HIGH TEMPERATURE MATERIALS

The problems associated with the achievement of high mach number flight are being discussed today in several of the exhibits. I would like to consider one of these problems, the materials problem. Essentially, there are two aspects to the materials problems. The first, to provide materials to withstand the aerodynamic heating that we encounter in the nose of missiles and the skin of aircraft -- and the second, to provide materials for the temperatures we expect in the propulsion systems of aircraft.

Before getting into specific materials problems, it is desirable to take a look at where we stand today and at the desired temperature goals for the future. We can do this with the aid of the first slide. On this we have plotted the maximum use temperature of jet engine turbine blade alloys against the year they were first used. We can readily see that from 1945 to the present time the maximum use temperature of the blade alloys has increased from about 1350 to about 1650°F, a gain of only 300°. The brackets on the temperature scale on the right indicate our approximate goals for the next 10 years. For the turbojet, material temperatures as high as 2500° are urgently desired. The desired temperatures for the ramjet are from about 2500 to 3100°, while for the nuclear rocket temperatures up to 5000°F are desired. When we consider the relatively slow progress in the past 10 years and compare it to the requirements for the next 10 years, we realize just how extremely difficult are the goals that have been established for materials research.

I would like to outline some of the basic materials problems associated with the attainment of these goals and to indicate very briefly some of the approaches that we are pursuing at this laboratory.

First, let us see why getting to a high temperature presents such a difficult problem. Here we have a crude model of a metal viewed on a submicroscopic scale. The atoms are represented by the plastic spheres. At temperatures near absolute zero these atoms are nearly stationary and in the regular array that we see. At any temperature other than absolute zero the atoms vibrate about their equilibrium positions; the higher the temperature the greater the vibration. The atoms on the model are vibrating more vigorously now, simulating a higher temperature. Of course, we have made no attempt to represent this motion to exact scale. In fact, at the temperatures we are talking about, the atoms vibrate at about 10^{13}, or 10 million, million cycles per second. We can also note that there is considerable randomness to the vibration -- some of the atoms have a far greater amplitude than do others. Because of this large amplitude, the atoms may slide past each other quite easily. Some of them attain amplitudes large enough to allow them to escape from their lattice sites. These effects are what basically cause our problem. The ability of the material to retain its dimension, that is to resist creep under load, depends on the ability of its atoms to remain in their original sties. However, if an atom escapes or if groups of atoms slide past each other, a microscopic deformation of the material results. With a sufficient number of such microscopic deformations, the whole engine part loses dimensional stability, creeps to excessive length, and in time may break into several pieces.

A second property of these escaping atoms is that they are chemically very reactive. They readily combine with oxygen and this corrosion can cause the failure of engine materials that are otherwise satisfactory.

The next slide will illustrate one of the ways of increasing the use temperature of materials. Here we have the microstructure of a high temperature alloy magnified 750 times. We can see a uniform dispersion of very fine particles -- these small black dots which makes the microstructure appear grey. In this alloy these fine particles are chromium carbide -- in other high temperature alloys they may be nickel aluminide or columbium nitride. The particles lock or key the atomic layers together, and the strengthening that results is roughly similar to the strengthening of concrete by the dispersion of gravel in cement. As long as the hard particles are there, the materials retain their strength. However, at the higher temperatures which are desired for advanced aircraft and missiles the hard particles, such as the type we see here, dissolve into the matrix and lose their effectiveness in keying the material against deformation. This, as we have seen before, is due to the increased mobility of atoms at the higher temperature. We can demonstrate this effect and a potential solution to the problem with these models. Each of these is a highly enlarged model of the microstructure of a high temperature alloy, such as we have seen in the previous slide. In each case the matrix contains small hard particles that key it against excessive deformation. The model on the left represents a common high temperature alloy which contains the conventional, hard, small particles. These particles are not especially resistant to temperature. Thus, if we play a flame on the model to simulate what happens when the alloy is subjected to high temperature, the particles dissolve, much like sugar dissolves in coffee; except, of course, in metals, both components are solid. The model at the right represents a type of structure with which we have been experimenting. In this case, the particles are especially chosen for their heat resistance and chemical inertness. On heating, these do not dissolve into the matrix even at much higher temperatures. An alloy containing such inert and refractory powder is analogous to the sintered aluminum powder product commonly referred to as SAP. With this technique the capabilities of aluminum have been extended by several hundred degrees fahrenheit.

