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FIBER-METAL COMPOSITE MATERIALS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SUMMARY

Investigations of metal matrix materials reinforced with polycrystalline ceramic and metal fibers are reviewed. The results of parametric studies with model systems and of developmental studies with practical engineering materials are described, and an indication of the future potential of fiber-reinforced composites is presented. Model system studies have demonstrated the feasibility of strengthening metal matrices with continuous and discontinuous fibers. Fiber composite materials have been fabricated that follow a law-of-mixtures behavior at room and elevated temperatures for tensile and stress-rupture applications. The importance of fiber length-to-diameter ratios and orientation and of fiber-matrix bonding and interaction also have been indicated. Results achieved in preliminary attempts to produce engineering materials have been encouraging. The potential of fiber-reinforced metal composite materials is suggested by the excellent strength of materials currently available in fiber form. Increased fiber strength can be achieved, which adds further to the advantage of composite materials.

INTRODUCTION

Although stronger and better materials are always in the offing and will always be in demand, composite materials may offer the only hope of achieving certain space-age requirements. Not only are unusual properties expected from such fiber-reinforced products, but it is expected that, ultimately, these products may be made economically by mass production techniques. Design of composites that utilize unique or unusual mechanical and physical properties of fibers would appear desirable. Beyond this, however, a more intriguing possibility exists; namely, that composites may evolve that will have properties far better than would be expected from the individual properties of the con-

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stituents comprising the composites (a large synergistic effect). This report will consider such fundamentals as fracture mechanisms and strength relations. Some type of base line for comparison is needed to determine whether the properties of composites may equal or exceed the potential expected based on the properties of the fibers and matrix materials. Composite properties have been related to fiber and matrix properties by a law-of-mixtures equation, which may be considered as such a base line. Because it is possible that the mechanical properties of the composite may be related to many metallurgical factors other than the strength of the constituents, the law-of-mixtures relation should be considered a first approximation. Thus, deviations from the law of mixtures may be related to such factors as relative strain-hardening rates of matrices and fibers, moduli of elasticity, creep rates (for high-temperature conditions), interfiber spacing, orientation effects, stress concentrations, and contraction of materials during elongation (Poisson ratio effects in tensile applications). Some of the factors causing deviations will be discussed.

LAW-OF-MIXTURES BEHAVIOR

The law-of-mixtures relation was first shown to be valid for tensile strengths of metallic matrix systems by Jech, McDanels, and Weeton in references 1 and 2, wherein a copper-matrix was reinforced with tungsten fibers. Subsequently, the law-of-mixtures relation was extended and shown to relate the yield strengths and the moduli of elasticity to volume percentages of fibers (using tungsten fibers embedded in a copper matrix) in the composite for both continuous and discontinuous fibers by McDanels, Jech, and Weeton (refs. 3 and 4).

The composites studied consisted of tungsten fibers embedded in a copper matrix. This system was selected because copper and tungsten are mutually insoluble, because molten copper wets tungsten, because tungsten has a high recrystallization temperature, and because tungsten has a much greater strength and modulus than does copper. The case of continuous reinforcement, in which the fiber extends the full length of the test section, will be considered first.

Bundles of tungsten fibers were placed in a tube so that all the fibers were oriented in a
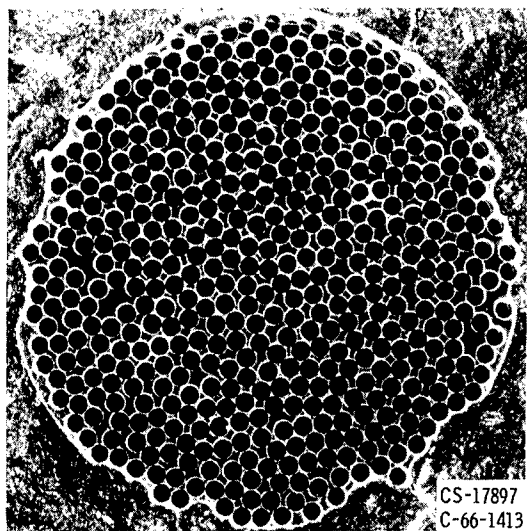

Figure 1. - Tungsten-reinforced copper composite containing 483 5-mil-diameter wires. Transverse section; unetched (ref. 3).
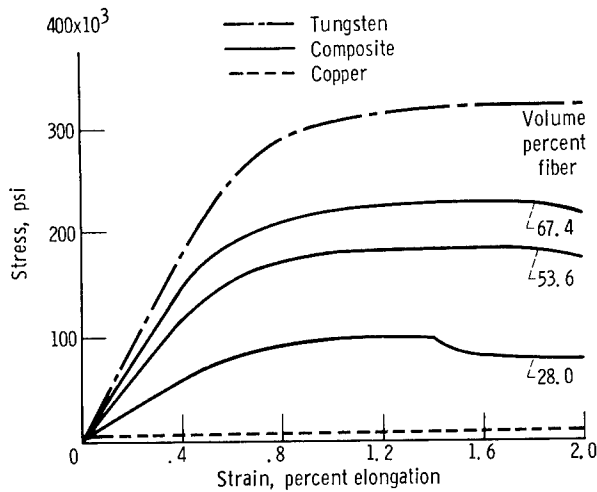
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Figure 2. - Stress-strain curves for tungsten wire, copper, and composites reinforced with continuous 5-mil-diameter tungsten wire (ref. 3).
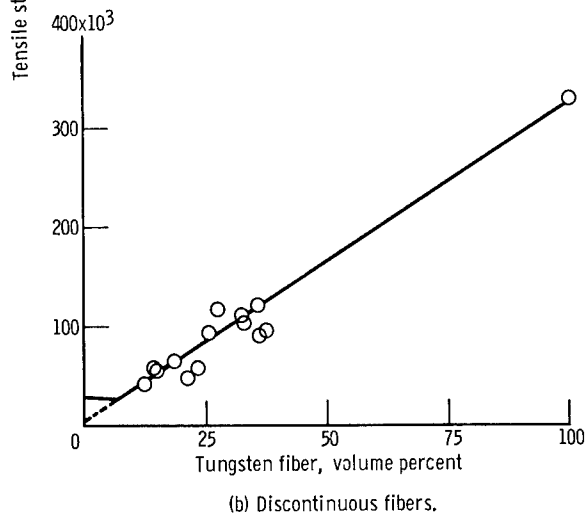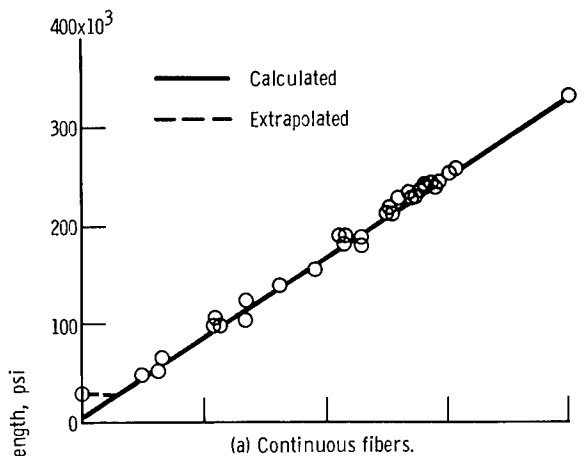

(a) Continuous fibers.

(b) Discontinuous fibers.

Figure 3. - Ultimate tensile strengths of tungsten-fiber-reinforced copper composites. Diameter of tungsten fibers, 5 mils (ref. 3).

direction parallel to the tensile axis. The wires were infiltrated with molten copper. The cross section of a typical specimen is shown in figure 1. The strength of such composites was found to be represented by

$$\sigma_c = \sigma_f A_f + \sigma_m^* A_m \qquad (1)$$

where

σ ultimate tensile strength

A   area fraction or volume fraction when unity length is considered and

$A_f + A_m = A_c = 1$

σ_m^* stress on matrix, taken from stress-strain curve, at equivalent strain to that at which ultimate tensile strength of fiber is achieved

and the subscripts are

c   composite

f   fiber

m   matrix

Figure 2 shows some typical stress-strain curves for tungsten, copper, and tungsten-fiber-reinforced copper composites. It may be seen that the copper carries only a minor stress of 8 000 pounds per square inch compared with the tungsten fiber, which carries 330 000 pounds per square inch. Figure 3(a) is a plot of ultimate tensile strength against volume percent of tungsten fiber for 5-mil tungsten-fiber-reinforced copper composites. Similar

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curves were obtained for 3- and 7-mil-diameter wires, and these data are discussed in more detail in references 3 and 4. The straight line of figure 3(a) extends from the tensile strengths of the tungsten fibers (thermally treated to duplicate the treatment given the fibers by the infiltration process used) to the stress carried by the copper matrix. It should be noted, however, that the copper stress used is the stress on the matrix, taken from the stress-strain curve, at an equivalent strain to that at which the ultimate tensile strength of the fiber was achieved. The dashed portion of the curve at the left indicates the range of volume percent fiber content over which the fiber does not contribute to the tensile strength of the composite. The fiber breaks before the maximum load of the composite is reached. The intersection of the dashed line and the solid line may be termed the critical volume percent and is defined as the lowest volume percent fiber content where fibers break as the maximum load-carrying capacity of the composite is reached. Thus, the tensile-strength - law-of-mixtures relation does not apply to composites containing volume percentages of fibers below the critical percentages. Another point could be made from figure 3(a), namely, that some of the composite materials are very strong. For example, at 50-volume-percent fibers, the tensile strength of the composite is over 150 000 pounds per square inch. At 75 percent, the strength is over 250 000 pounds per square inch.

