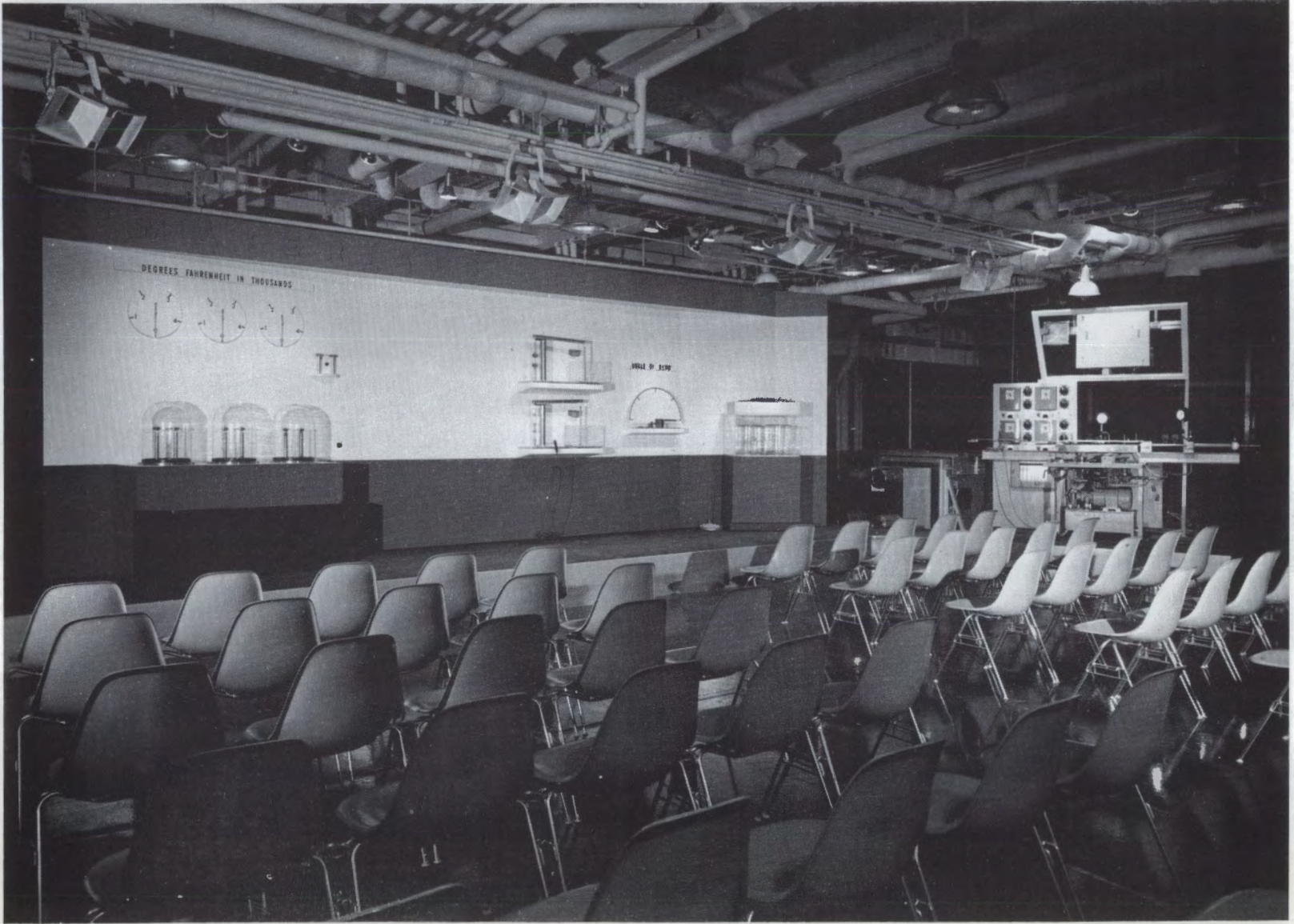


MATERIALS RESEARCH

Presented
at

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Cleveland, Ohio
October 4-7, 1966

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION



MATERIALS RESEARCH

Materials technology is the key to continued successful development of advanced propulsion systems. This holds true for space flight as well as for flight in the air atmosphere. Propulsion systems involve such a wide range of environments that many specialized materials are required. One of the objectives of this Center is to perform research on materials and on the structures in which they are used so that suitable materials will be available in advance of need. In our presentation we shall describe only a few of the materials problems and the research being conducted to solve them.

We can organize this materials research presentation according to the temperature range for the intended application. The first slide (fig. 1) demonstrates how this temperature range has been expanded over the past two decades and how wide it is at present. Both maximum and minimum temperatures of interest are plotted versus calendar year. If we go back to the period before World War II, when the principal source of propulsive power was the piston engine, we find a temperature of about 1000° F at the upper end of the scale. This temperature was associated with the stressed hot parts of the engine, such as the piston and cylinders. At the lower end of the scale, the extreme is about 70° F below zero, and is associated with the outer skin temperature of airplanes in the upper atmosphere. When the jet engine was introduced early in the 40's, the high temperature of interest approximately doubled, the critical components now being turbine blades and vanes. No important change occurred in the limit of the lower temperature of interest. With the advent of the space age, such components as the exhaust nozzles of rocket engines required materials to withstand temperatures of the order of 5000° F or higher. Liquid oxygen was introduced as a fuel for the rockets, bringing the lower temperature of interest to approximately 300° below zero. Re-entry vehicles expanded the upper temperature limit of interest to well above the melting point of the most refractory

materials available today and introduced the need for a new materials application approach known as ablation. The ablation process calls for materials which continuously vaporize, melt, and decompose, thus cooling the structure beneath. Higher thrust requirements also brought about a further reduction in the lower temperature in interest associated with the storage of liquid hydrogen as a fuel. Thus, at the present time, we need materials to withstand temperatures from near absolute zero to temperatures well above the melting point of the most refractory materials.

I will first describe some of our work to provide advanced materials for use at the lower extreme of the temperature scale. There is a need for light weight, high strength propellant tanks for liquid rocket engines. Certain aluminum alloys and stainless steels are currently being used for such tanks. In order to decrease weight, we are developing glass fiber reinforced plastics for tanks. Although these materials are lighter and far stronger for a given weight than metallic materials, they are porous at cryogenic temperatures. The fluids will actually seep through the tank wall. We are investigating the use of thin non-porous liners for these tanks and have made progress in the application of both metal foils and plastic or polymeric films. We would like to describe for you some of our results with polymeric films. Currently available plastic films become brittle at cryogenic temperatures and tend to burst when fluid pressure fluctuations occur. One of the goals we have set for ourselves is the development of plastic films that can be used at 300⁰ to 420⁰ F below zero without becoming excessively brittle. One of the ways we are approaching this problem is through control of processing.