We are studying methods for utilizing this same concept to similarly increase the use temperature of high temperature alloys. In our initial studies of this method of strengthening, we are incorporating a finely divided stable ceramic-aluminum oxide, into a nickel matrix. Here we can see the type of result that has been obtained to date. The specimen on the right is nickel, and the specimen on the left is nickel into which there has been incorporated the finely divided stable aluminum oxide. We are heating both of these specimens to 2000°F by an electric current and can observe the load carrying capacity. The water that enters these plastic tubes applied high dead weight load to the specimens and we can see that the specimen on the right is now beginning to sag, while the one on the left is maintaining the load. We will continue to load the specimen on the left until it sags or fails and you will notice that it was able to withstand approximately twice

the load of the pure nickel specimens. It should be noted that this is happening at a temperature where the conventional hardening particles that are normally used in a nickel base alloy have already dissolved into the matrix. With this technique we hope to increase the use temperature of nickel base alloys from their current level of about 1650°F to perhaps as high as 2100°F.

I have discussed one method of providing materials for use at higher temperatures. Mr. _____ will now discuss a second approach to this problem.

Mr. _____ has discussed one very promising method for increasing the temperature capabilities of certain high temperature alloys. However with these nickel and cobalt base materials, we are limited because we are approaching the melting temperature of the matrix -- for example the nickel base alloys are liquid at about 2600°F. To further advance the use temperature, we must go to matrix materials which have very high melting temperatures. These, of course, could again be improved by the type of stable dispersion that we have been discussing. Among the materials that we are considering as base metals for future alloys are columbium which melts at a temperature of 4500°F and tungsten which has a melting point of 6100°F. Alloys of these materials, especially those of tungsten, can be expected to be very strong at much higher than current temperatures.

I would like to illustrate the outstanding strength of one very high melting point metals. In this rack I have identical bars of an ordinary steel, a nickel base high temperature alloy and the high melting point metal, tungsten. From each bar is suspended an identical weight which loads it to 10,000 psi. The bars are heated by passing a current through them. By observing the dials above each bar we can note at which temperature each material fails. I'll place this screen in front of the tungsten to protect your eyes from the bright light. The steel fails at about 1600°F. The super alloy fails at about 2100°F, and we can observe that the tungsten fails at 3800°F.

We have recently developed an apparatus by which we can measure the strength of the high melting point metals at very high temperatures. This apparatus permits us to test small experimental size specimens in controlled atmospheres. The next chart shows our data on the strength of tungsten at these very high temperatures. For comparative purposes, we have included the materials we used in our last demonstration. Note the outstanding strength of the tungsten at these high temperatures. For example, at a stress of 10,000 psi, we can see that by using tungsten we achieve an improvement of about 1700°F over the nickel base alloys.

You may have noted that we ran our test with the tungsten enclosed in a glass tube. The purpose of this was so that we could remove the air thereby reducing the oxidation. If I had heated the specimen in air, the result would have been quite different. To illustrate this, I have a sheet of tungsten which I will heat to the same temperature by passing an electric current through it. We can see the voluminous clouds of oxide smoke as the specimen comes to temperature. This very high rate of oxidation that we just observed, of course, prohibits the use of tungsten in air atmospheres. Unfortunately, the lack of oxidation resistance is

characteristic of the entire class of high melting point metals.

We have been studying methods to reduce the tendency for oxidation of these metals and I would like to indicate some of our progress to date with a brief motion picture. One of the metals that we have been studying is columbium and in our tests we compare the oxidation rate of a small button of pure columbium to the oxidation rate of various columbium alloys. These buttons were placed in a furnace at 1500°F and time lapse photography was used to observe the oxidation. Thus you will see in approximately one minute the changes in the appearance of these specimens that actually occurred over a period of three hours. Note how the pure columbium is mushrooming in all directions as a result of oxidation. The specimen on the right contains 5% chromium and although a slight oxide does form on the surface after exposure to temperature, this oxide forms at a much slower rate. Measurements made during oxidation tests such as we are seeing indicate that the rate of oxidation of pure columbium is reduced by a factor of 10 by the addition of the 5% chromium. We are continuing our studies of the oxidation of this metal and although the rate is still not satisfactory, perhaps of greater importance is the fact that we have changed the character of the oxide. The oxide of the chromium alloy is now tight and adherent and should lend itself to further improvement.

