensitive ultrasonic immersion test technique was developed from this study for locating laminar flaws in uniform cyclindrical tubes. The single transducer pulse-echo mode of operation was found capable of detecting artifically prepared laminar flaws of 0.01 square inch in areas located on and parallel to the outer tube wall. Utilizing the above technique a number of the aluminum catapult tubes were successfully inspected.

A final report dated November 1960 was published describing all work done under this program.

IV- Development of Hall Effect Semiconductor Remote Application Clip-On Current Measuring Device

Under project "Automatic Checkout System for Combat Vehicles" authorized by the Army Ordnance Field Services, a Hall Effect semiconductor remote application clip-on current measuring device was developed by Frankford Arsenal as one of the transducers to be used to diagnose the electrical system of a tank without disturbing the major electrical connections. Present commercially available inductive types of clip-on ammeters are limited in range and type of output signal for intended application. In addition to the inductive types available, the Rome Air Development Center under Contract No. AF30(602)-2237, as depicted in Final Report RADC-TR-61-53, dated 1/16/61, refers to a laboratory type Hall Effect semiconductor millammeter device. However, the current range of this device does not meet the requirements of the intended application.

The Frankford Arsenal developed transducer design is finalized, several working models manufactured and is now integrated into the diagnostic system. Within this system, the device provides an electrical analog signal (of current flow) to a computer.

V- Hall Effect Semiconductor Potentiometer Studies

Under project OMS No. 5520.11.44100.03 authorized by the Army Ordnance

Corps, a study was initiated to determine feasibility of utilizing the Hall Effect principle for a potentiometer. Possible advantages of its use are size reduction of component, infinite resolution, brushless construction, broad frequency range and simplicity of construction.

During the first year's effort, major emphasis was placed on evaluation of characteristics of commercially available Hall crystals as concerns linearity, repeatability of signal and sensitivity. A breadboard model potentiometer has been tested using two crystals electrically connected in series. Although the configuration used as a first approximation is crude, the results show soundness of principle. Linearity of induced voltage as a function of flux density was checked as well as pole face shape effect on field distribution and the voltage and current levels necessary.

Next year's effort will be devoted to potentiometer application characteristics investigation including the following:

a. Linearity of Hall voltage with probe position.
b. Output resolution.
c. Input impedance.
d. Output impedance.
e. Temperature sensitivity.
f. Shock and vibration characteristics.

An interim report has been issued dated 31 May 1961 describing all work done under this program.

VI- Ultrasonic Studies of Weldments

Under project OMS 5010-11-80600-51-01 authorized by the Ordnance Material Research Office, evaluation studies of ultrasonic inspection techniques as applied to the determination of weld quality were conducted. Those factors limiting acceptance of the technique by industry were to be determined and possible solution considered.

During the current year program objectives were pursued through a series of experiments in which a number of aluminum butt weld plates with included material defects were inspected using existing recommended ultrasonic procedures*. Technically sound ultrasonic techniques were found sensitive to defects such as porosity, dross and lack of fusion. The test results indicated that additional effort is required to develop application methods capable of providing a high degree of repeatability. In addition, data presentation systems which are readily understandable by inspectors must be devised.

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*Such as techniques depicted by
(1) ASTM E-7 Committee
(2) Society for NDT Handbook by McMasters
(3) Ord Corps Ultrasonics Inspection Handbook ORD-M608-4

Next year's efforts will be devoted to the development of application systems capable of providing a high degree of repeatability, ease of application and employing readily understood presentation means. In addition, studies will be initiated concerning feasibility of ultrasonic inspection of resistance welds.

BUSINESS MEETING

MR. SHEPPARD - Gentlemen, I will now call the business meeting to order. Is there any old business that anyone would like to discuss from the floor? Mr. Goldspiel.

MR. GOLDSPIEL - May we have a report on what was done by the Steering Committee on the control of attendance to this and future conferences?

MR. SHEPPARD - It committed at the last conference that the current Steering Committee send to all members of that conference a questionnaire to obtain their opinion whether to confine our attendees to employees of the Department of Defense exclusively, or whether it should extend to other Government and Civilian Agencies. As most of you know, a questionnaire was prepared and distributed. At the same time that the questionnaire was sent out, the Committee reviewed the bylaws that were placed on this conference at its inception. Based upon what is read in the bylaws and the information received in response to the questionnaire, it is the decision of the Steering Committee that attendance to Conferences will be restricted to Department of Defense Activities without any participation by other activities, either Government or Civilian. Are there any comments?

MR. CAMBIS - This is Cambis from Edwards Air Force Base. Bill, I presume by your comments that this means that actual membership of the conferees would be restricted to Department of Defense Agencies. In my comments on the answer to the questionnaire I made a suggestion that the Steering Committee or possibly some other group might be permitted to invite either representatives of industry or the manufacturers of specialized equipment to augment or supplement the problem, depending, of course, on the peculiarity of the problem or problems submitted. Has this been given any consideration?