I am sure you are aware that the properties of metals are often dependent upon how they are processed - - how they are forged or rolled.

The properties of polymeric films also depend to a great extent on how they are processed - on the proper orientation of the polymer

molecules. We have been investigating whether processing methods can be developed for available plastics that will improve their low temperature properties. At your extreme right is a machine used to provide heat and strain to plastics so as to orient the molecules and thereby improve the properties of the plastic. The machine stretches a plastic film in two directions simultaneously at any selected temperature. Here is a small square of plastic similar to the one contained in the machine. It is covered with a fluorescent paint to improve visibility for this demonstration. Heat is supplied under the specimen. You may view the stretching process of a similar square of plastic from the mirror above the machine.

The effect of this type of processing on the behavior of this material at cryogenic temperature can be observed in the apparatus at the right of the front panel. Here are two belljars. Both are filled with liquid nitrogen. Its temperature is approximately the same as that of liquid oxygen, and it is being used because it eliminates the safety hazard associated with liquid oxygen. Inside of each of these belljars is a plastic film. The one at the left is a commercially available product. The one at the right is identical in composition, except that it has been processed in the manner described to reduce its brittleness at cryogenic temperature. As I apply pressure under each of the films, you can observe how each one deforms. These plastics have been coated with a fluorescent paint to improve visibility; normally, they are translucent white. Note that a bubble is being formed under the action of the pressure. The commercially available film bursts before this bubble can grow to any appreciable size. The specially processed plastic film can withstand a large bulge indicating that it can be stretched considerably more than the commercially available product. Thus, we have succeeded in producing a plastic film which is better able to withstand pressure at rocket fuel temperatures.

Let us now focus our attention on a room temperature materials application. The next slide pictures (fig. 2) the world's largest solid propellant rocket motor case. It's dimensions are roughly 22 feet in diameter and 70 feet long, with a propellant case wall thickness of approximately $3/4$ inch. When filled with propellant and test fired, this motor developed 3-million pounds of thrust for 2 minutes, and this is only a half-length model. The full-length motor is designed to develop 6-million pounds of thrust for 2 minutes. This much thrust would lift 1500 medium sized automobiles. Two approaches to the production of such large motor cases involving different fabrication techniques and materials were investigated in feasibility studies funded by the Air Force and NASA. One tank made by one of these approaches failed during preliminary hydrotest at a pressure only slightly greater than one-half the planned proof pressure. The wreckage is shown on the next slide (fig. 3). Careful study of the wreckage revealed the failure origin to be an internal crack about the size of a paper match stick associated with a repair weldment. Unfortunately, small cracks like these can neither be avoided nor detected prior to testing of the rocket. In the case of this particular motor case, the weld material was seriously weakened by the crack and catastrophic failure resulted. Subsequent tests have shown that the successfully fired motor case would not have been so sensitive to such a flaw; hence, its successful test performance.

The next slide (fig. 4) shows the manner in which materials are now tested to determine in advance the possibility of material weakening as a result of the presence of cracks. The upper weldment is the type used in the failed motor case. It is called a double-pass submerged arc weld; it is a very coarse, sometimes dirty weld. The lower figure represents, schematically, the weldment used in the successfully fired motor case. It is a clean, multipass tungsten inert gas weld.

Controlled size cracks are purposely introduced into the weld material and the weld subsequently tested for strength. A stress

analysis reveals the stress necessary to cause catastrophic failure which, as you can see, was about three times as great for the weldment of the type used in the successful tank. A conventional smooth bar test of the weld zone would have shown little difference in strength between the two types of weld. The message we mean to convey at this point is that spectacular failures of the type shown may now be avoided through the proper testing of precracked specimens. Such studies comprise an entirely new field of science called fracture mechanics. While our illustration here relates to the room temperature application of solid rocket motor cases, fracture mechanics plays an indispensable role in design at all temperatures.

We are also conducting research to provide improved materials in the intermediate temperature range, from about 1500^o to 2400^o F. An example of the need here is the powerplant for the SST. In order to achieve the high power requirements needed by the SST, its engines must operate with higher combustion gas temperatures than present day engines. An increase of only 100^o F in gas temperature substantially increases engine thrust. To accommodate higher gas temperatures, improved materials are needed. These materials must retain their strength as well as be resistant to oxidation at the higher temperatures.

We should emphasize the great difficulty associated with providing materials having the needed oxidation resistance to operate satisfactorily at high temperatures in these engines. Alloys of some of the refractory metals such as tungsten already have the strength necessary to permit operation at temperatures several hundred degrees above the intermediate range. However, they would literally burn up in the oxidizing environment of the engine combustion gas. Thus, "the oxidation barrier" is greater than the "strength barrier" as we seek higher use temperatures.

Nickel-and cobalt-base superalloys, having much lower melting points than tungsten, have a much lower high-temperature strength

potential. However, they have the advantage of inherent oxidation resistance. We are therefore emphasizing research to improve the high-temperature strength of the superalloys. Unfortunately, the oxidation resistance of even these alloys decreases as temperature increases, and we are therefore concerned with improving their oxidation resistance as well as their strength. In superalloys the oxidation attack or degradation can be quite subtle and is completely revealed only upon examination of the surface layers of the exposed metal with the aid of the metallurgical microscope at high magnification.

The next slide (fig. 5) shows the microstructure of a typical nickel-base alloy before and after it had been exposed to air at 1900^o F for 100 hours. The region adjacent to the surface of the sample which was exposed to the heated air is quite different from the rest of the sample. A wide zone has been depleted of strengthening elements by their reaction with oxygen from the air which diffused into the metal. The greater the width of this affected zone, the more the strength of the alloy is reduced. Measuring the depth of this zone is one way to measure oxidation resistance. The smaller this zone, the better the oxidation resistance.

We have recently developed a strong, extremely oxidation resistant nickel-base alloy at this Center. The next slide (fig. 6) compares the oxidation resistance of this alloy and two representative currently used nickel-base alloys, Rene' 41 and MAR M-200, in terms of the total affected zone width. Comparison is made after several hundred hours exposure to air at 1900^o F. The NASA alloy has a significantly smaller affected zone than either of the commercial alloys, about one-seventh as large as Rene' 41 and one-half as large as MAR M-200 indicating its better oxidation resistance. Also, the strength of the NASA alloy is comparable to the strongest commercial alloys.