The oxidation test that we have just seen was conducted in a normal air environment. If the materials are to be used in a nuclear powered aircraft or the nose cone of a missle, then at the same temperature the rate of oxidation would have been even more severe. This occurs because the nuclear radiation and the high temperatures associated with high mach number flight causes the air molecules in the atmosphere to dissociate. These atoms are in an unstable state and they want to recombine with each other to form more stable molecules. We can demonstrate the increased activity of dissociated oxygen in the apparatus shown here. Oxygen atoms can be generated in this discharge tube and pumped into the test section where they simulate conditions encountered in reactors and in missile re-entry. Here we have a strip of platinum connected to a source of electricity. The current is adjusted to cause the platinum to heat slightly, but this temperature could be withstood indefinitely. If we now turn on the discharge tube and cause the oxygen atoms to pass over the platinum, they recombine on the platinum strip. Since this recombination is a reversion to a more stable state, it releases heat. This heat raises the temperature of the platinum so that it glows much more brilliantly. The presence of the active atoms that we have just seen have several important effects. For example, our recent research indicates that metals oxidize about 400 times as fast in the presence of these atoms as they do in normal air. Furthermore, as we have seen, their recombination raises the temperature and further accelerates oxidation. Our research to date on the effect of dissociated gases has served to define the problems, and emphasize the need for additional studies to counteract these effects. We are continuing this research at this laboratory. A third approach to the problem of achieving high temperature materials will be discussed by Mr. _____.

The high-melting point ceramics also offer considerable promise for use at very high temperatures. Many ceramics have the necessary oxidation resistance and high melting point, but they also have the serious drawback that they are brittle. One important effect of this brittleness is that it frequently causes failure due to thermal stresses.

By the use of this polariscope, I will show you what a thermal stress is and how it acts. I have here a disk of a brittle ceramic with several holes in it. This disk might represent a ceramic fuel element for a nuclear rocket and the three small holes: cooling passages. In a nuclear reactor, the fuel element would be heated very non-uniformly. Let's see what happens when a non-uniform temperature distribution is imposed on such a disk. I will do this with a similar disk which has been placed over the electric coil on the end of this rod, and we can observe on the screen what is happening when the coil is heated. The heating coil appears in silhouette in the center. The inside of the disk is getting hot and wants to expand. The outside, on the other hand, is still cold and is restraining the center. The net effect is that we are imposing a thermal stress on the disk -- particularly in the vicinity of the cooling passage holes. The changes in color indicate the thermal stresses. If the disk were ductile, it would deform and absorb the stress. But the ceramic has no capacity for deformation and the stress builds up until it exceeds the strength of the material and the disk fractures.

The possibility of failure by thermal stress would be reduced if the ceramic could be made less brittle. The reason for the brittleness of ceramics is not completely understood, but research here as well as at other laboratories has provided a small start that may eventually lead to ductile ceramics.

The next chart indicates the kind of results we would like to achieve in a ceramic. At the left is a curve for an ordinary ceramic. On the vertical axis we have plotted applied load and resulting deflection is plotted on the horizontal axis. As the load is increased, deflection increases proportionally, and when a critical load is reached the specimen breaks. This is characteristic of brittle materials. On the right is the curve we would like to achieve. Beyond a critical load we would like the material not to break, but rather to have a large deflection. This added deflection is what we call ductility. A ductile material is not as susceptible to failure by thermal stress, as is a brittle one.

Recent research indicates that some ceramic materials may be inherently ductile and that one factor that tends to embrittle them is surface imperfections. Our approach has been to study the role of surface imperfections on the tendencies of ceramics to be brittle. Freshly-cleaved crystals of some materials are ductile. Their ductility is lost with time, presumably because imperfections are generated in the surface layers. We have found that suitable treatment of the surface layers restores the ductility. In the movie sequence to follow the effect of removal of the surface imperfections on a sodium chloride crystal by water polishing is demonstrated.

F I L M

Most of our research on this problem has been fundamental in nature and employed materials such as sodium chloride. Recently we have been applying the results of our fundamental studies to more practical materials. One of them is the high-melting-point ceramic magnesium oxide, which melts at a temperature of more than 5000°F and is of interest for nuclear applications. In the next film we will demonstrate the brittle fracture of ordinary magnesium oxide and the restoration of ductility due to surface treatment. You will see in the first sequence that the load-deflection curve is practically straight to fracture, as one would expect for a brittle material. In the second sequence, however, the curve bends off, indicating ductility. Watch closely; the first sequence is short.