MR. SOBAK - This is Sobak of the Ordnance Tank Automotive Command, OTAC. Phil, you bring us to a point that only leads us back into the same situation we have just gone through and I believe, at the moment, that it probably would be out of order to pursue this point because the mandate of the last conference imposed upon the outgoing Steering Committee has been accomplished. I believe that we should live by the will of the majority. Now, if there is any interest in the new Steering Committee in pursuing some additional thoughts and it desires to investigate this matter any further, I doubt if it could ever be resolved within this group. Believe me, there are a lot of headaches and heartaches in ironing out this question. I suggest this matter be tabled. Also, any discussion on this matter should be tabled because the present Steering Committee followed the mandate of the last conference.

MR. SHEPPARD - Gentlemen, if you take the time to read the objectives of the conference as spelled out in the bylaws, you will find that each member who attends and presents comments at this conference must have legal representation. At this conference we discussed the pros and cons of individual contractors and manufactured equipment. Further, we give them an insight into future procurement problems. This, Gentlemen, is the basic reason why industry must be eliminated

from this as a Department of Defense group.

MR. HEFFIN - Heffin, Naval Ammunition Depot. I also think that industry should not be a party to this conference. However, I would like to remind the Conference that yesterday we saw a demonstration of a piece of equipment at the Quartermaster Center and a representative of the company which developed the equipment was here. This seems to be a very nice way to do it. I would like to propose that this be an acceptable one.

MR. SHEPPARD - Gentlemen, I hate to give a firm reply on this. Your comments are very good. However, they are repetitive of what has already been said in past conferences and meetings. I have been on the Steering Committee for two years and this has been a major point for a firm decision. A firm decision has been made. Because of the firm and decisive voting, the decision is that the conference will be restricted to Department of Defense personnel. I cannot see any bending or relaxing of this commitment. If there is any change which could not be considered a basic change to that decision, I am positive that from reading over the bylaws, this must be discussed by your new Steering Committee. With that comment, I wish to pass this subject and go on to the next. Do we have anything else in old business to discuss? All right. Is there any new business from the floor? Edmiston.

MR. EDMISTON - This is Edmiston from the Army Chemical Center. I waited until new business was announced because I felt we could rehash an awful lot of these things on old business. I propose that under new business we establish a precedence of reading at every conference the scope and objectives of this organization to educate new members and to refresh the older members. I propose, also, that in the handling of all problems we make certain that the person who submits the problem is at the conference to discuss all aspects and ramifications of the problem. Every so often we do have a case where a problem was not fully explained and certain other facets which would have been important to us and our suggestion on how to attack the problem were not able to be discussed. I also propose that the slate for a new Steering Committee be selected by a nominating committee prior to the start of the conference. This would allow for the participation of all members and permit the conference attendees to pursue other business.

MR. SHEPPARD - Thank you, Paul. Is there any other new business? If it is something that concerns the new Steering Committee and not the entire group, I suggest strongly that you present it to that committee at a later date. Mr. Inglis.

MR. INGLIS - One of the things that is being emphasized is the benefits of this group. I think, for the matter of the record here at today's session here, that one of the most outstanding benefits after the presentation of the problems has been the fact that the host facility, the Brooklyn Navy Yard, the Naval Ordnance facility, Frankford Arsenal and others have offered to confirm their technical knowledge and suggestions by actual test. In other words, not only is technical opinion given, but proof of it is being furnished by a host of installations. I believe that this should be made a part of the minutes of this conference.

MR. SHEPPARD - Thank you, Warren. I think this is excellent. It will be included in the minutes as suggested. Is there any other new business? I have been advised by our host Steering Committee member, Mr. Budnick, that a new brochure on the QM Research and Engineering functions will be available to all conferees. It is not available at this time but will be sent with the copy of the formal minutes of this conference. It is a different brochure from that which you have been given. Mr. Dave Stein.

MR. STEIN - Stein, Picatinny. Bill, I would like to propose, in the way of new business, that in future meetings at the end of the conference, perhaps at a time like this, we have a statistical breakdown; if you will, a listing of all facilities by name which have at least one representative attending the conference. I think it is significant to us to be aware of any facility which in the past has been represented at this conference and does not have at least one representative at any one particular conference. I think this is significant information. I feel this procedure would make us aware of such a situation. If it were to become serious enough, I think this information would permit us to take the necessary action to preserve the entire conference.

MR. SHEPPARD - If I do not have any who disagree with us, I will ask the host to list one of the pages of the minutes, a complete list of those activities which were represented at this conference. Would you take this matter up with your new Steering Committee, please? Saul, excuse me for interrupting, and I will still give you time to push your idea if you wish it, but I think that we had better think about a place for our next conference. First, it is the decision of your new Steering Committee that they will accept invitations for the next meeting site. They will, either before or during their initial committee meeting, decide on the selection of this site. However, prior to reaching a final decision, they will send out an official letter to the Commander of the activity and request an official invitation. I solicit, at this time, informal invitations or suggestions for the site for the Thirteenth Defense Conference on Nondestructive Testing. Mr. Heffan.

MR. HEFFAN - Heffan, Naval Ammunition Depot. On behalf of the Naval Ammunition Depot, it is my pleasure to invite the Thirteenth Defense Conference on Nondestructive Testing to be held at the Naval Ammunition Depot, Concord, California. Concord is about thirty five miles east of San Francisco. With the nice climate that is available most of the time, and the meeting and motel facilities, plus the fact that we have had several successful conferences here this past year, I believe that you would enjoy having the 1962 conference there.