Another problem area associated with nickel- and cobalt-base superalloys is that of alloy instability and embrittlement. This is of significance as we seek materials for long time use, particularly in commercial applications such as the jet engine powerplant of the SST. For example, jet engine parts must be able to deform under unexpected loads. If materials can deform sufficiently, they will not break when such loads are applied. Many superalloys are ductile when first put into service, but after long exposure at intermediate temperature they lose their ability to deform. The jet engine and superalloy producers are very concerned about this problem and have made notable contributions. We are also active in this problem area. The next slide (fig. 7) illustrates the degree to which we were able to reduce embrittlement in a cobalt-base alloy, L-605, which is widely used in jet engines. The slide shows how the ability of the alloy to deform, changes with the time of exposure to high temperature. It was found that silicon, which can be present in this alloy in amounts up to 1 percent according to manufacturer specifications, contributes to embrittlement of the alloy. By reducing silicon content to less than 0.23 percent, the alloy has a relatively high elongation even after 200 hours exposure at 1600⁰ F. This is shown by the upper curve. If the silicon content is nearer the maximum called for by the manufacturer, the alloy has a much lower ductility. We can demonstrate this effect by means of a simple bend test in this apparatus. I will insert two specimens of this alloy in this fixture. These specimens are sheet strips about 4 inches long and 1/2 inch wide. Their thickness is 0.050 inch, comparable to the thickness of sheet used in some jet engine parts. Both specimens have been exposed at 1600⁰ F for 200 hours. The only difference between them is that one contains less than 0.23 percent silicon; the other contains nearer 1 percent silicon. For ease of identification, the high silicon specimens are painted red. As I rotate this handle, the specimens will be bent in a fashion very much the same as clamping one end of

them in a vise and pulling at the other end with a pair of pliers.

The specimen with the high silicon content fails after bending through an angle of about 60° , as shown by the indicator on the dial. The low silicon content specimen, however, continues to deform. In fact, it is impossible to break it within the limits of this apparatus. Both specimens are equally strong in that they can sustain the same load. However, by a simple compositional change, it has been possible to improve significantly the ductility of an alloy that is used in commercial and military aircraft engines, thereby greatly extending its usefulness.

It is a continuing objective of our research to find materials of much higher strength than those now available. A particularly promising approach is that of making composite materials from fibers. It is frequently true that fibers of a material will be much stronger than the same material in bulk form. This is illustrated in the next slide (fig. 8). The strength of a tungsten fiber or wire can be increased about twofold from about 200 000 to 400 000 psi as its diameter is decreased from 0.040 to 0.004 inch. By way of comparison the strength of bulk tungsten, i. e., a bar of this material, is shown on the lower right to be only about 80 000 psi. The research problem is to determine whether such fibers can be put together in some form analogous to reinforcing rods in concrete to produce usable practical materials. The next slide (fig. 9) shows an experimental sample of tungsten fibers in copper matrix at $\times 50$. The advantage of fibers and fiber composites will be demonstrated in this apparatus. Here are two loading frames. The specimen in the top loading frame is simply a strip of tungsten. The lower loading frame contains two composite material specimens loaded in series. The upper specimen is merely a bundle of 300 tungsten fibers, each 0.005 inch in diameter, but their load carrying area adds up to exactly the same as the solid specimen above. The lower specimen has the same total cross-sectional area of tungsten fibers, but they are bonded together by a copper matrix to make a usable solid

composite material. The specimens are loaded by metal shot which is introduced into these boxes. I will now open the top gate and allow the shot to load the upper specimen. Note that it fails when the level of the shot in the box rises to about 1 inch. This produces a stress of about 80 000 psi. By opening the lower gate, I can load the composite material specimens. The level of the shot in the box is about four times as high as that in the upper box and the composite material specimens did not break. The stress applied is about 320 000 psi. The demonstration clearly shows that a bundle of fine wires of tungsten is much stronger than the bulk material. It also shows that such wires when embedded in a matrix of a softer, ductile material such as copper is much stronger than bulk tungsten.

An appreciable portion of our effort in these fiber-reinforced composite materials is directed towards their use at intermediate temperatures near 2000^o F for applications such as an advanced turbojet engine. At these temperatures, a major new problem is introduced - metallurgical reaction between the fiber and the matrix. The specimen we used in our demonstration was made up of tungsten wires in a copper matrix. The next slide (fig. 10) illustrates the problem of metallurgical reaction. The figure at the left shows the microstructure of a W-Cu composite at a magnification of 500. You can see that there is no evidence of reaction with the copper matrix. Since we are seeking composite materials for use near 2000^o F, however, a different matrix material is needed. Copper is not suitable. It melts a little below 2000^o F. We must therefore substitute a high-temperature material such as the nickel or cobalt superalloys. The center figure shows what happens when other matrix materials are used; we find, that when only a few percent of nickel, here 10 percent, are added to the copper, a severe reaction takes place between the fiber and the matrix. Deterioration of the tungsten grains that are adjacent to the matrix material is evident. The figure at the extreme right shows a nickel matrix with tungsten fibers. The fibers were

completely disintegrated upon exposure to high temperature. We have been studying various methods of minimizing such reactions. The next slide (fig. 11) illustrates one of these methods. The figure at the left shows the case where the tungsten wires were infiltrated with an alloy of copper and 10 percent nickel in the liquid state. The figure at the right shows a composite made by applying high pressure to the matrix which is in powdered form in the solid state. Both specimens here held at 2200° F for $1\frac{1}{2}$ hours. Notice that the matrix here is 100 percent nickel, which is very reactive with the tungsten in the molten state, but far less reactive when kept in the solid form. We are also studying other approaches; for example, using high selectivity in the choice of matrix and fiber to minimize the reaction and coating the wires to protect them from the matrix while at high temperature. Although the solution to this compatibility problem appears difficult, we believe that combinations of high strength fibers in suitable alloy matrices will be found. This will permit these materials to be used at high temperatures thereby realizing their exciting potential.

At the highest temperatures of interest, above about 3000° F, for applications such as rocket exhaust nozzles, we must resort to the use of refractory metals such as tungsten. Tungsten has the highest melting point of any metal, about 6100° F. This is several thousand degrees above that of nickel- or cobalt-base alloys which were discussed earlier.

Over the past several years we have been contributing to the developing technology and fundamental understanding of this metal and its alloys. Tungsten has a disadvantage in that it is very brittle at room temperature.

We have shown that we can produce an alloy of high purity tungsten containing 2 percent rhenium that will be ductile at room temperature in the cold-worked condition in either sheet or bar form. This was achieved by adding rhenium using electron beam melting in high vacuum.

Through our basic research utilizing high purity single crystals and the tool of thin film electron microscopy, we are developing an understanding of how rhenium contributes to this beneficial effect.