Figure 3(b) is the same curve obtained previously (fig. 3(a)) both experimentally and by calculation (eq. (1)) for the continuous fibers. However, the data points of figure 3(b) represent ultimate tensile strengths obtained from composites reinforced with discontinuous fibers. In these composites, fibers were cut into 3/8-inch lengths. They were oriented in the direction of the specimen axis and infiltrated in the same manner as was done for the full-length fibers. Composite specimens containing a maximum of 41-volume-percent fibers were made by this method. The data points fell fairly close to the previously obtained curve. This, of course, was very encouraging, because it permitted a prediction that discontinuous or short-length fibers, such as whiskers or other strong materials, could be embedded in the matrix materials and could be expected to contribute significantly to the strength of the composite. In fact, for many future practical composites, strengths obtainable for specimens with relatively short fibers are expected to be within experimental accuracy of those obtained for continuous fibers.

Figure 4 shows schematically the four stages of stress-strain behavior of the composites during tensile testing. Evidence that such behavior occurred in the tungsten-fiber - copper-matrix materials is presented in reference 3. For very low percentages of strain, both the fiber and the matrix were elastic (stage I). As the material was strained further, the fiber was still elastic, and the matrix became plastic (stage II). With further elongation, the fibers became plastic, and the matrix remained plastic (stage III). At some point the fibers began to break (stage IV). The fact that composite curves did not fall off in strength very rapidly indicated that the segments of the fibers
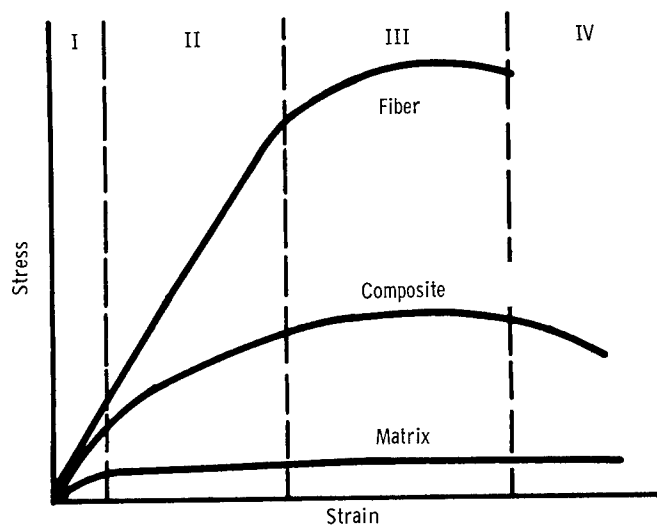
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Figure 4. - Schematic illustration of four stages of deformation of fiber, matrix, and composite. Stage I, elastic deformation of both fiber and matrix; stage II, elastic deformation of fiber, plastic deformation of matrix; stage III, plastic deformation of both fiber and matrix; stage IV, failure of both fibers and matrix (successive failure of fibers).
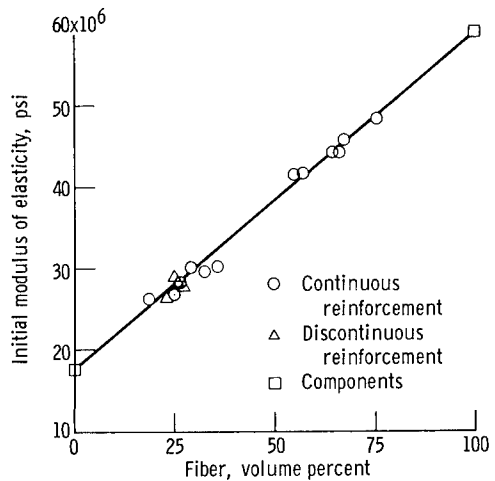

Figure 5. - Initial modulus of elasticity of tungsten, copper, and composites reinforced with continuous and discontinuous 5-mil-diameter tungsten fibers (ref. 3).
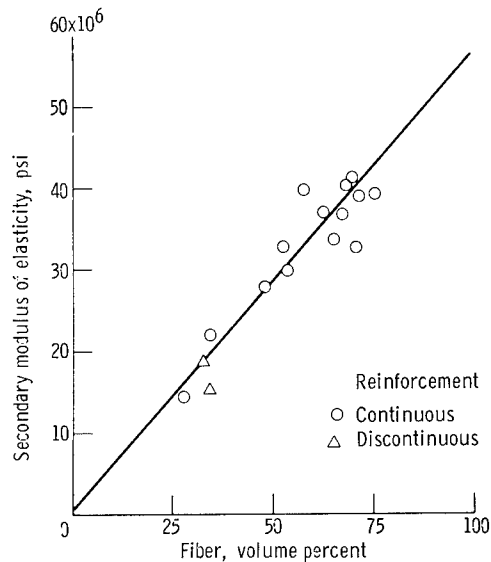

Figure 6. - Secondary modulus of elasticity of composites reinforced with continuous and discontinuous 5-mil-diameter tungsten fibers (ref. 3).

that had fractured were still retained and bonded to the matrix, and that they reinforced the material by acting as short-length fibers.

Some of the experimental work, which indicated that the composites had four stages of behavior, as shown in figure 4, is presented in figures 5 to 7. Figure 5 shows the dynamic modulus of elasticity obtained by sonic techniques. The results represent stage I behavior, the initial modulus of elasticity of the composites reinforced with either continuous or discontinuous fibers. The linear relation between the modulus of the composite and the volume fraction of the fibers is a law-of-mixtures relation.

In considering stage II behavior, it is necessary to examine the stress-strain curves of the composites in stage II and their component phases at strains larger than those representing only the elastic behavior of the least elastic component. It is shown in reference 3 that the copper stress-strain curve deviates from proportionality at about 0.03-percent strain. Above this strain, copper deforms plastically and exhibits an almost flat or horizontal stress-strain curve. The slopes of the stress-strain curves of composites were shown to decrease from the first linear slope in this strain region of stress-strain curves to the second, almost linear, region at somewhat higher strains. A linear secondary slope would not be expected for a composite with a matrix that did not exhibit a linear stress-strain curve in the region of plastic strain of the matrix. This portion of the curve in the schematic diagram (fig. 4) is termed secondary modulus of elasticity. Again, as was the case for the initial modulus curve, the secondary modulus of elasticity obeyed a linear relation with fiber content (fig. 6). At 0-volume-percent fibers, the curve extrapolated to a value equal to the approximate slope of the plastic portion of the stress-strain curve of copper. At 100-percent fiber content, the curve extrapolated to a value equal to the modulus of elasticity of tungsten. This indicated that the matrix was plastic and that the fiber was elastic in stage II.

As the composite was strained further, the fiber also became plastic (stage III). Figure 7 shows the yield strength obtained from the stage III portion of the stress-strain curves of the composites. The yield stress
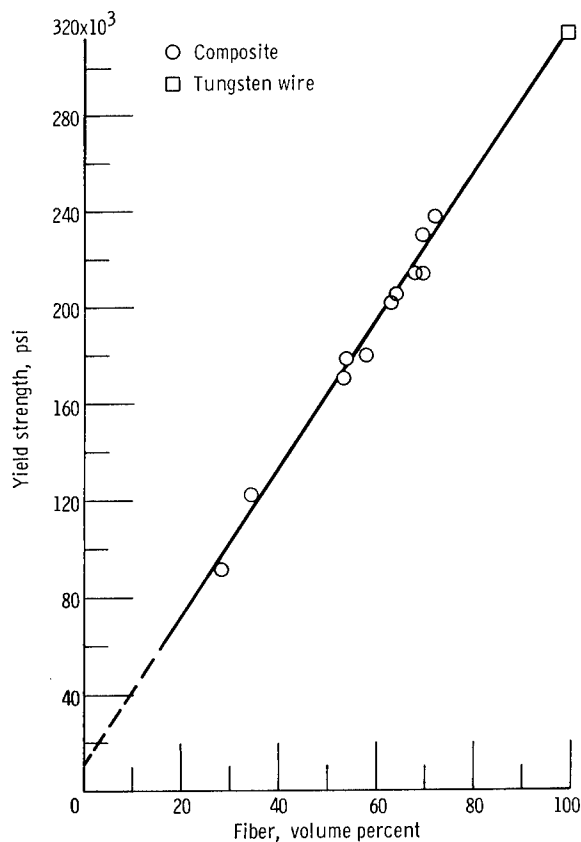