MR. SHEPPARD - Thank you, Mr. Heffan. Are there any other invitations? Mr. Holloway.

MR. HOLLOWAY - On behalf of the Naval Ordnance Laboratory I would like to invite the Thirteenth Defense Conference on Nondestructive Testing to be held at that facility. For those of you who are not familiar with this location, the Naval Ordnance Laboratory is just north of Washington, D. C., in White Oak, Maryland. I know I need not say any more about the facilities available in Washington. We would be very happy to have you there and most happy to host the conference.

MR. SHEPPARD - All right, Navy. Are there any other invitations? If there are not, I will close the business session for this meeting for this conference. Gentlemen, during the first official meeting of the Steering Committee for the Twelfth Defense Conference on Nondestructive Testing we decided that one of the members of this Steering Committee would be the official historian for the Conference. We elected Mr. Jim Holloway, Naval Ordnance Laboratory, as the historian. I suggest strongly to the new Steering Committee, after seeing the job that he has done, that you continue with this idea. I think it is an excellent way of keeping a record of the accomplishment of the Nondestructive Testing Conference. I have asked Mr. Holloway to give us today the information that he has obtained; information as feedback for this conference. At this time I will turn this meeting over to Mr. Holloway.

MR. HOLLOWAY - During the last several months those members who presented formal and informal papers at the Ninth, Tenth, and Eleventh Nondestructive Testing Conferences have been bombarded by a number of questionnaires. The questionnaire was designed to obtain information on the status of the problem solution. At this time I would like to present some of the replies of the Eleventh Conference. The problem concerning the welds on the inner centrifuge was presented by Mr. Robert Snyder, Aviation Medical Acceleration Laboratory, U. S. Naval Air Development Center. Mr. Snyder indicates that this problem is still actively being pursued at his installation. Although they are installing a new centrifuge in 1962, the problem of monitoring the welds in this apparatus still remains. It is requested, therefore, that all members consider this problem. You will find it very well covered in the minutes of the Eleventh Conference.

A second formal problem presented last year was concerned with the inspection of the wall in rocket motor tubes for soundness. This problem was presented by the Quality Assurance Directorate, U. S. Army Chemical Corps Materiel Command. This problem is being actively pursued and will be taken up in fiscal year 1962. Again, the details can be found in the minutes of this conference. We offer this for your further consideration.

The third problem was concerned with the inspection for separation and lack of bond between the motor case insulator and the propellant in rocket motors. I presented the problem for the U. S. Naval Ordnance Laboratory, White Oak, Maryland. This problem has reached a partial solution. This consists of a means of determining unbondedness in the liner of the rocket motor by means of the old tried and true method of tangential radiography. However this technique has been refined to a point where an unbonded condition can be picked out by means of a density ratio on the film. This is an old method. It has been refined by Aerojet General who are using it at this time.

Captain Metzger, Warner Robins Air Materiel Area presented two problems. The first was concerned with the inspection of critical structural parts without removal. For those conferees who would like a little more information on this, apparently these were steel bolts and aluminum forged fittings for which there was the problem of inspection for cracks. This problem is still being actively

pursued. Captain Metzger has indicated that the Air Force, in general, and Warner Robins Air Materiel Area, in particular, have embarked on a rather extensive program of creating specialized radiographic inspection manuals for most aircraft. These manuals will give quite complete descriptions of methods and points of inspection, and cover such things as angles, exposure time and other pertinent details. The use of portable magnetic inspection and dye penetrants should be used to supplement radiographic inspection methods when considered applicable. Captain Metzger indicated that, to date, no one method has been found to be more than partially satisfactory, but apparently he has found some solution to his problem.

Another problem presented by Captain Metzger concerned the nondestructive testing for corrosion determination in inaccessible areas on the C130 aircraft. If you remember the problem, there were certain milled skins on the wing section and cargo floor of the aircraft which are inaccessible and for which we would like to determine the extent of corrosion. This problem is still being actively pursued. Some corrosion problems have become so severe that early detection of corrosion has changed the concept of excessive costs. This opens the door to possibly more exotic methods of inspection. He stated that presently known test methods offer little promise. He continues here that, since the Eleventh Conference, corrosion in integral fuel cell panels on the C130 aircraft has become a larger problem than some of those presented at the conference. It would be to his advantage for members of this conference to review this problem as presented in the minutes of the Eleventh Conference and to give it special consideration. If any one can help him, I know that he would be most happy to hear from you.