The research we will describe in a little more detail deals with our alloying studies to develop tungsten alloys with improved strength at very high temperatures. The next slide (fig. 12) shows the strength of the NASA alloy which is a specially processed material containing 0.2 percent hafnium, 0.02 percent carbon. You will note that the total alloying addition is only 0.22 percent. The strength advantage over unalloyed tungsten is about a factor of 6 at 3500^o F. This is the strongest metallic material known at 3500^o F. The temperature advantage of this alloy can be demonstrated in the apparatus to your extreme left. Each belljar contains a specimen which can be heated by passing an electric current through it. In the belljar on the right, is a nickel-base alloy, Rene' 41; in the center belljar is a bar of pure tungsten. The jar on the right contains the NASA tungsten alloy. The belljars are used to provide a vacuum environment so that the two refractory metals do not oxidize as they are heated. When I press the button, the current will pass through each of the test specimens. The degree of heat can be observed from the brightness of the specimen and by the dial indicator above each belljar. The protective shield has been placed in front of the jars to protect your eyes from the glow. The same amount of weight is suspended from each specimen. Under the action of the load you will observe that each specimen will deform. As it deforms, it will ultimately slip through the suspension posts and lose contact with the source of current. I will now press the button, energizing the specimens. Note that the Rene' 41 was able to withstand only approximately 1700^o F, tungsten 2300^o, but the refractory metal alloy nearly 3800^o F, or 1500^o F higher than unalloyed tungsten. It should be emphasized that this achievement in tungsten alloy development is due in part to the availability of the processing equipment here in this building.

Here we can process from tungsten powder (show) to bar stock (show) at higher temperatures than anywhere in the world.

To recapitulate, I have described several materials research programs being conducted at this Center which contribute either directly or indirectly toward providing improved materials for current or future use in aircraft and space power propulsion systems. For use at the lower end of the temperature scale, we have developed plastic films for liners in rocket fuel tanks. We have shown how proper processing techniques can make such fibers more ductile at cryogenic temperatures. For room-temperature applications, the science of fracture mechanics has been applied to predict the effect of cracks upon material strength. For application in the intermediate temperature range up to about 2200^o F we have developed a new nickel-base alloy. This alloy has better oxidation resistance than available commercial nickel-base alloys and high-temperature strength comparable to the strongest nickel-base alloys. The problem of embrittlement in superalloys has been described. We have shown how we were able to reduce embrittlement in a cobalt-base alloy that is used in many commercial and military jet engines by a simple compositional change. Also for application in the intermediate temperature range we are developing ultra-high-strength composite materials. Finally, we have developed the strongest known metallic material above 3500^o F, a tungsten alloy.

We are continuing our research both to provide new materials and to obtain an understanding of the fundamentals of materials behavior to provide a basis for further advances.