F I L M

While the success in imparting at least a little ductility to ceramics has been very exciting from a scientific standpoint, it should be emphasized that our ceramics research to date has been on single-crystal specimens of limited size and represents only a start. Much work must be done before ceramics can be considered as practical engineering high-temperature materials.

Gentlemen: We have indicated to you the very high goals that have been established for materials research. This (morning) we have described three of the methods of attack that we are pursuing. These are: (1) the improvement of existing alloys by dispersions of stable high-melting-point particles, (2) the study of alloy systems based on the high-melting-point metals, such as tungsten and columbium, and (3) the study of the underlying reasons for the brittleness of ceramics. Only by successful research along these and similar lines can we hope to attain the high temperatures required for future aircraft.

HIGH TEMPERATURE MATERIALS
COMPARATIVE STRENGTHS OF
SEVERAL MATERIALS
TUNGSTEN
HIGH-TEMP
ALLOY
HUNDREDS OF °F

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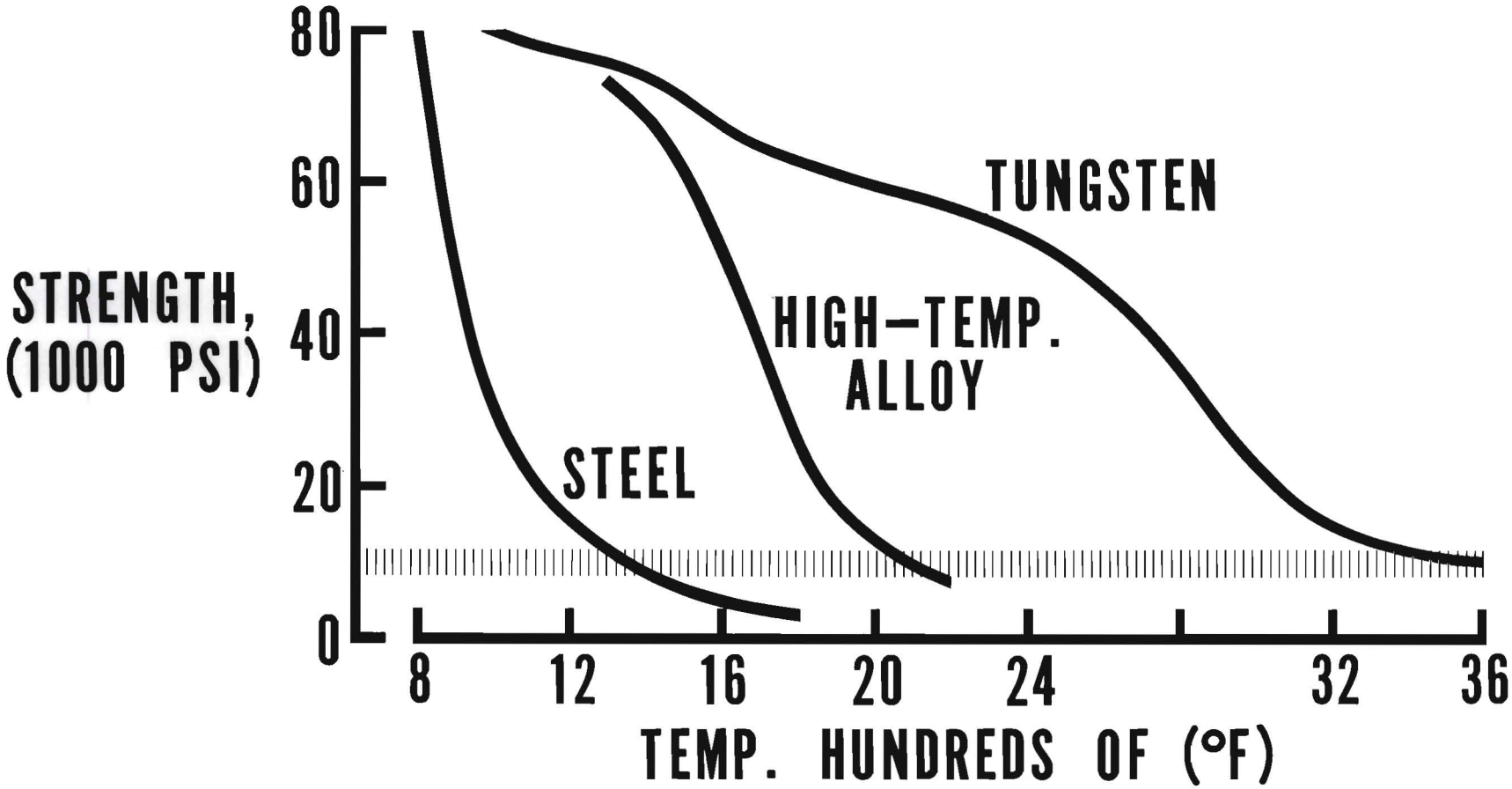
COMPARATIVE STRENGTHS OF SEVERAL MATERIALS