Mr. F. G. Schrader, Naval Air Station, Alameda, California, presented a problem on crack determination in chromium plated cylinders in the cylinder heads of reciprocating engines. He says that he has found a solution to this. The solution was found about May 1960 and employs the technique of caustic etching followed by low power magnification examination. He indicates that the method used is a standard caustic etch of aluminum alloys which is followed by a neutralization with nitric acid. The caustic blackens the surface of the plated area and the nitric acid removes this discoloration. The crack is indicated by a black line where the caustic seeps into the discontinuity. Mr. Schrader presented a problem at the Tenth Conference concerning fluorescent penetrant operational problems. I believe that, at that time, he was looking for an emulsifier or some method if improving the means of the fluorescent penetrant operation. He says that he has found a solution to this problem. He uses an emulsifier which penetrates faster due to its lower viscosity. In addition, the emulsifying time was increased from one half of a minute to one minute. The emulsifier conforms to Specification MIL-I-25135, Type I, class 2. Anyone who has a similar problem and who wishes more information about it, can get more information by contacting Mr. F. G. Schrader, O&R Department, Code 341, Naval Air Station, Alameda, California.

Back to the Eleventh Conference again. The problem on the brazed joint soundness of the aircraft heat exchanger was another problem which was presented. This one was from the Indianapolis Air Procurement District. There is an

indication that this problem is still being actively pursued and that either an ultrasonic or eddy current method might be the possible solution.

I have skipped one of the replies on the Eleventh Conference from Mr. Wallace K. Thomas, Army Rocket Guided Missile Agency concerning the standardization of penetrameters for radiographic inspection of solid propellants. Since we are all concerned, or may have been concerned with the testing of solid propellant motors, this problem of penetrameters is an urgent one. Mr. Thomas indicates that it is still actively pursued at his installation. He feels that the best possible approach or method of solution may be the incorporation of a penetrameter specification into MIL-STD-286, Propellant, Solid, Sampling Examination and Testing.

One more problem solution from the Eleventh Conference; an informal problem from Picatinny Arsenel concerning the inspection of shape charge cones. Mr. Warren Inglis covered that very well in his presentation.

Gentlemen, that is all I have at this time. Thank you.

MR. SHEPPARD - Thank you, Mr. Holloway, I think you have done an excellent job on that report, Jim. Mr. Steve Sobak from Ordnance Tank Automotive Command would like to make an announcement about the Society of Nondestructive Testing Meeting. Could you give it at this time, Steve?

MR. SOBAK - This portion does not have to be recorded. I will make my announcement brief. The meeting is scheduled to be held in New York City during October 1962. I solicit the participation and attendance of all who are interested in nondestructive testing. I feel sure you will find the meeting worthwhile. Thank you.

MR. SHEPPARD - I wish to thank each member for their cooperation in answering the various questionnaires sent out by the Steering Committee or its members. Without your replies we would not have been able to make the various reports and show what progress has been made on the different problems which are being pursued further. It is becoming more obvious that there has been a great deal of progress and that numerous solutions have been reached. I wish to say, further, that I am very glad to have had the opportunity of getting to know, informally, the number of people working on these projects and the tasks involved in their solutions.

At this time I wish to announce the new executive secretary and invite him to join me on the stage. By the vote of your new Steering Committee, the members have selected Mr. Richard Rowand of the Air Force to be the new Executive Secretary. Could we give him a show of hands?

Next, I would like to take a little time to show my appreciation for a few gentlemen who worked with me during the past year. Some of them worked with me the prior year. They have had to put up with a great deal during the past year in setting up this conference and in handling the business of these conferences.

They did an excellent job. They accepted every responsibility that was offered. I don't think it could have been handled any better than they did. I will now read the names of the members of the Steering Committee and will ask the people to stand. Mr. Steve Sobak, Mr. Bernie Hoffman, Mr. Richard Rowand, Mr. Jim Holloway, and Mr. Budnick.

Mr. Budnick, if you would remain standing for a moment. As I told you out in the hall, personally, and to make it official, it is considered that you have done an excellent job. I don't think it could be handled any better, any more efficiently. I would like to extend the appreciation of the group, as well as my personal appreciation to both you and General Tribe for inviting us and putting up with us during this conference.

If there are not any more comments, I will close the Twelfth Defense Conference on Nondestructive Testing and turn this meeting over to the new Executive Secretary, Mr. Richard Rowand.

MR. ROWAND - Thank you, Bill. This is a real honor right at this moment. Maybe a year from now I will not be saying this. I think before we go any farther that we should have a round of applause for Bill Sheppard in recognition of his excellent work despite all of the pressures he had last year.

The site for the Thirteenth Conference will be determined during the first Steering Committee Meeting. The members will be advised at a later date what particular organization was selected. Unless there is any other business, I think the motion for adjournment is in order. Yes?

MR. HEFFIN - Heffin, Naval Ammunition Depot. I came to this conference with a problem; that was getting information on testing some turbine blades. I have spoken to several of you. The information that I received, I can definitely say, will save the Government approximately $14,000. This is the price I can put on the information which I received. I think this conference is definitely worth while for the additional reason that you can get valuable and effective information in a rapid and easy way. I request that my remarks be made a part of the minutes of this conference.

MR. ROWAND - It will be so done.