OPERATING TEMPERATURE CHRONOLOGY

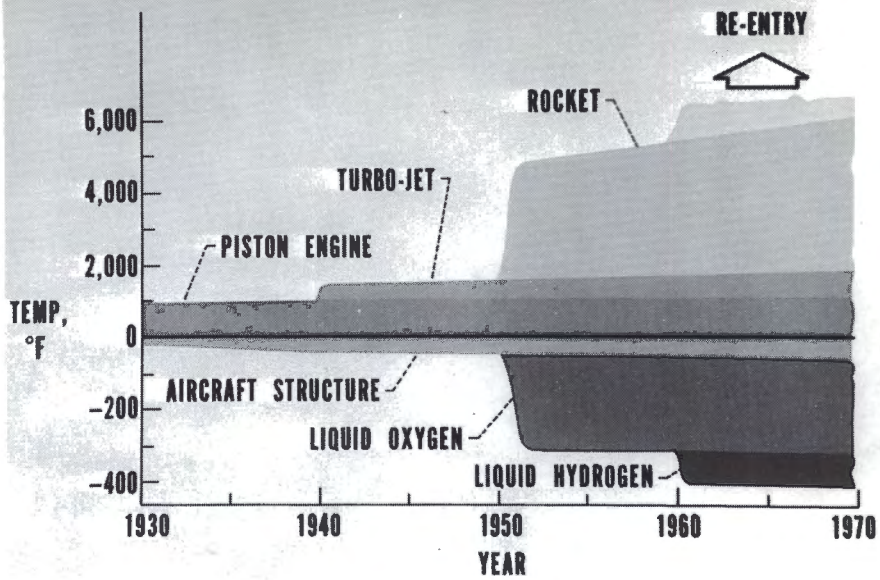


Figure 1

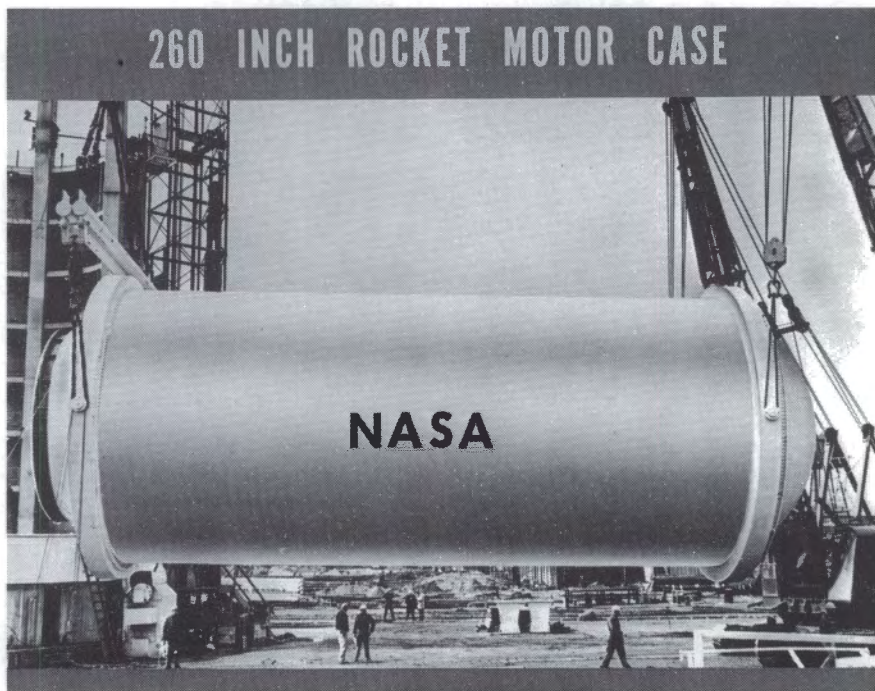


Figure 2

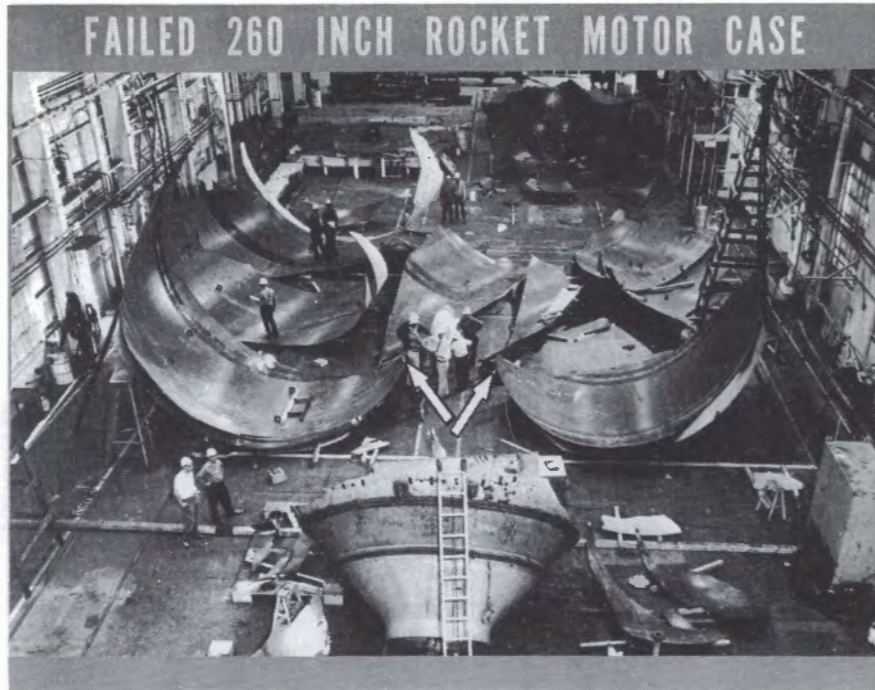


Figure 3

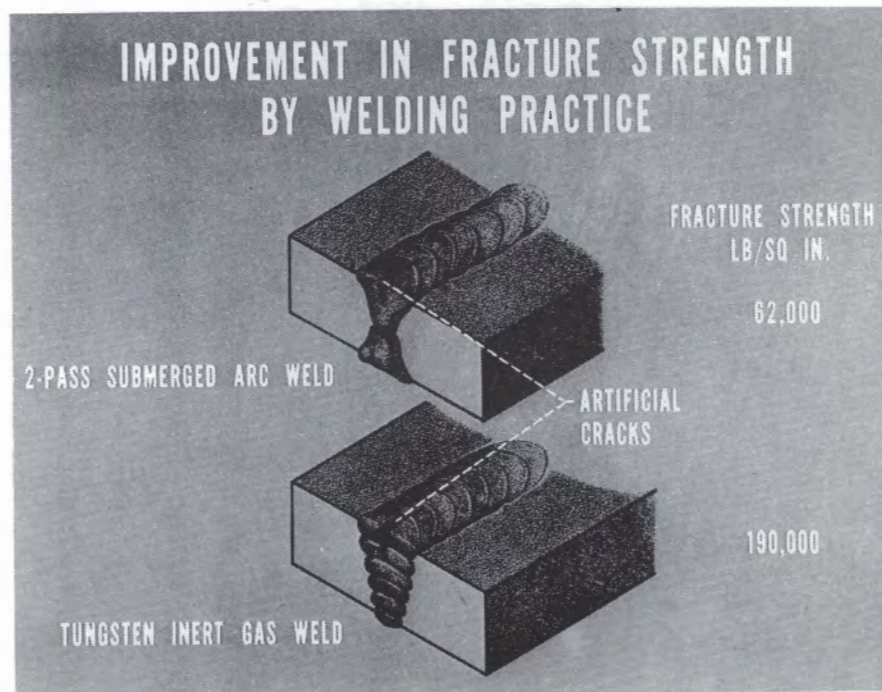
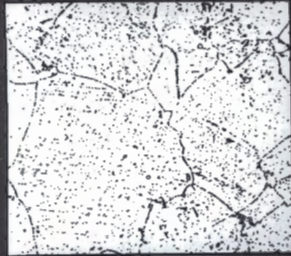