Figure 7. - Yield strength (based on secondary modulus) of composites reinforced with continuous 5-mil-diameter tungsten fibers (ref. 4).
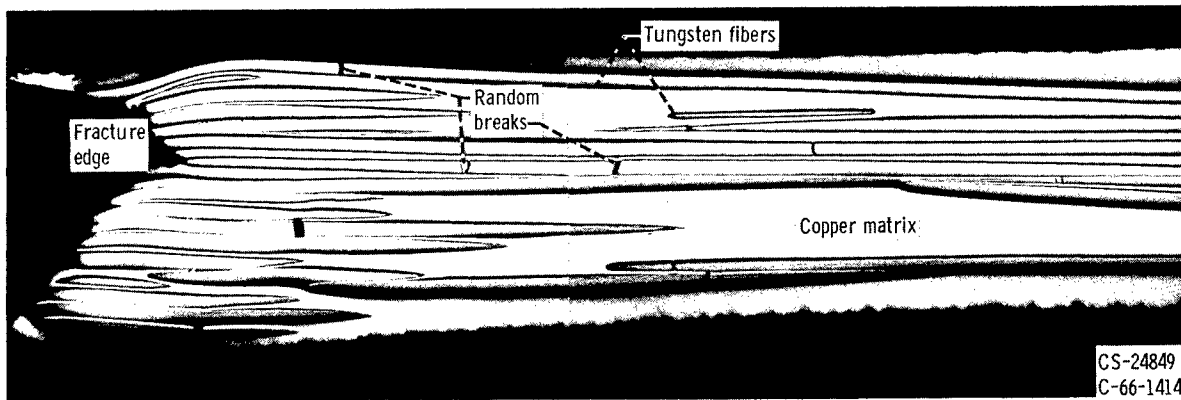
Figure 8. – Longitudinal section through failed composite showing fracture edge and neckdown of fibers. Continuous 3-mil-diameter tungsten fibers; copper matrix (ref. 3). X50. (Reduced 50 percent in printing.)

at 0.2-percent offset was determined by drawing a line parallel to that portion of the curve where the fibers were elastic and the matrix was plastic (stage II portion of curves). Again, the data points fall on a straight line, and the curve extends from the yield strength of the copper to the yield strength of the tungsten. Thus, yield strengths also obey a law-of-mixtures relation.

Inspection of the stress-strain curves of the composite specimens of figure 2 (p. 3) shows that, with continuing strain, the composites reach their ultimate strength. After the attainment of the ultimate strength, some fibers fracture within the composite (stage IV). The fracturing of all the fibers in a composite would not be expected to occur simultaneously; with additional strain of the composite, other fibers fracture at random locations along their length. Figure 8 shows breaks that formed in fibers some distance away from the main fracture. Most fractures would occur after the ultimate strength of the composite was obtained but before the final fracture of the specimen occurred. The segments of the fractured fibers retained their bond with the matrix and acted as short-length (discontinuous) fibers reinforcing the composite. The latter is believed to be the case because either the composite stress-strain curves were very flat or they tapered off in strength slowly, relative to the stress-strain curves for the tungsten fibers alone; this effect indicated that the reinforcement was continued by the fiber segments. Elongation of the composites was greater than that of the fibers tested individually.

FIBER-MATRIX LOAD TRANSFER CONSIDERATIONS

In discontinuous specimens, load is transferred from fiber to fiber by shear through the matrix. The area of matrix fiber interface available for effective load transfer with discontinuous fibers is reduced compared with that of continuous fibers. Therefore, care
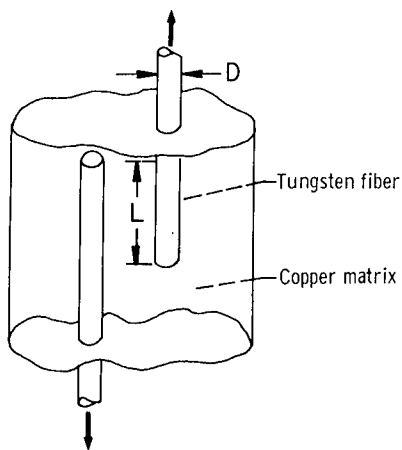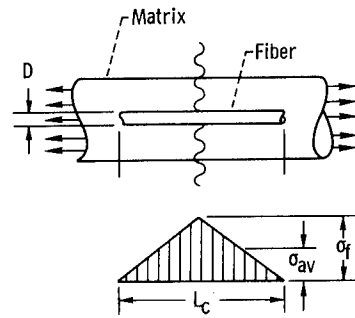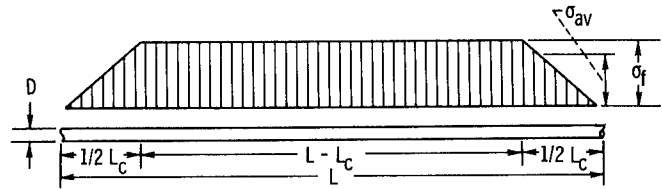
Figure 9. - Schematic diagram of shear load transfer mechanism in fiber-reinforced composites reinforced with discontinuous fibers. Length L corresponds to value of L in equation (2) (refs. 1 and 3).

(a) Fiber of critical length and stress level in fiber along length. ($L_c/D = 1/2(\sigma_f/\tau)$.)

(b) Fiber much greater than critical length and stress level along length. ($L \gg L_c$.)

Figure 10. - Schematic illustration of stresses in critical length of fiber in matrix and effect of increasing length on average fiber stress.

must be taken to prevent shear failure of the matrix or matrix-fiber interface rather than tensile fracture of fibers. The shear transfer of loads has been described for metallic composites (refs. 1 to 4), and a schematic model is shown in figure 9. The discontinuous fibers in the composite are alined parallel to each other and to the tensile axis. By using this model, the value of the length-to-diameter ratio or the aspect ratio necessary to utilize fiber strength fully was found to be represented by

$$\frac{L'}{D} = \frac{1}{4}\frac{\sigma_f}{\tau} \tag{2}$$

where

L   length of fiber overlap needed to transmit stress to cause tensile failure of fiber

D   diameter of fiber

σ_f tensile strength of fiber

τ shear strength of fiber-matrix interfacial bond or shear strength of matrix, whichever is less

When the entire fiber is embedded in the matrix, the required fiber length would be twice the value of L given in equation (2), which is

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$$\frac{L_C}{D} = \frac{1}{2}\frac{\sigma_f}{\tau} \tag{3}$$

where L_C is the total fiber length, that is, the minimum fiber length needed to permit the fiber to contribute its tensile strength to the matrix by shear. The other terms are the same as those noted for equation (2). (See fig. 10 for a schematic illustration.) Figure 10(a) shows a schematic illustration of the tensile stress distribution along a fiber of critical length embedded in the matrix. This is the shortest length fiber that permits a stress buildup with the fiber to cause tensile fracture. The average stress on a fiber of critical length is illustrated in figure 10(b) and is equal to one-half of the stress-carrying capacity of the fiber material. This, of course, assumes that the buildup of stress from the end of the fiber to the center is linear. The bottom portion of the figure shows a fiber that is much greater than the critical length and illustrates how this affects the average stress-carrying capacity of the fiber. Again, the stress is assumed to build up linearly from each end to the value to cause tensile fracture of the fiber, but a greater proportion of the fiber can carry the full tensile load. Thus, the average stress on the fiber is considerably higher than the average stress on a fiber of critical length and, in fact, may approach the average stress of the continuous fiber.

Kelly and Tyson (ref. 5) derived a modified law-of-mixtures equation that showed the relation between strength of discontinuous fiber composites and fiber lengths. It should be remembered that the fiber length also bears a relation to the aspect ratio, and thus, the following equation shows the importance of length and/or aspect ratio:

$$\sigma_c = \sigma_f V_f \left(1 - \frac{1}{2\alpha}\right) + \sigma'_m (1 - V_f) \tag{4}$$

where

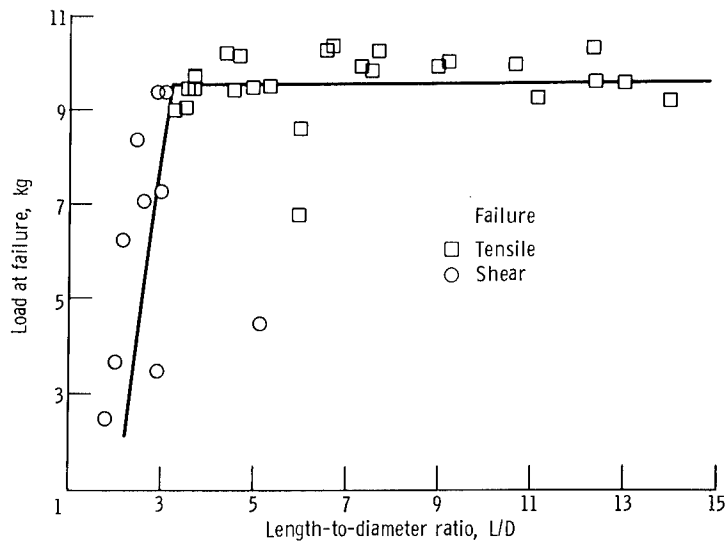|	
σ	tensile strength
V_f	volume fraction of fibers or same as A_f of eq. (1)
α	actual fiber length/critical fiber length, L/L_c
σ'	same as that for σ^* of eq. (1)
$1 - V_f$	V_m, volume fraction of matrix or A_m of eq. (1)
L	actual fiber length
L_c	fiber length needed to permit fiber to contribute its tensile strength to composite, that is, critical fiber length