MR. BUDNICK - Despite words to the contrary, I enjoyed having all of you here. I know General Tribe and his entire staff did, also. Again, if any of you are down in this area, please feel free to call me at any time. The extension is either 268 or 519. I will arrange a private tour of our facilities if you do not go through today. Now, I, too, wish to make a statement for the record and which will be included in the minutes of this conference. In talking to Warren Inglis last night and again today, he indicated that he has received considerable benefit from this conference. The demonstration and the discussion of the Insulation Value Tester, and again I'm going to take credit for the Quartermaster Corps, he thinks, has great potential for one of the problems with which he is faced. He is going to take time to go into this in more detail. He

indicated that some of the technical knowhow which was demonstrated here will go a long way in solving his problem. I am not too sure about this next part, but Warren talked to someone about a valve or some small device. This again has a potential for another of his problems. I would ask Warren to talk for himself, but he had to leave because of prior commitments. So, for the record, Warren Inglis has found this, the Twelfth Defense Conference on Nondestructive Testing of great benefit to him. He has found here the potential answer to two significant problems at his own installation. Again, the minutes of the conference will include this comment. Thank you.

MR. ROWAND - Thank you, Mr. Budnick. Is there a motion for adjournment? Motion has been made and seconded. Any objections? The meeting is adjourned.

TWELFTH CONFERENCE ON NONDESTRUCTIVE TESTING

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Abbe, Earl H.	Springfield Armory Springfield, Mass.	X3215
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Stellabotte, Michael L.	AML, NAMC Attn: XM712-MLS Philadelphia 12, Pa.	X3580

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Wright, George W.	Attn: MOMQQL Brookley Air Force Base Mobile, Ala.	X3884
HOST: Budnick, Morris L.	QM R&E Command, U. S. Army Air Delivery Equipment Division Standardization Branch Natick, Mass.	X268

LOCATIONS AND DATES OF PREVIOUS CONFERENCES

Organizational Meeting, Watertown Arsenal, Watertown, Massachusetts
3 - 4 Oct 51

2nd Conference, Frankford Arsenal, Philadelphia, Pennsylvania
23 - 24 Jan 52

3rd Conference, U. S. Naval Gun Factory, Washington, D. C.
19 - 20 Nov 52

4th Conference, Research and Development Laboratories, Fort Belvoir, Virginia 17 - 18 Mar 54

5th Conference, Naval Ordnance Plant, Indianapolis, Indiana
16 - 17 Mar 55

6th Conference, Detroit Arsenal, Centerline, Michigan
9 - 10 Mar 56

7th Conference, U. S. Naval Ordnance Test Station, China Lake, California
19 - 20 Feb 57

8th Conference, San Antonio Air Materiel Area, Kelly Air Force Base, Texas
4 - 5 Dec 57

9th Conference, Army Ballistic Missile Agency, Redstone Arsenal, Alabama
15 - 16 Oct 58

10th Conference, Naval Air Material Center, Philadelphia, Pennsylvania
6 - 7 Oct 59

11th Conference, Oklahoma City Materiel Area, Tinker Air Force Base, Oklahoma
13 - 15 Sep 60

SUPPLEMENTARY TECHNICAL PAPER 10969-09

INTEGRITY OF MISSILE-MOTOR-LINER BOND UTILIZING ULTRASONICS

By

E. D. BESSER AND G. T. STUART

A recent missile development has two propellant grains which are installed in the motor tube; one provides initial boost thrust and the other the sustainer thrust.

The motor tube is a nominal 5-inch diameter, 4130 steel tube with a 0.075-inch wall thickness; roughly 72 inches long, threaded at the front end and lock-ring grooved at the aft or nozzle end.

When fired, the booster accelerates the missile. After a given time delay and complete exhaustion of the booster section, the forward sustainer unit is fired. The longer grain is the booster (Fig. 1). A ported bulkhead separates the sustainer from the booster portion of the motor tube (Fig. 2). The sustainer unit fires through the bulkhead and the booster portion of the motor tube. A heat barrier must therefore be provided in the booster section to prevent overheating of the missile motor wall.

A laminated, asbestos-phenolic liner is the heat barrier used in the motor tube (detail in Fig. 2). Improper bonding between the liner and the motor tube can cause "burn-throughs" of the steel tube which results in missile failure. The integrity of the liner-motor bond is therefore important to the proper functioning of the missile.

The motor-tube specifications require that the first 8 inches of the liner aft of the bulkhead shall be bonded to the motor tube and shall be free of any internal delaminations. The remaining portion of the liner is considered less critical. This portion may have delaminated or unbonded areas not greater than eight square inches in total area. Any one delamination shall not exceed two square inches, nor shall unbonded areas greater than one square inch be adjacent to one another within one inch.

Delaminations, air bubbles, blisters, or resin pockets can normally be determined by visual inspection; however, to verify the integrity of the bond between the motor wall and the asbestos-phenolic laminate requires nondestructive techniques during motor tube production.

The development of an applicable nondestructive method for the verification of the bond integrity became a joint effort between the Source Inspection and the Product Evaluation Branches of the Naval Ordnance Test Station.

X-ray techniques were tried, but found to be inadequate. Voids, bubbles, and delaminations could be seen in an X-ray negative or on a fluorescopic screen; however, bond integrity could not be resolved.

Ultrasonic techniques were investigated and found to be quite applicable. Immersion techniques, however, were avoided because the asbestos-phenolic liner absorbs water. To provide a known means of testing and calibrating, sections of motor tube were prepared and lined with layers of phenolic-filled asbestos as specified for the motor-tube liner. By the use of silicone-grease spots or double layers of cellophane between the liner and the steel tube, known void areas were built into the test sections.