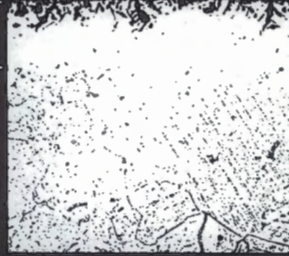
Figure 4

OXIDATION OF TYPICAL NICKEL-BASE SUPERALLOY



UNEXPOSED SAMPLE

TOTAL
AFFECTED ZONE



EXPOSED TO AIR FOR
100 HR AT 1900° F

Figure 5

OXIDATION OF NICKEL-BASE ALLOYS AT 1900° F

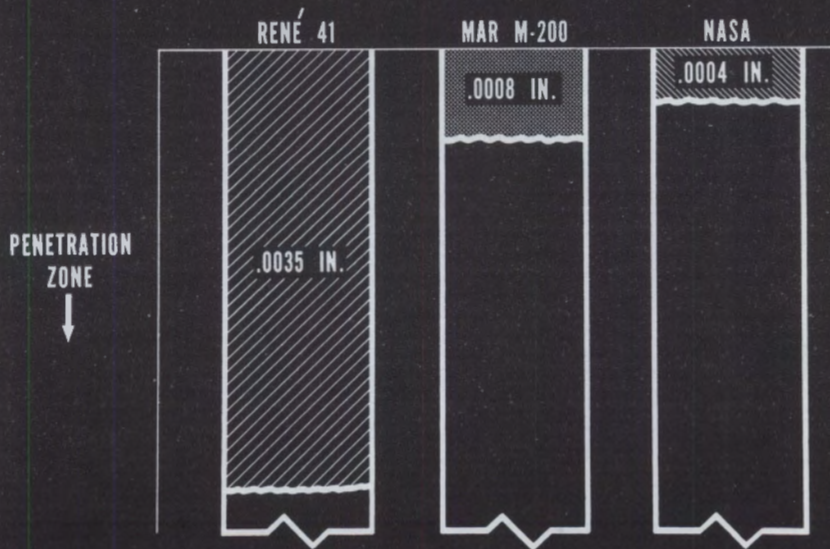


Figure 6

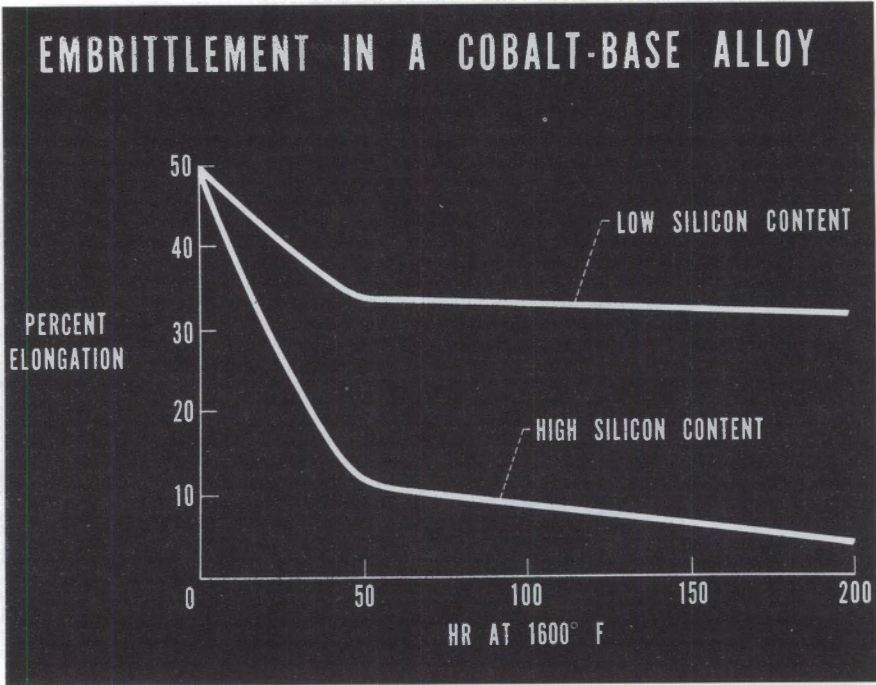


Figure 7