Figure 11. - Experimentally determined length-to-diameter ratios for tungsten fibers in copper. Pullout load method; room temperature.
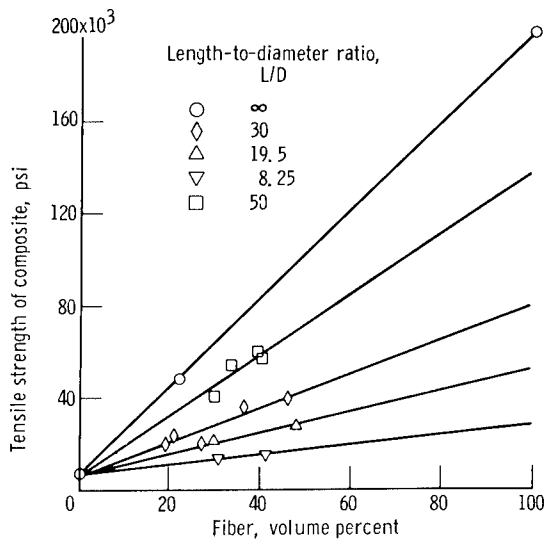

Figure 12. - Ultimate tensile strengths of tungsten-fiber - copper-matrix composites as function of volume fraction of fibers and length-to-diameter ratios. Temperature, 1212° F, (600° C); 0.008-inch-diameter fibers (ref. 5).

Equation (4) indicates that discontinuous fibers will not contribute 100 percent of their strength to the composite. On the other hand, with lengths many times the critical length (large aspect ratios), the values calculated are effectively the equivalent of values obtained with continuous fibers (eq. (1)). For example, with a length-to-critical-length value of 10, a total of 95 percent of fiber strength would be contributed; with a length-to-critical-length value of 100, 99.5 percent of the fiber strength could be utilized. It is believed that it will prove relatively easy to produce composites with fibers with length-to-critical-length values such that better than 90 percent of the fiber strengths will be usable.

Values of the critical length or length-to-diameter ratio were found experimentally by determining the maximum length of a fiber that can be pulled out of the matrix. Such a procedure would seem to give a very direct method for determination of the critical length-to-diameter load. Unpublished results of such an experiment by R. W. Jech at Lewis Research Center are shown in figure 11. Here, the stress required either to break the wire or to pull it from copper is plotted as a function of the length of the portion embedded in the matrix divided by the diameter. The point at which the curve of

figure 11 becomes horizontal is one-half of the critical length-to-diameter ratio, since this is a point at which the shear load of the matrix or the interface is equal to the tensile load of the fiber. When this length is exceeded, the fiber fractures.

Kelly and Tyson (ref. 5) obtained curves such as those shown in figure 12 for specimens tested at different temperatures and for different sizes of wires. The critical length-to-diameter ratios were calculated using the slopes of such curves and the length-to-diameter ratios. These ratios showed not only the effects of increasing temperature on the critical length-to-diameter ratio but also the effects of temperature on the shear strength of the matrix at the fiber-matrix interface. The latter was possible with the utilization of equation (3) and the experimentally obtained critical length-to-diameter ratios. The calculated shear strengths decreased with increasing temperature, and the need for the utilization of larger length-to-diameter ratios at elevated temperatures was made evident. The fiber tensile strength and the matrix shear strength both decreased with increasing temperature, but the copper shear strength decreased at a much higher rate. This would be expected, since the copper is at a much higher fraction of its melting point. Thus, matrix strength plays the predominant role in determination of required length-to-diameter values for the tungsten fiber - copper composites. These composites may be representative of high-melting-point fibers embedded in low-melting-point matrices.

Cratchley (ref. 6) also showed some of the effects of differing aspect ratios on the strength of composites. Figure 13 is a plot of the tensile strength against volume percent of composites containing fibers of different length-to-diameter ratios. With an aspect ratio of 830, the extrapolated strength of the fiber was approximately equal to the known strength of the fiber as determined individually, which would indicate that this material was essentially obeying a law-of-mixtures relation. Cratchley also observed that decreasing fiber diameter increased the efficiency of reinforcement for a given fiber length.

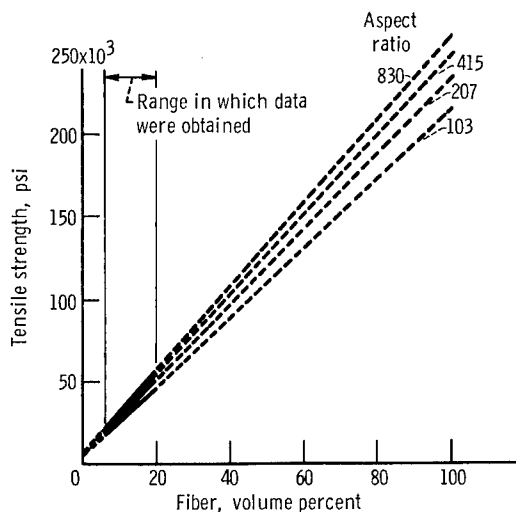Figure 13. - Effect of aspect ratio on strength-composition relation for stainless steel fiber-reinforced aluminum composites. Discontinuous 2-mil-diameter fibers (ref. 6).

DEVIATIONS FROM LAW-OF-MIXTURES

BEHAVIOR

Some of the factors or phenomena that could conceivably produce synergistic effects in a composite (in this case, properties greater than those predictable from law-of-mixtures calculations)

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are the following: alloying reactions at the fiber-matrix interface, surface strengthening of the fiber within the matrix by healing or elimination of surface defects, size effects, crack interruption, dissipation of stresses at the crack apex by a ductile matrix, blockage of cracks by strong fibers, orientation of grains at surfaces of fibers, orientation of grains throughout fibers, restraint and constraint of fiber deformation by the matrix, restraint of the matrix by fibers, thermal expansion effects, corrosion prevention by the matrix, and probably numerous other effects.

Very few data have been published that indicate that synergistic effects may be obtained by combining fibers in metallic matrices. An example of possible improvements in composite strengths above law-of-mixtures values has been published by Piehler (ref. 7). The composites made by Piehler consisted of steel fibers (0.8-percent carbon wire) embedded in silver. Full-length fibers, either 7 or 19 per cross section, plated with varying thicknesses of silver, were packed together in a hexagonal stacking pattern inside a silver tube and consolidated by a combined mechanical working and heat treating procedure. Figure 14 shows the tensile strength of composites made by this method on a strength-against-volume-percent basis. It may be seen that the tensile strength of the composites falls above the line drawn between the tensile strength of pure silver and the tensile strength of fibers. The author attributed the higher strength of the composites relative to the law-of-mixtures line to restraint factors, in particular to the restraining of localized necking of fibers by the matrix.

It should be mentioned, however, that it is possible that metallurgical factors other than mechanical restraint could have accounted for the apparent increase in properties above the law-of-mixtures values. It is felt that there is a possibility that the materials used to represent the matrix and fiber strengths were not representative of their counterparts within the composite.

One of the major considerations relative to the successful fabrication and use of composites is the compatibility of materials. It has been mentioned that alloying and other metallurgical reactions between fibers and matrix materials within composites during fabrication and use may improve properties of constituents within the composites and concurrently cause the composite as a whole to be improved above expected values. Unfortunately, the converse possibility also exists, namely, that reactions between materials can be deleterious.

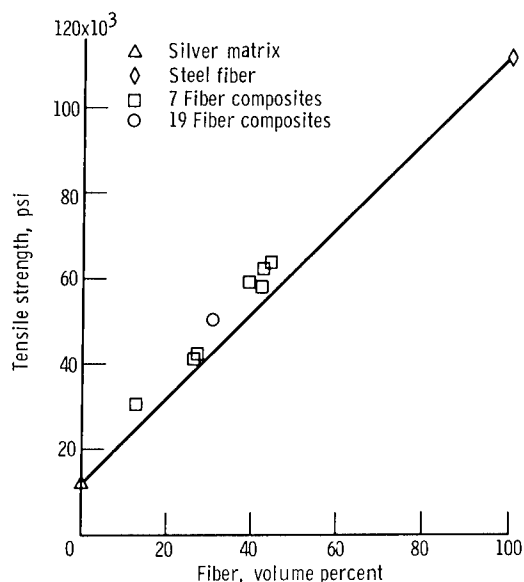Between the extreme possible effects noted

Figure 14. - Ultimate tensile strengths of steel fiber-reinforced silver composites (ref. 7).

previously, that is, either large synergistic reactions or catastrophic damage, there may be varying degrees of improvement or damage to composite properties. For example, where mutually insoluble materials are combined, there may be no reactions or loss or gain of strength resulting from the combinations. Even where alloying effects or reactions take place, the reactions may be controllable and resulting composites may have highly desirable properties.

At this time, no known evidence has been presented in the literature to indicate that the synergistic effects noted previously have been obtained from alloying reactions occurring in composites. As for deleterious effects, relatively few metal-fiber - metal-matrix systems are known that would permit the creation of composites consisting of nonreactive materials, such as mutually insoluble constituents. Ceramic-fiber - metal systems would, in many cases, be expected to be relatively nonreactive. However, it is known that, under some conditions, even the most thermodynamically stable oxides may dissolve in metal matrices. In whisker-bearing composites, too, reactivity between whiskers and metal matrices could also prove deleterious.