A Metroscope[1] sonic gage, being used at the beginning of the program for verification of the wall thickness, was applied to the bond problem. It was noted that by reducing the signal amplitude, a well-bonded area was represented on the 'scope by a straight-line trace (Fig. 3). The crystal in the probe was contoured to the diameter of the tube. By calibrating the instrument on "no-bond" and "good-bond" areas, voids or poorly bonded areas provide clear indications (or 'pips") on the screen (Fig. 3).

The Sonoray[2], an ultrasonic pulse detector, also gave good results when a 2.25 megacycle transducer was utilized. A fine setting of the automatic gate made it possible to readily detect void areas by their characteristic wave-form.

Since using the Metroscope and Sonoray to monitor missile manufacturers' techniques, the rejection rate has been reduced from 19 percent to less than 5 percent during a two-year period. An even greater improvement might have been realized were it not for manufacturing difficulties in securing a good bond--particularly aft of the ported bulkhead.

The Metroscope technique has also been applied successfully in checking the bond between the steel wall and a pressure-sensitive, silicone-fiberglass liner used in spherical missile-motors. Motor walls as thin as 0.030 inches and motor diameters as large as 24 inches have been successfully inspected. The spherical motors are initially lined. Subsequent loading of propellant is accomplished by evacuating the motor. Any bubble- or gas-formation between liner and motor wall during vacuum loading enlarges a poorly bonded area. These areas contribute to potential missile failure.

The successful application of ultrasonics to the Sidewinder Missile was cited by CAPT V. P. Healy[3] in his keynote address to the "Missiles and Rockets Symposium" at Concord, California in April 1961. Since ultrasonic inspection techniques have been used on Sidewinder missile motors, there has been no known failure attributable to motor-liner inadequacy.

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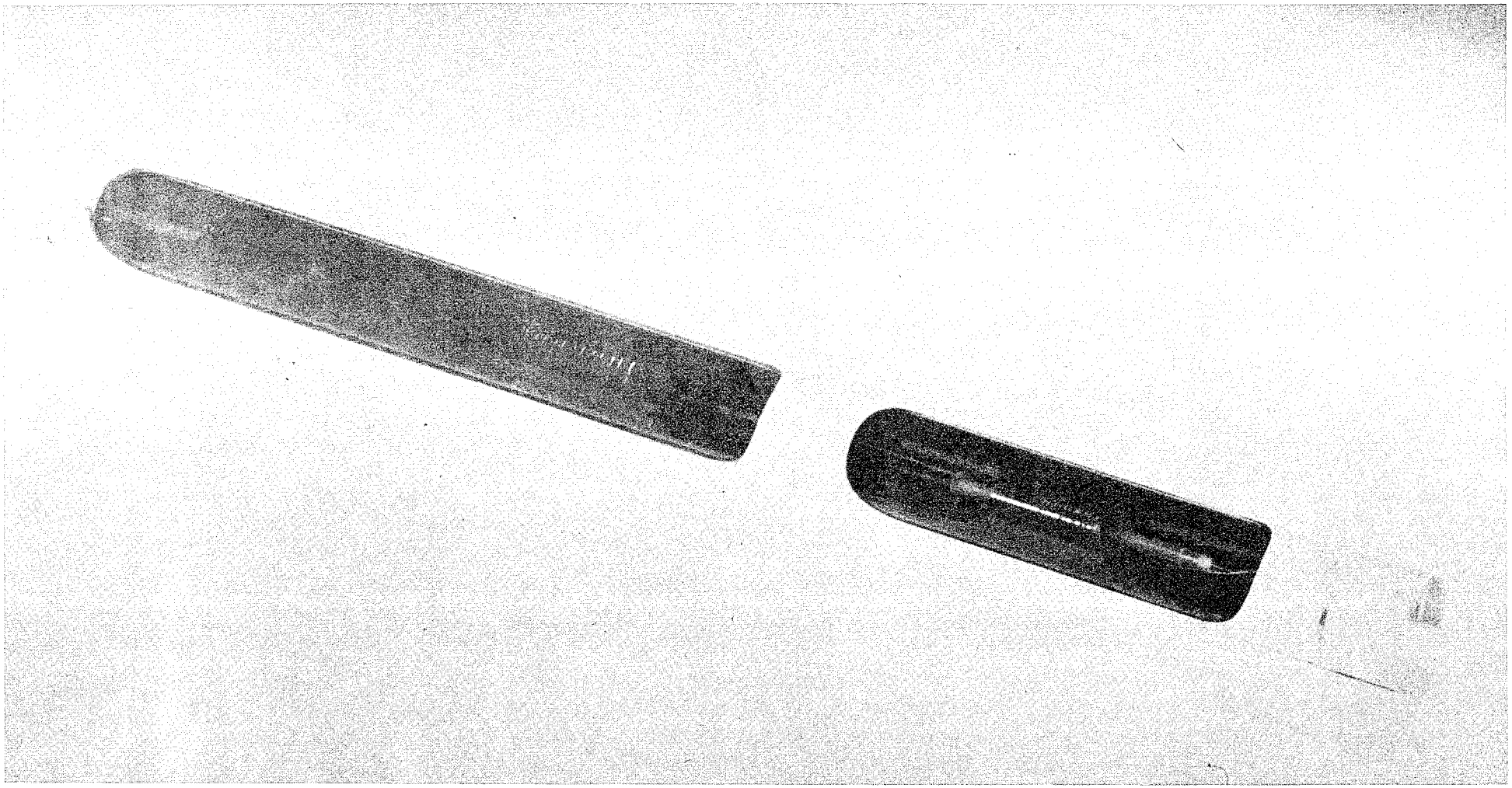
[1] Photocon Instruments, Altadena, California
[2] Branson Instruments, Stamford, Connecticut
[3] Director of Research for Anti-Submarine Warfare, BuWeps, Washington, D. C.