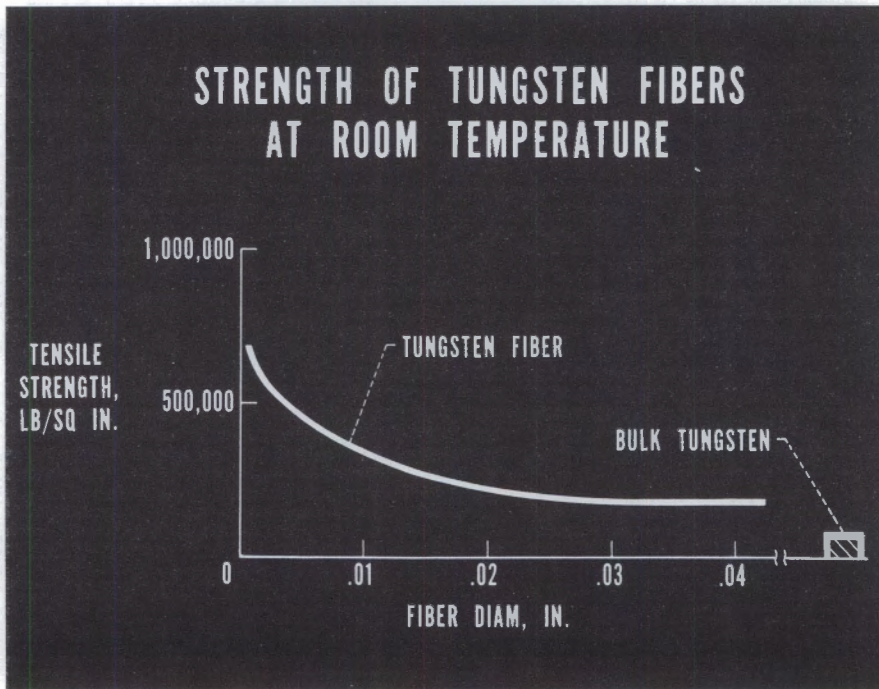


Figure 8

COMPOSITE MATERIALS FROM METAL FIBERS

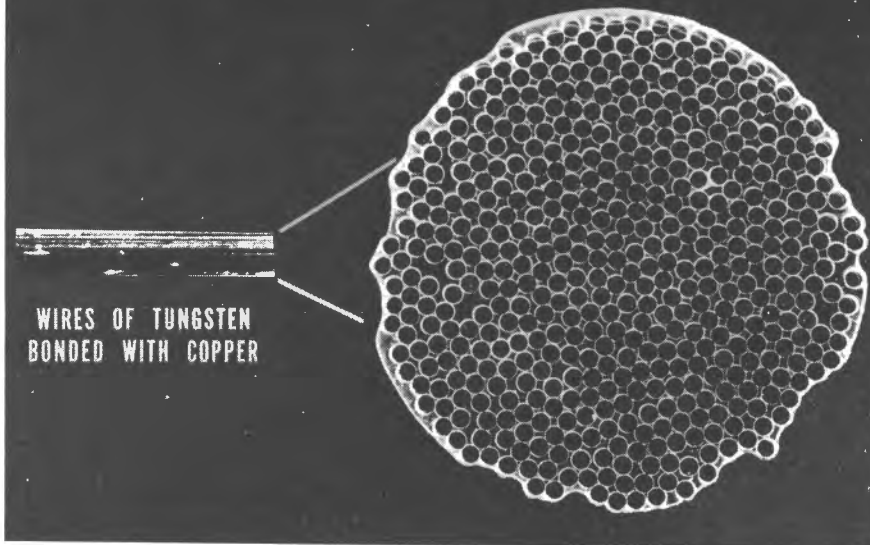


Figure 9

REACTION IN COMPOSITE MATERIALS

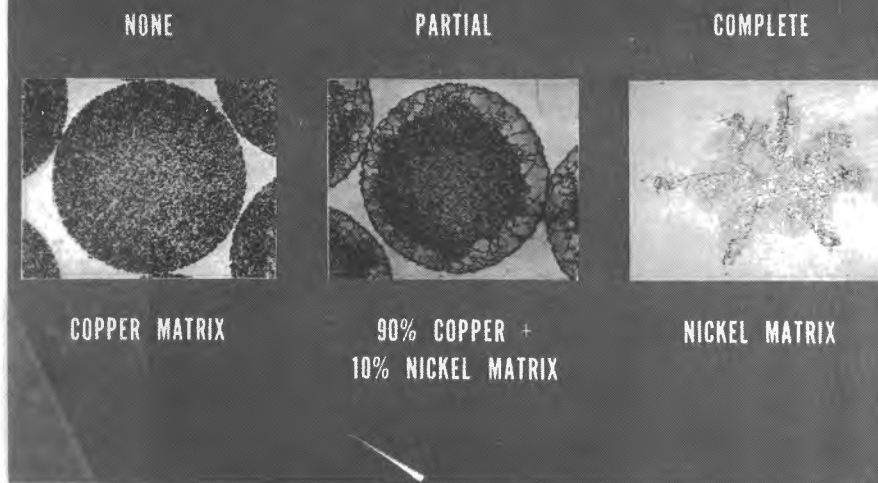


Figure 10

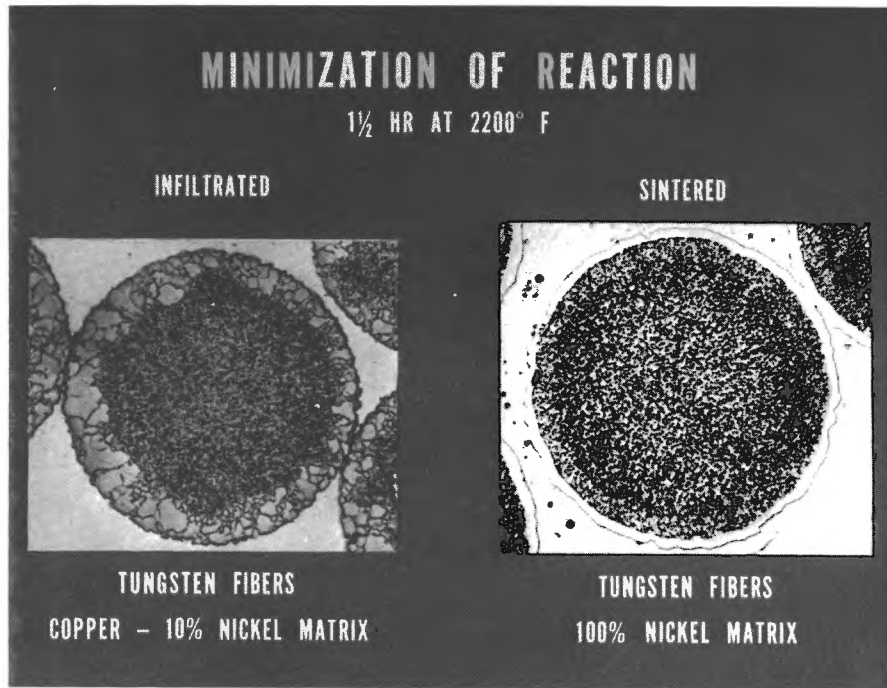


Figure 11

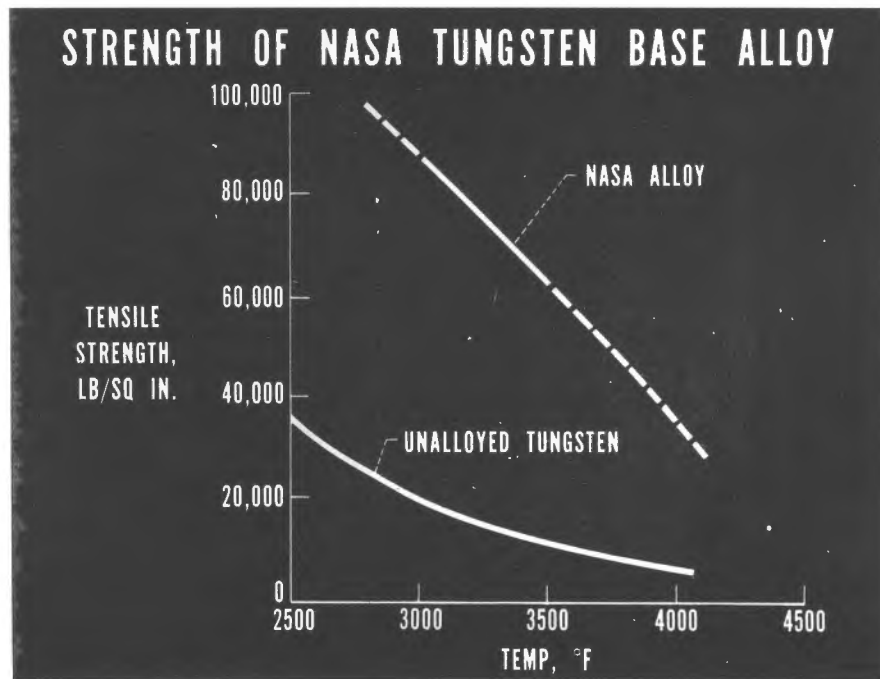
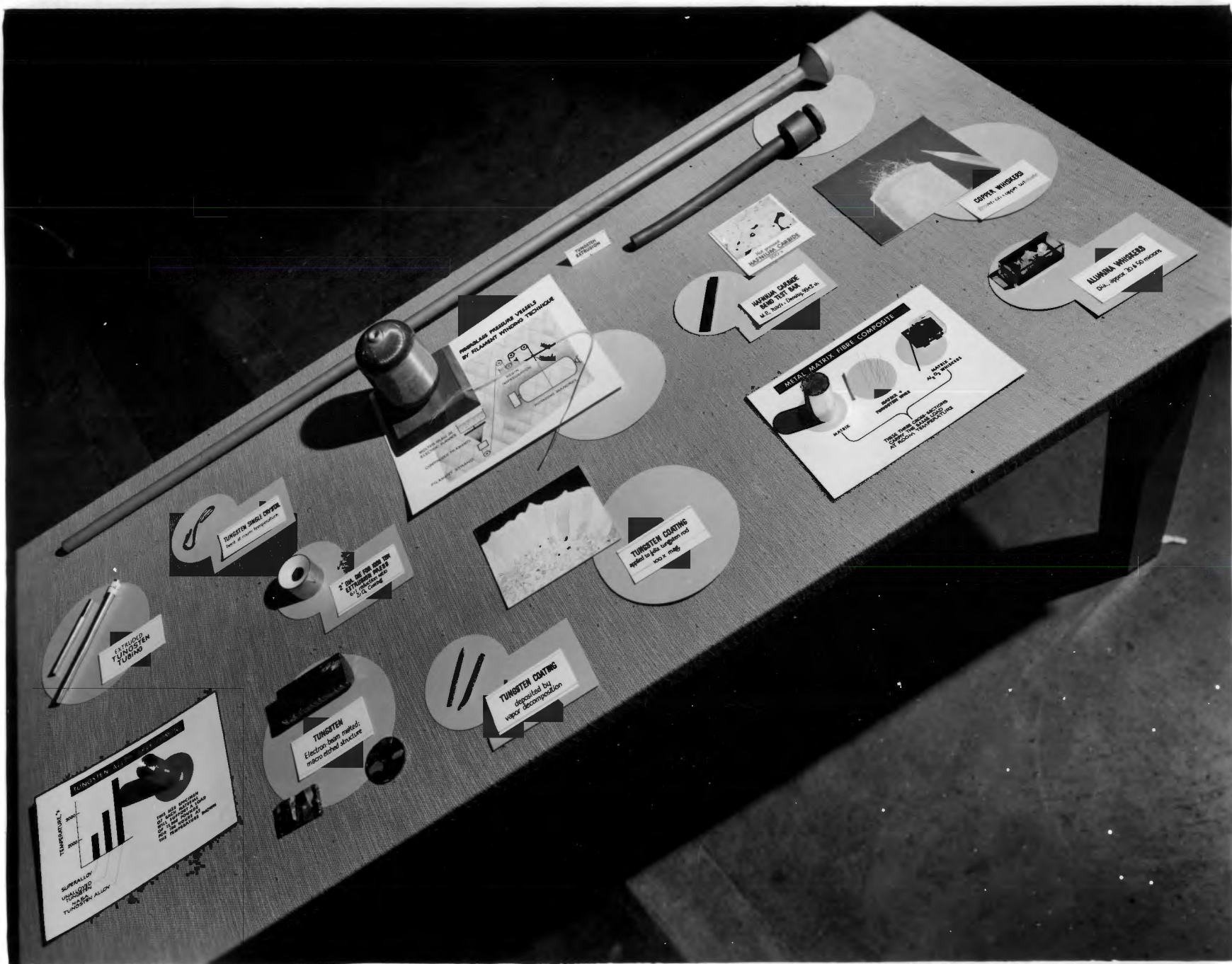