Further, it is almost a certainty that some of the most intriguing combinations of materials (from a potential strength standpoint) will prove most difficult to combine without harmful reactions. Rather than attempt to predict such problems, investigations that have already been completed will be described.

The first investigation concerned with compatability problems was conducted at the Lewis Research Center. The principal objective was to obtain an understanding of some of the effects of alloying on the tensile properties of composites (ref. 8). The initial work described earlier was conducted with the tungsten-fiber - copper-matrix system, and these materials are mutually insoluble (nonreactive). Alloying elements with varying degrees of solubility in tungsten were added to copper, and the resulting alloys were infiltrated about bundles of tungsten fibers. The infiltration conditions were the same as those used for the previous work (refs. 3 and 4). Room-temperature tensile tests were made of the composites, and a metallographic study of the microstructure of the fiber - metal-matrix interface was conducted. The results were compared with those obtained in references 3 and 4, in which copper was used as the matrix. Three types of reaction were found to occur at the fiber-matrix interfaces within the composites. The reactions that occurred are believed to account for the properties of the materials presented in the figures. The reactions were as follows:

  (1) Diffusion-penetration reaction accompanied by a recrystallization of the grains at the periphery of the tungsten fibers
  (2) Precipitation of a second phase near the periphery of the fiber with no accompanying recrystallization of the fiber
  (3) Solid solution reaction with no accompanying recrystallization in the fiber
  The microstructures of cross sections of some of the composites studied are shown
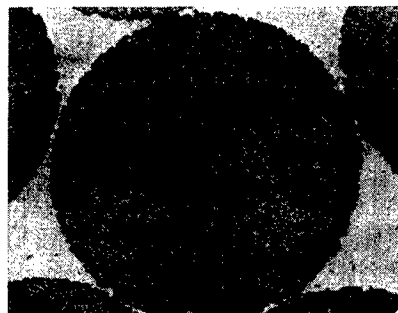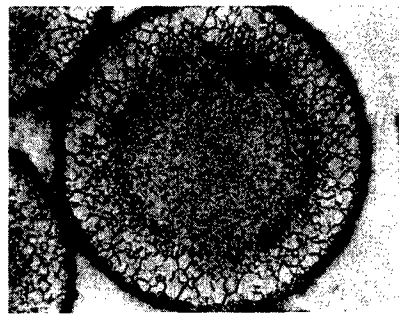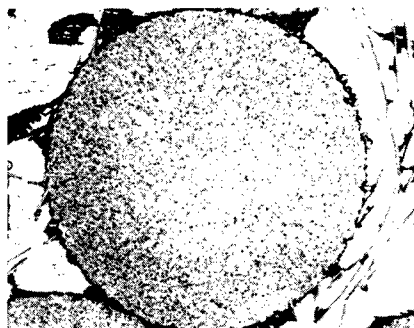
(a) Copper – 5-percent-nickel matrix.
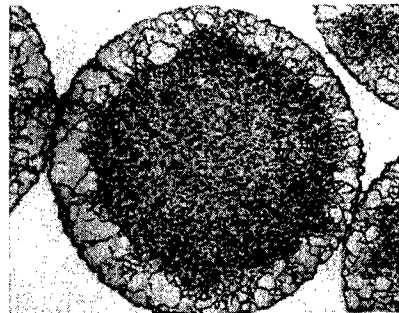
(b) Copper – 10-percent-nickel matrix.

(c) Copper – 1-percent-cobalt matrix.

(d) Copper – 5-percent-cobalt matrix.

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(e) Copper – 10-percent-zirconium matrix.

Figure 15. – Tungsten fibers in copper alloy matrix. Transverse section; as infiltrated (ref. 8). X750. (Reduced 50 percent in printing.)

in figure 15. The 5-percent-nickel addition to copper did not cause the formation of a visible reaction recrystallization zone at the fiber-matrix interface (fig. 15(a)). The microstructure of this specimen is identical in appearance to one that would be obtained if pure copper were the matrix. The 10-percent addition of nickel, however, formed a large reaction recrystallization zone at the periphery of the fiber (fig. 15(b)).

The tensile strengths of some of the composites investigated are plotted against the volume percent fiber content in figure 16. It can be seen that the tensile strengths of the alloyed composites were all lower than those obtained for copper composites in which
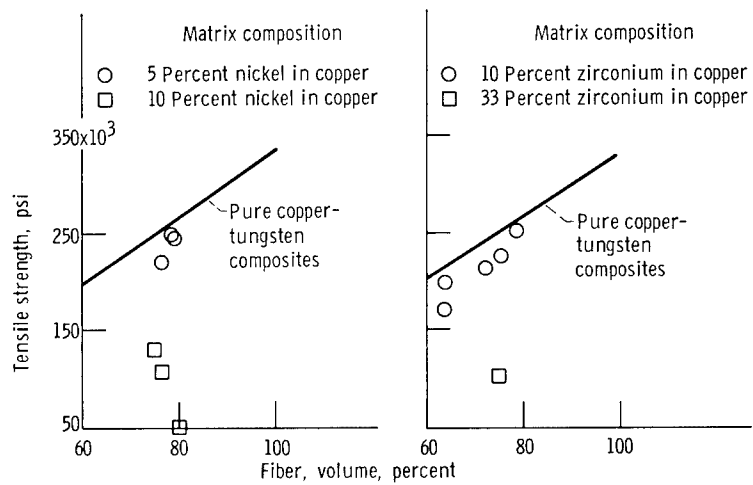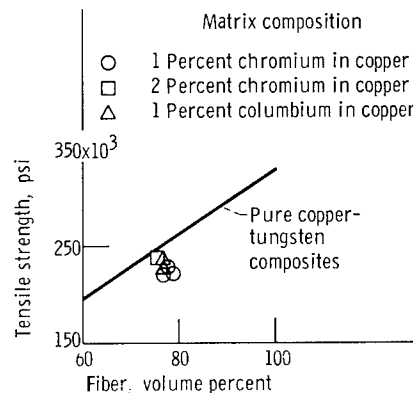
Figure 16. - Strength-composition diagrams comparing tungsten-fiber-reinforced copper composites to tungsten-fiber-reinforced copper alloy composites (ref. 8).

alloying with the fiber did not occur. With 5 percent nickel added to the copper, very little damage was done to the strengths of the composites (fig. 16(a)). With 10 percent nickel, a considerable reduction in tensile strength was observed. Thus, the percent of alloying additives to a matrix material may produce drastic differences in the properties of the composites. The most damaging type of reaction observed was the diffusion-penetration reaction accompanied by recrystallization of the fiber, which occurred with the copper-nickel and copper-cobalt matrix composites.

Figure 16(b) also shows the effects of adding 10 percent zirconium to the matrix on the tensile strength of composites. There is some reduction in the strength of the material, but again as was the case for 5-percent-nickel additives, the damage was relatively slight. In the case of the data point representing the 33-percent-zirconium addition, there was a significant lowering of the tensile strength of the composite relative to the

tensile strength of the pure copper-tungsten composites. This was attributed to the brittle nature of the matrix itself rather than to any alloying reaction between the matrix additive (zirconium) and the tungsten fiber.

Figure 16(c) shows a strength-composition diagram for copper-tungsten composites to which small percentages of chromium or columbium were added. These elements form solid solutions with tungsten, and it was not possible to add larger percentages of these materials without raising the melting point above 2200° F. All the data points for these materials fall very close to the line obtained for the pure tungsten-copper composites.

Tensile strengths of the copper - 5-percent-nickel composites were much higher than those obtained for the copper - 10-percent-nickel composites and were only slightly lower than strengths of the copper composites. The lower tensile strength values for the copper - 10-percent-nickel composites were attributed to the reaction zone formed with the fiber. As the depth of penetration or the area of the reaction zone increased, the tensile strength of the copper - 10-percent-nickel composites decreased.

Additions of as little as 1 percent cobalt to copper recrystallized the tungsten fiber. The composites also had tensile strengths much lower than that obtained for the pure copper matrix composites. It was also found that, as the depth of penetration of the alloying element into the tungsten fiber increased, the tensile strength and ductility decreased. It was shown for the copper - 5-percent-cobalt system that low tensile strength and poor ductility were due not only to the contribution of the alloy zone formed with the fiber, but also to the brittle nature of this zone and its effect on the unalloyed portion of the fiber. Since recrystallized tungsten wire is extremely brittle at room temperature, the re-crystallized zone of the fiber was believed to act as a brittle skin. It was felt that the recrystallized zone fractured or cracked early in the tensile test and that the crack acted as a circumferential notch around the fiber. Since tungsten is known to be notch
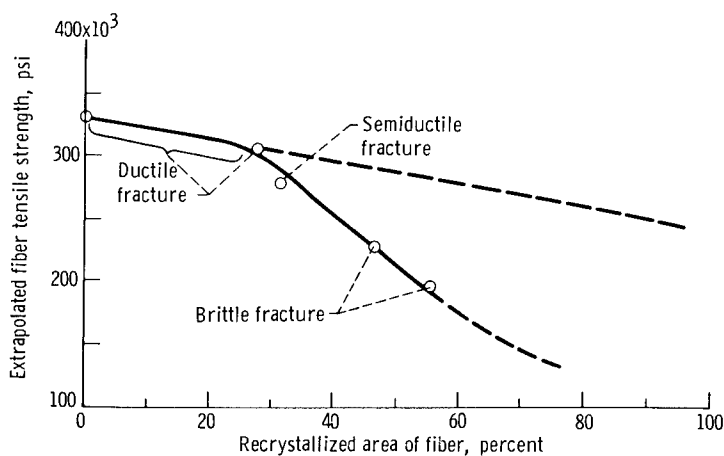
Figure 17. - Fiber tensile strength as function of percent recrystallized area of fiber. Matrix of copper plus 5-percent cobalt (ref. 8).