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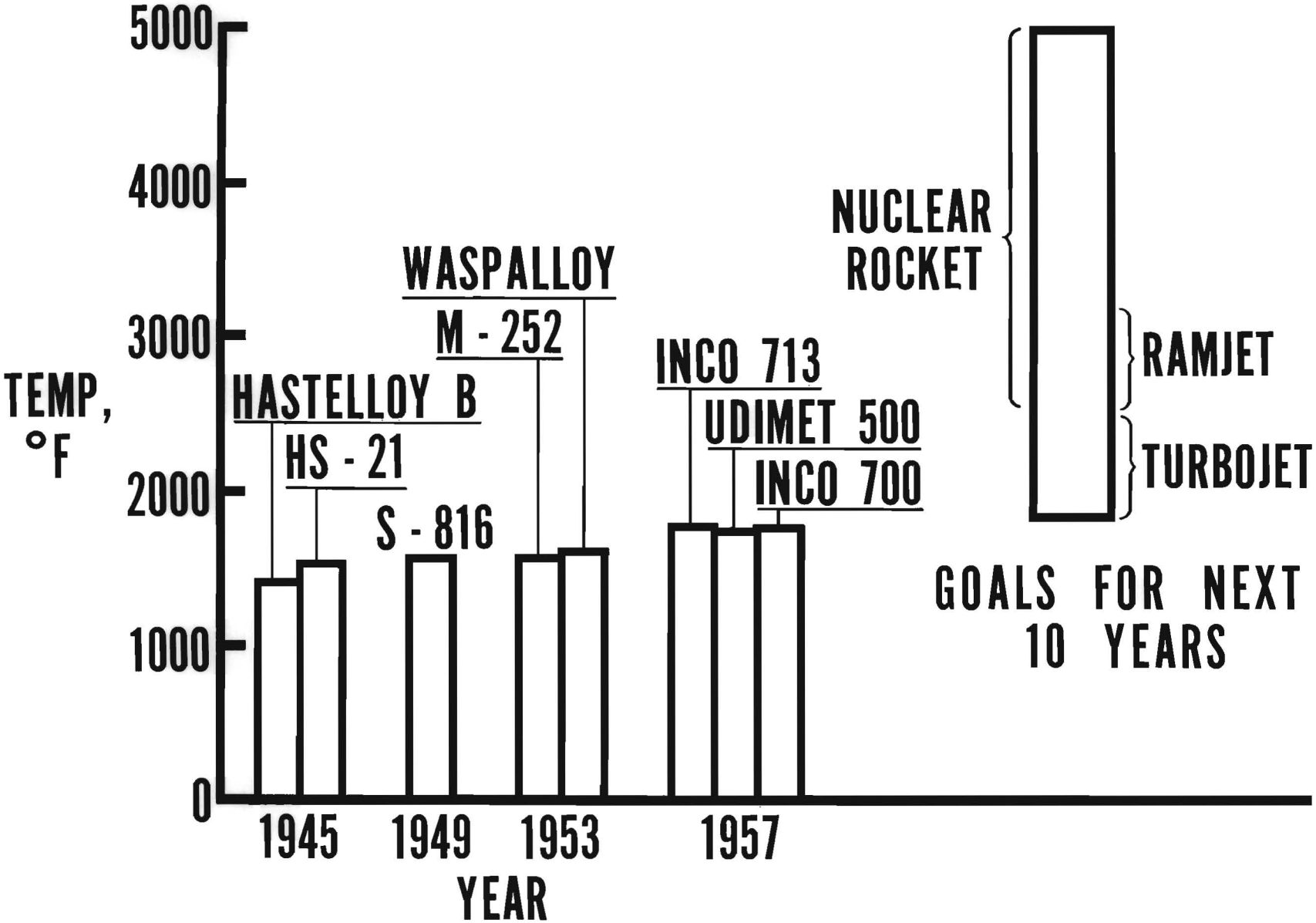
OPERATING TEMP. OF MATERIALS