TOP.

FIGURE 1. Sidewinder Motor Tube (cutaway). Longer booster grain at left. Sustainer grain at right.



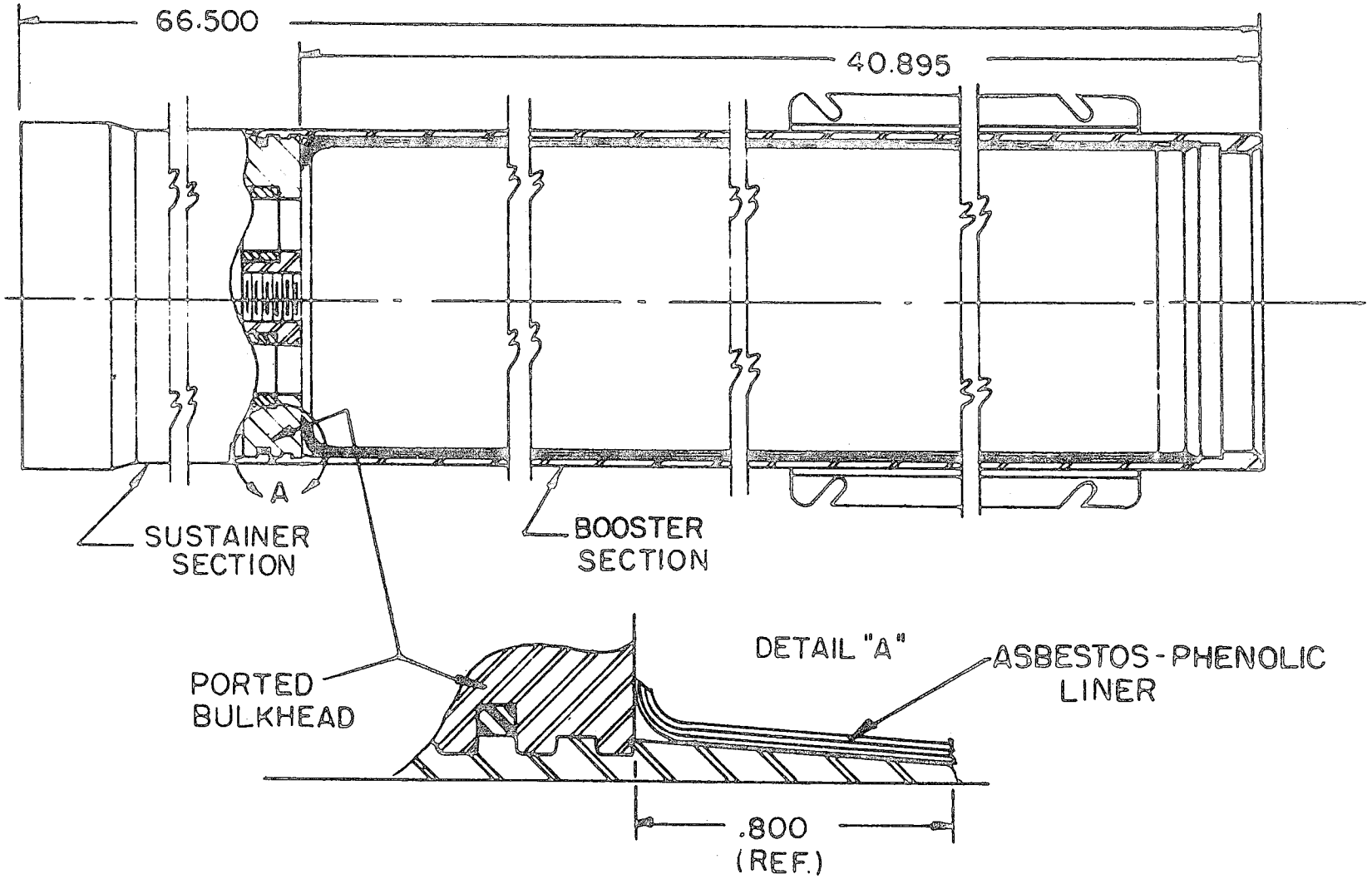
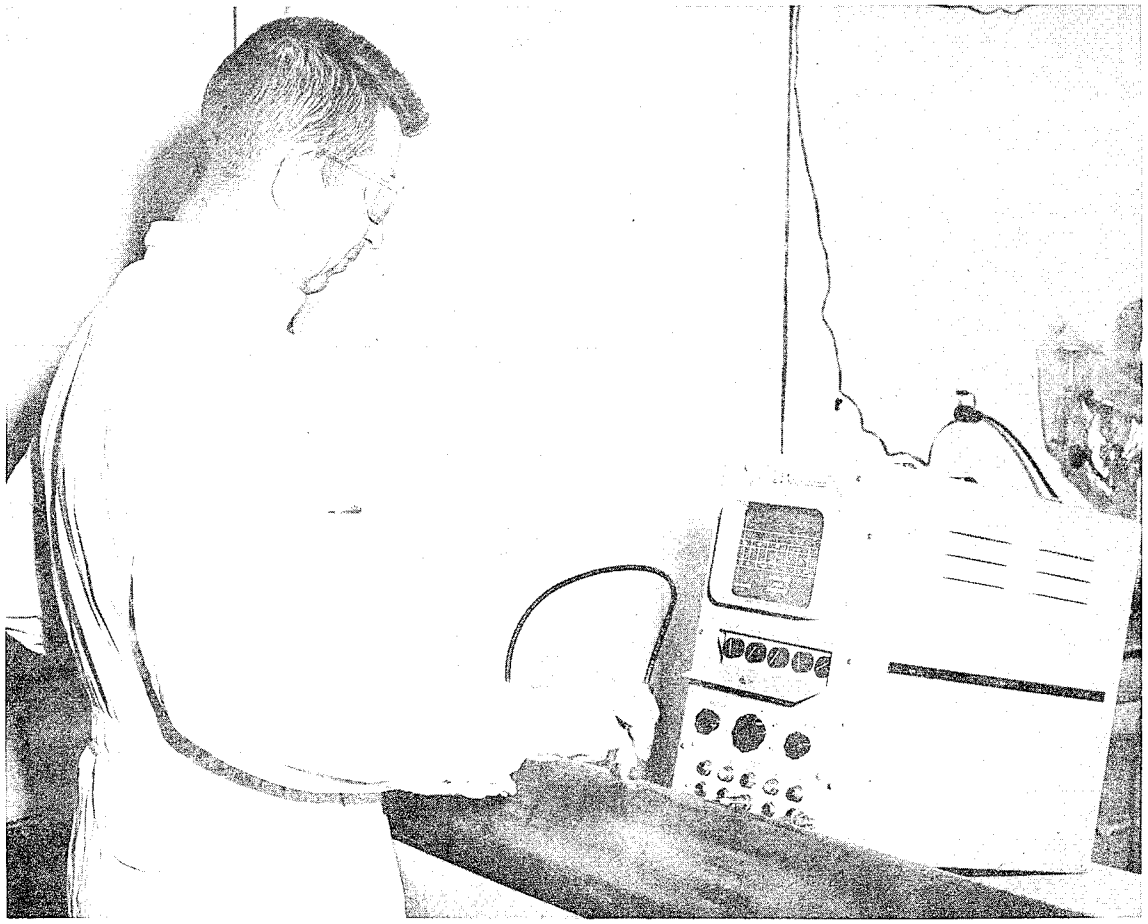