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sensitive, the formation of a notch by the brittle alloy zone would cause premature failure of the unalloyed portion of the fiber. Fibers that are notch sensitive might thus be readily damaged by alloying reactions at the interface between the fiber and the matrix. These postulates were made with the aid of the plot shown in figure 17. The graph gives values for fiber tensile strengths (for fibers embedded in a copper - 5-percent-cobalt alloy matrix) obtained from graphical extrapolations of composite data as a function of the recrystallized area of the fiber. Relative brittleness of the various composite fractures is indicated in figure 17 also. As the percentage of the recrystallized fiber zone increased (i. e., as depth of recrystallization increased), the fiber strength decreased. More important though, at a given point (about 30-percent recrystallization), the properties fell off at a rate greater than that which would be expected if the damage to the strength of the fiber were in direct proportion to the area of recrystallization. This, coupled with the change in ductility of the fractures, indicated that the fiber core that was not recrystallized was being notch sensitized during the tensile test, as was noted previously.

In addition, since many metallic fibers gain a considerable portion of their strength from mechanical deformation processes or from heat treatments, both of which impart considerable strain energy to the material, it would not be surprising if, during incorporation of such fibers into a matrix, properties of the fibers were reduced. A reduction in properties could result from thermal or mechanical treatments used to fabricate the composites as well as by alloying reactions. It has been found (ref. 8) that very highly worked fibers would not necessarily be severely damaged by alloying reactions. Although all of the alloying additions made to the matrix lowered the strengths of the composites somewhat, some alloying elements did only superficial damage to the fibers at temperatures at which other elements did severe damage to the fibers.

HIGH-TEMPERATURE STRENGTH OF FIBER COMPOSITES

Most of the research and development programs relating to fiber-reinforced metal composites have been concerned with short-time low-temperature properties. The elevated-temperature potential of fiber-reinforced metal composites, however, is also of great interest.

Tensile Strengths of Composites at Elevated Temperatures

An understanding of the strengthening mechanisms for fiber reinforcement at elevated temperatures was felt to be a requisite to permit the design of high-temperature compos-
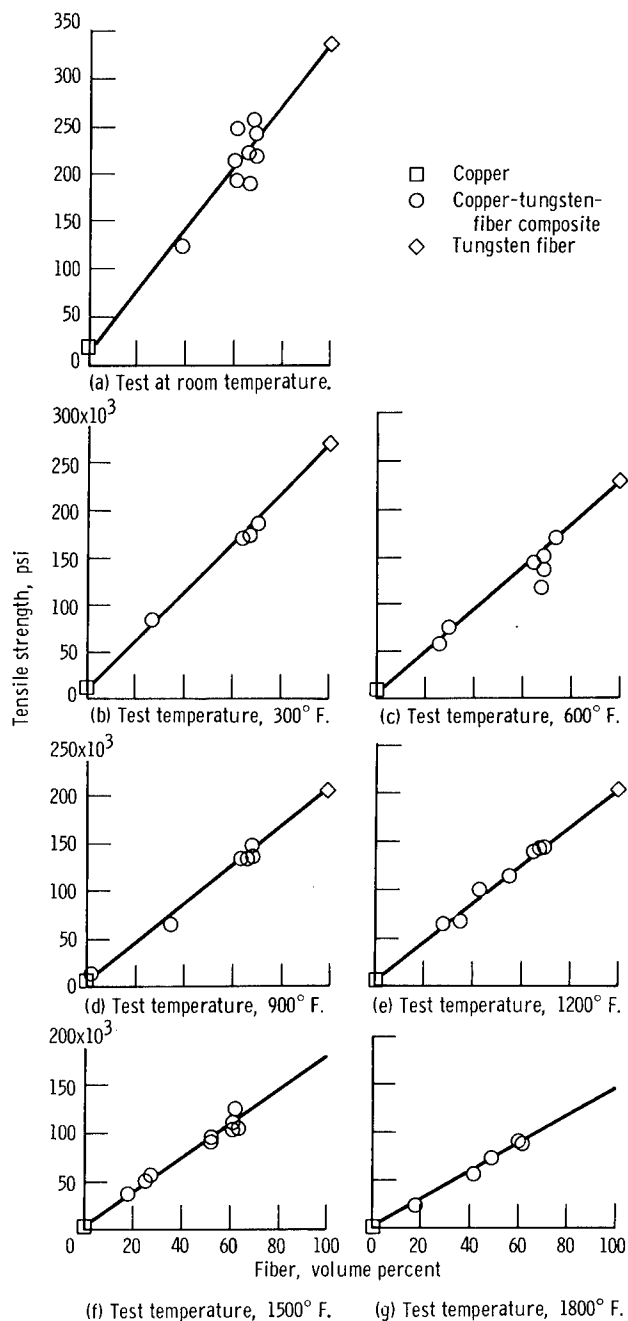
Figure 18. - Tensile-strength - composition diagrams for tungsten-fiber-reinforced copper composites as function of temperature. Continuous tungsten fibers (ref. 9).

ites. To gain an insight into the high-temperature behavior of materials, a tungsten-fiber - copper-matrix model system (mutually insoluble materials) was again used as a basis for comparison (ref. 9). Fibers extended the full length of the specimens and were uniaxially oriented. In addition, tungsten fibers were embedded in matrices of copper - 2-percent-chromium and copper - 10-percent-nickel alloys. Tensile tests were run at temperatures ranging from room temperature to 1800° F. A previous study (ref. 8) had indicated that chromium, which formed a solid solution at the tungsten-matrix interface, did not reduce room-temperature tensile properties significantly. Nickel in the copper-base matrix, on the other hand, severely damaged the fibers by causing recrystallization. A comparison of the properties at elevated temperatures with previously obtained room-temperature results would permit not only a comparison of behavior of a mutually insoluble materials system but systems with damaging and nondamaging reactions.

Elevated- and room-temperature tensile test results for the materials tested at temperatures of 300°, 600°, 900°, 1200°, 1500°, and 1800° F are presented in figures 18 to 20. Figure 18, which presents data for tungsten fibers in a pure copper matrix, indicates that, at least for temperatures up to 1200° F, the law-of-mixtures relation has been observed. Individual fiber strengths were difficult to obtain accu-

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Figure 19. - Tensile-strength - composition diagrams for tungsten-fiber-reinforced copper alloy composites as function of temperature. Continuous tungsten fibers in copper - 2-percent-chromium alloy (ref. 9).

rately at temperatures above 1200° F because of difficulties in controlling the test apparatus atmosphere. Therefore, no data are shown for the fiber strengths at 1500° and 1800° F. Presumably, the straight line of the figures would extrapolate to the fiber strengths at these temperatures also.

Figure 19 gives test results for both tungsten-fiber-reinforced copper (dashed line) and the tungsten-fiber-reinforced-copper - 2-percent-chromium alloy (solid line). The curves for the specimens tested at room temperature with the alloy matrix are almost identical with the curve for the pure copper-tungsten composite. At elevated temperatures too, the composite strengths fall almost on the law-of-mixtures curve for the pure copper-tungsten composite. The fiber strengths that are given in figure 19 are obtained from specimens digested from the copper-chromium matrix. The diameters of these fibers increased slightly as a result of the diffusion of chromium into the fiber and the formation of a chromium-rich layer at the fiber periphery. Dissolving the matrix did no noticeable damage to the fibers that were embedded in the copper-chromium alloy matrix.