Figure 12



REFRACTORY METALS



Refractory metals are those which have a melting temperature above 4000° F.

RESEARCH, DESIGN AND FABRICATION

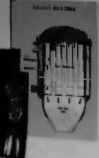
While space vehicles such as the Saturn S-200 (Structure 1) will utilize highly refractory alloys of hafnium, zirconium or tungsten for leading edges and heat shields to withstand the high temperatures resulting from reentry heating during return into the earth's atmosphere, the life support, housing, in the presence of oxygen, the ship may be coated with an oxidizable ceramic coating capable of providing protection for short times at temperatures up to 3000° F. Based on advanced and available data on being tested.

RESEARCH


Refractory metal ingots are being developed jointly by NASA and the AFOSR (Advanced Research Office) at Wright-Patterson Air Force Base, Dayton, Ohio, to meet the needs of the high-temperature materials program. The program is being conducted by AFOSR.

RESEARCH

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CONVENTIONAL MATERIALS




Conventional high temperature materials are presently used to withstand aerodynamic heating in advanced aircraft.

The result of the research program will be reported in March 1967. The first report will be on the results of the research program on the use of the materials in the design of the engine nozzle, and will be followed by reports on the use of the materials in the design of the engine nozzle, and will be followed by reports on the use of the materials in the design of the engine nozzle.

The S-200 is a conventional research vehicle that has reached orbit and is in orbit at 100,000 feet. The program requires the use of the S-200 at an altitude temperature above high speed flight. However, the vehicle cannot be used for the purpose of the program.

All current research on the use of the S-200 is being conducted by AFOSR. The program is being conducted by AFOSR.



Materials are the key to success in space. Historically, improvements in aircraft and their propulsion have been preceded by successful development of improved materials. Attainment of our goals in space requires accelerated progress in materials research.

NASA

The initial impetus of materials in the space program has been significant. Every one of NASA's major research or development centers has conducted studies on the field. An extensive research effort is being devoted to long-range research on solid-state physics and metallurgical concepts, studies on applications of materials to critical NASA problems, and research and development leading to specific hardware. Basic materials studies are supported with subcontracts, awards, lectures, and seminars.

LONG RANGE RESEARCH

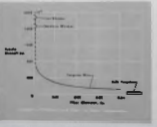
At a 200-mile altitude the atmosphere is so thin that the aerodynamic heating is negligible. However, the materials must be able to withstand the high temperatures of reentry. The materials must be able to withstand the high temperatures of reentry. The materials must be able to withstand the high temperatures of reentry.

FOUR BROSSE STATION

Additional long-range facilities for aerospace propulsion research and development are located at NASA's Four Brosse Station in Huntsville, Alabama. Operated as an arm of the Lewis Research Center, Four Brosse Station is the site for long-range heating of nuclear propulsion components as well as basic materials research in the use of the S-200 as a research vehicle. In NASA's nuclear test reactor, all basic data and dynamic tests of complex nuclear engines and components are performed in other facilities located here.

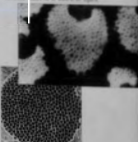
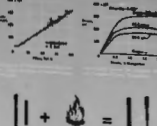
HIGH STRENGTH MATERIALS

Spacecraft materials must provide high strength over a wide range of temperatures (-423° to 5000° F). One way this may be achieved is to use high-strength composite materials.



Unless approached in the problem of providing super-strength materials for spacecraft are under investigation. The strength of some materials available only in the form of filaments or whiskers may be from 10 to 100 times that of the bulk material. The problem is to assemble these filaments or whiskers into a form which can be utilized as a structural material.

One way to provide the materials in the research base necessary is to establish suitable composite materials based on carbon. Such a material consists of bundles, consisting of high-strength filaments woven in a matrix of resin. Similar composites have been synthesized using glass or synthetic plastic and tungsten filaments in a matrix of copper. Frequently ultra-high-strength whiskers will be used to combine a matrix, matrix.

Incorporation of filaments or whiskers into a matrix composite may be accomplished by numerous ways. Liquid infiltration or powder metallurgical techniques have generally been used. Results of tests with single composites indicate that their properties may be predicted with reasonable accuracy. Varying the relative amounts of fiber and matrix results in composite having and results range in composite having properties "tailor made" to suit the need.

REINFORCED PLASTIC MATERIALS

Space vehicles require high-strength, low-density structural materials for maximum payloads. Glass-reinforced plastics have the highest strength-to-density ratios of any present day production-type material.



High-performance structural materials for spacecraft are under investigation. These materials must be strong enough to support the weight of the vehicle in their liquid state, they subject the average bulk materials to temperatures ranging from -297° to +423° F. Such low temperatures cause many materials to become too brittle and weak to provide for practical use. Fiberglass-reinforced glass-reinforced plastics appear to be promising composites with the correct materials for very high strength and strength-to-density ratios (about 2 to 4 million lb. in. at -423° F.).

SIMULATED MICROMETEOROID COMPARISON OF IMPACT RESISTANCE OF ALUMINUM AND REINFORCED PLASTIC MATERIALS





The present state of space vehicles must be able to withstand meteoroid impact resulting from a point of micro-meteoroid particles which may be as small as 0.001 inch in diameter. Laboratory tests indicate that reinforced plastic materials have excellent impact resistance when hit and penetrated by high-velocity particles, as illustrated by the test specimens at the right.



FRACTURE
MECHANICS



Increasing number of failures of high thrust rocket nozzle
necessitate a program for strength-weight ratio of nozzle prop-
ellant tank for space vehicles.