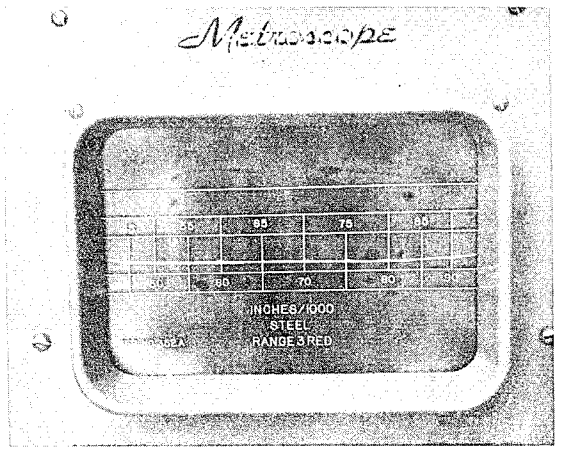
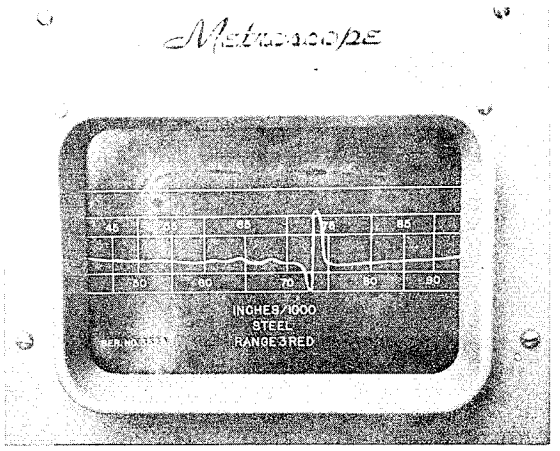


FIGURE 2. LINED MOTOR TUBE





GOOD BOND



POOR BOND

FIGURE 3. ULTRASONIC TESTING OF MOTOR TUBE