A contrasting picture from that noted for chromium was obtained with the tungsten fibers in the copper - 10-percent-nickel matrix (fig. 20). First, at room temperature, as was noted in the earlier studies, the strengths of the copper-nickel-matrix -
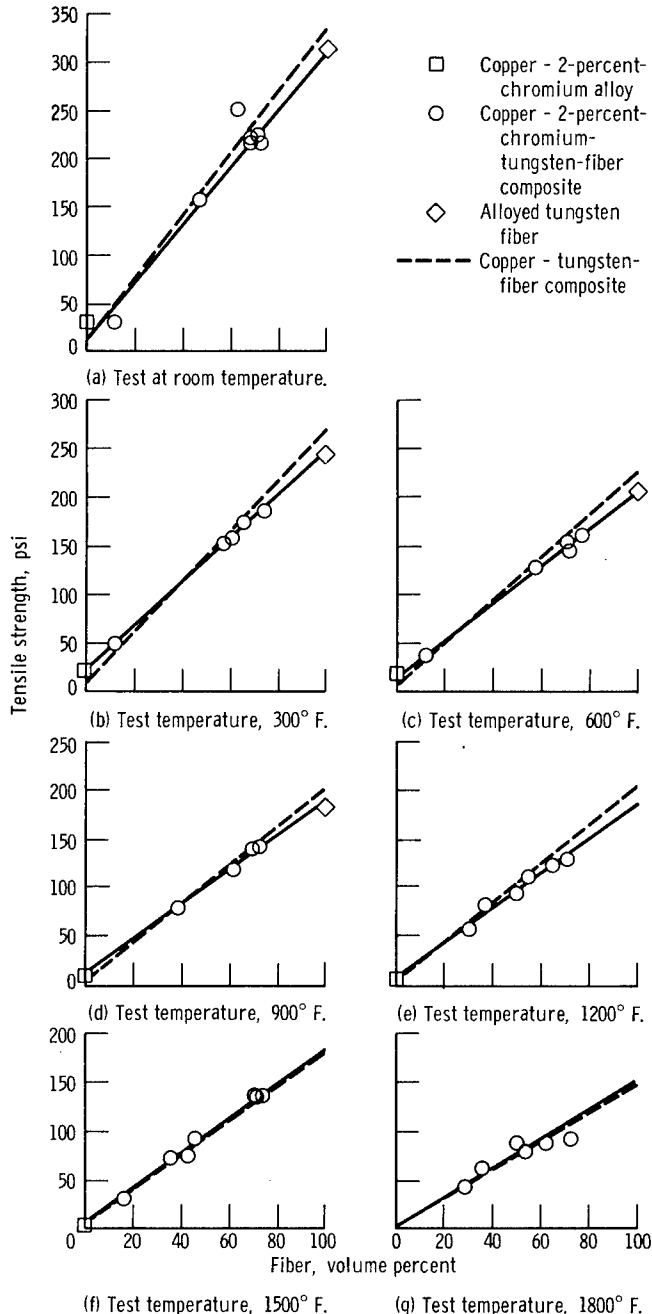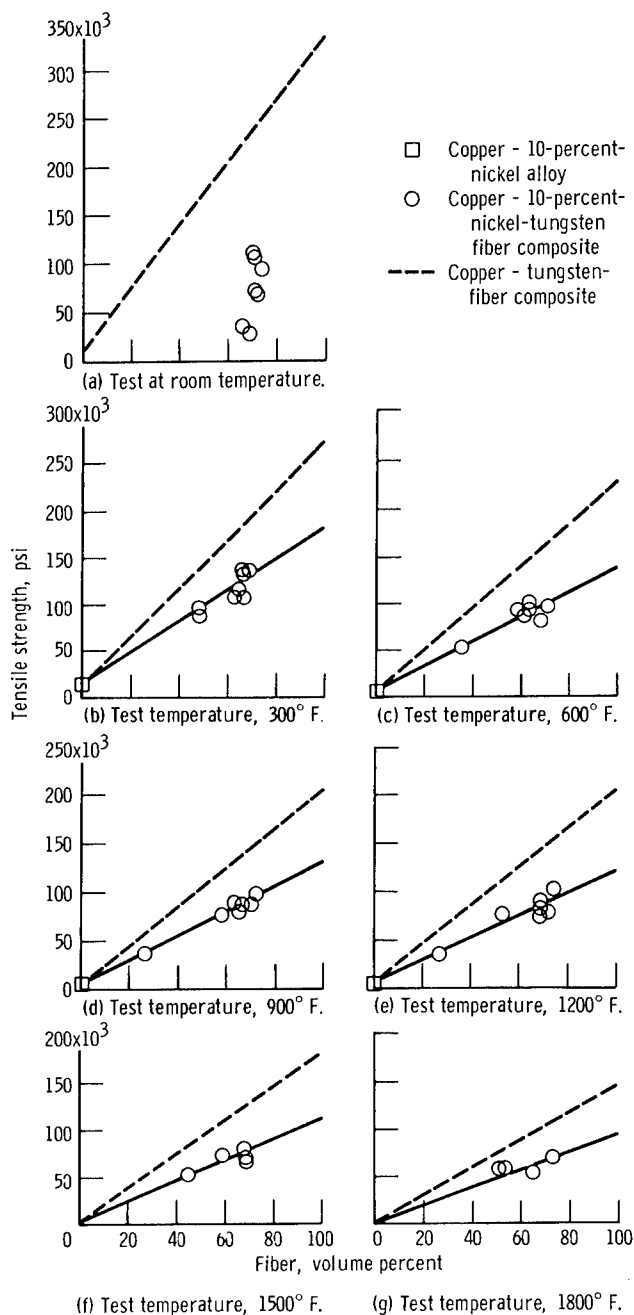
Figure 20. - Tensile-strength - composition diagrams for tungsten-fiber-reinforced copper alloy composites as function of temperature. Continuous tungsten fibers in copper - 10-percent-nickel alloy matrices (ref. 9).

tungsten-fiber composites were much lower than the strengths of the pure copper-tungsten composites. However, when the temperature was raised to 300° F, the tensile strengths of the composites were increased relative to the room-temperature strengths. Presumably, the test temperatures above 300° F were above the brittle-to-ductile transition temperature of the fibers. Unlike the situation that existed for the chromium-bearing matrix composites, the copper-nickel-tungsten fiber composites were appreciably weaker than the copper-tungsten fiber composites at all test temperatures. Attempts to obtain the strength of the tungsten fibers (with the recrystallized zone resulting from the nickel) by dissolving the matrix and extracting and testing the fibers failed. The fibers were so brittle that they failed in handling. Extrapolation of the composite strength against volume-percent fiber curves would be expected to give the strength of the matrix and of the fibers.

Figure 21 summarizes the strengths against test temperatures for fiber specimens and for 70-percent-fiber composite specimens of copper-tungsten, copper - 2 percent chromium tungsten, and copper - 10 percent nickel tungsten.

Orientation and Length-to-Diameter Effects at Elevated Temperatures

The importance of the shear strength of the matrix has previously been shown for discontinuous fiber composites tested at room temperature. Since the strength of the matrix may decrease rapidly with increasing temperature, the need to provide adequate shear length of the fiber-matrix interface becomes more acute as the temperature is raised. The drastically increased fiber length necessary at elevated temperatures to prevent pullout of fibers from the matrix may be seen in unpublished data of R. W. Jech presented in figure 22. The figure also shows the minimum length-to-diameter ratio (L/D) necessary to avoid pullout of a tungsten fiber from a copper matrix for a range of temperatures.

Misalinement of fibers is a further source of concern with discontinuous-fiber-reinforced composites, particularly in those cases where the matrix is weak. Effects of fiber length and orientation on the elevated-temperature tensile behavior of discontinuous-tungsten-fiber-reinforced copper composites are being studied at the Lewis Research Center (unpublished work of D. W. Petrasek, R. A. Signorelli, and J. W. Weeton). The effects of decreasing
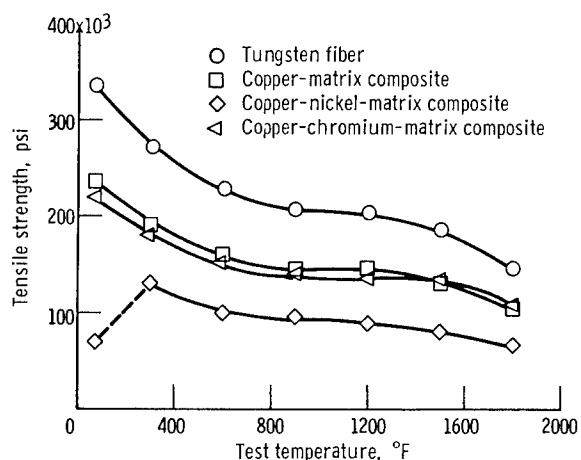
Figure 21. - Tensile strength as function of test temperature for composites containing 70-volume-percent fibers and for individual constituents. Continuous tungsten fibers (ref. 9).
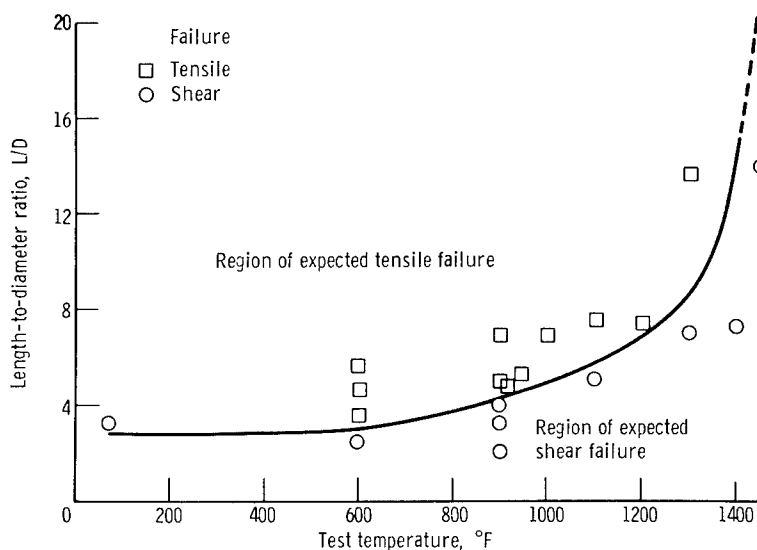
Figure 22. - Experimentally determined length-to-diameter ratios as function of temperature for tungsten fibers in copper.
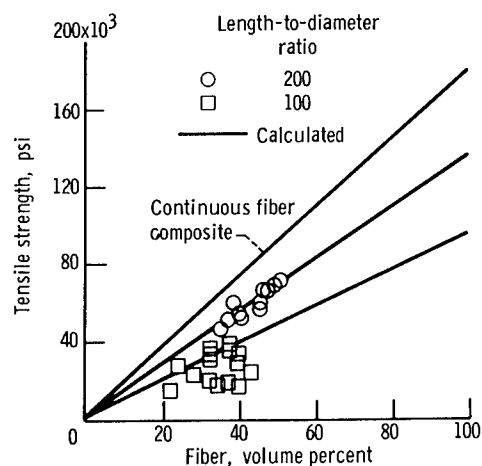
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Figure 23. - Tensile strength at 1500° F as function of volume percent fibers and fiber length for tungsten-fiber-reinforced copper composites.
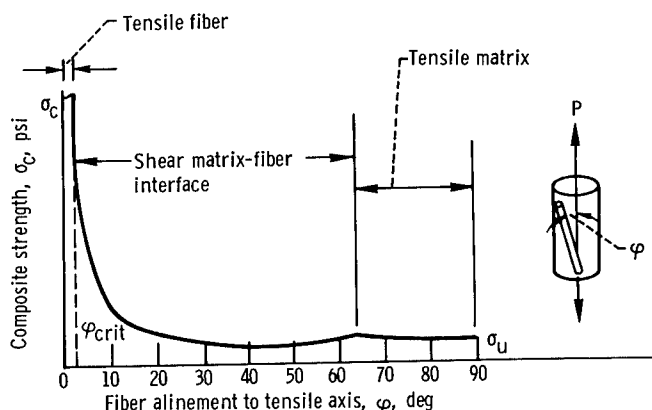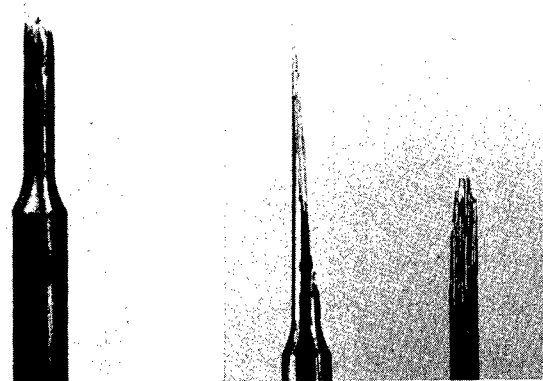

(a) Tensile failure $(\sigma = \sigma_c / \cos^2\varphi)$.

(b) Shear failure $(\sigma_c = \tau/\sin \varphi \cos \varphi)$.

Figure 24. - Test specimens that failed in tension or shear.

Figure 25. - Failure mode as function of alinement of fibers in discontinuous fiber-reinforced composite.

the fiber length on tensile properties that have been determined to date are shown in figure 23. Plots of tensile strengths for continuous and discontinuous fibers with an L/D of 200 and 100 are shown. The curves are calculated by using equation (4), which assumes perfect axial alinement of fibers. Comparison of the curves shows an expected decrease in efficiency of reinforcement with decreasing length of fiber. In the case of the specimens with an L/D of 200, the data points fall fairly close to the calculated curve. In the specimens containing fibers with an L/D of 100, there is a considerable scatter of data with several points well below the calculated line. It is believed that the lower strengths resulted from an increased misalinement that might be expected with shorter fibers. A correlation of the low strengths with misalinement was obtained by an examination of the failed specimens. Figure 24 shows a specimen with alinement adequate enough to cause tensile failure of the specimen. Also shown is a specimen where misalinement caused a shear failure of the matrix.

Figure 25 is a schematic showing how the failure stress on a composite may vary with the orientation of the fibers. It also illustrates the failure modes that would be expected for composites with different orientations of fibers. This figure was constructed from the equations of Stowell and Liu (ref. 10), which were also presented by Kelly and

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Davies (ref. 11). For a small misalinement of the fibers from the axial direction, the tensile strength increases slightly. This increase results from the increased cross-sectional area of the fiber relative to the specimen axis, when the fiber is slightly mis-alined. The elliptical area of the fibers in the cross section of the composite specimens would be slightly greater than the circular area of perfectly alined fibers. Also, it must be assumed that the matrix has a sufficiently high shear strength to cause tensile failure of the fibers within the composite. Further misorientation leads to shear failure of the matrix, which is indicated by the sharp decrease in the curve with increasing misorientation. It would be expected that, at 45°, the resolved shear stress would be a maximum and the composite strength would be a minimum. A specimen failured by shear is shown in figure 24. As the angle is increased above 45°, there is a tendency to stress the matrix alone in tension rather than to shear, which would cause tensile failure of the matrix.

Stress-Rupture Strength

There are almost no data available for creep or stress-rupture properties of fibers. Paradoxically, some of the most immediate foreseeable applications of fiber-reinforced materials are for aerospace turbine materials with attractive high-temperature creep-rupture properties, rather than for high-temperature high-tensile-strength materials.

To gain an insight into the stress-rupture behavior of fiber reinforced composites, it was felt that the strengthening mechanisms associated with the reinforcement of metallic matrices by fibers for creep-rupture conditions should be studied. The program at the Lewis Research Center was expanded to include an investigation of stress-rupture behavior of tungsten-reinforced copper composites at 1500° F (0.8 of the melting point of copper). Fibers were continuous and uniaxially oriented, and tests were run in purified helium.

Figure 26 shows some cross plots of stress-rupture data obtained recently for fibers and fiber composites (unpublished work
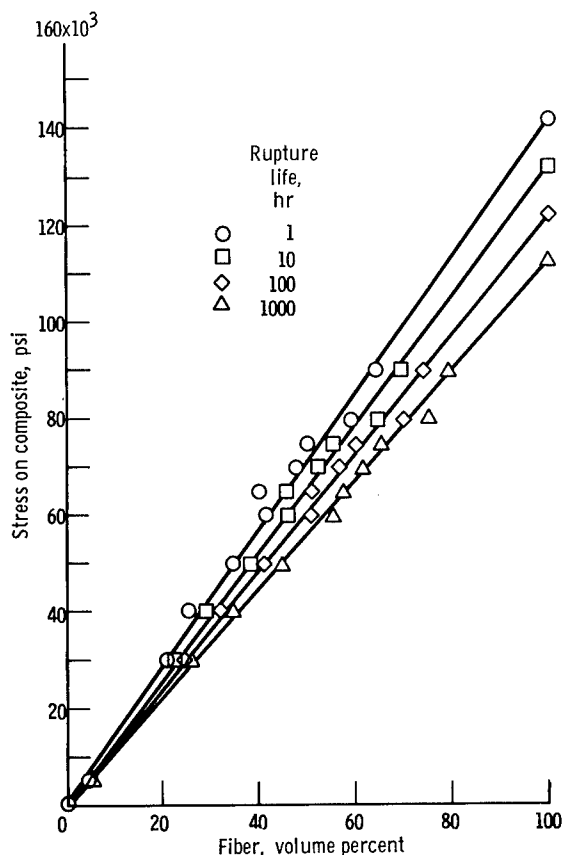

Figure 26. - Stress-rupture strengths for tungsten-fiber-reinforced copper composites as function of volume percent fibers. Continuous 5-mil-diameter tungsten fibers; test temperature, 1500° F; helium atmosphere.

of D. L. McDanels, R. A. Signorelli, and J. W. Weeton). The solid portions of the curves in this figure are plots representing the volume percent of fibers studied and the stress and rupture time data obtained to date. Data were initially obtained for given composite stresses and were related to volume-percent fiber and rupture time. From such plots, the stresses for rupture in 1, 10, 100, and 1000 hours were obtained as a function of fiber content. The data points for 100 percent fibers were obtained from actual stress-rupture tests of tungsten wire (ref. 12). It was very encouraging to note that the law-of-mixtures relation applied over the entire portion of the curves (up to volume percentages of about 70 to 75). As a first approach, the curves indicate that for a wide composition range, law-of-mixtures plots may be utilized to predict the strengths of composite materials in stress rupture.

DEVELOPMENT OF FIBER-REINFORCED ENGINEERING MATERIALS

The research described previously in this report has been concerned with approaches of a relatively fundamental nature. Model systems have been discussed, and strengthening mechanisms have been presented. In most of the work that has been done, material combinations were selected to avoid or minimize reaction between fiber and matrix materials.

With the best technical knowledge available, it is possible to consider the production of a composite material that will have useful properties. Some of the attempts made to produce fiber-reinforced composites utilizing a developmental approach, as well as examples of some of the problems that might be encountered in combining materials for engineering composites, will be presented in this section.

Typical problems that may be encountered are illustrated in figures 27 and 28. Figure 27 shows a microstructure of some tungsten fibers embedded in a columbium-nickel
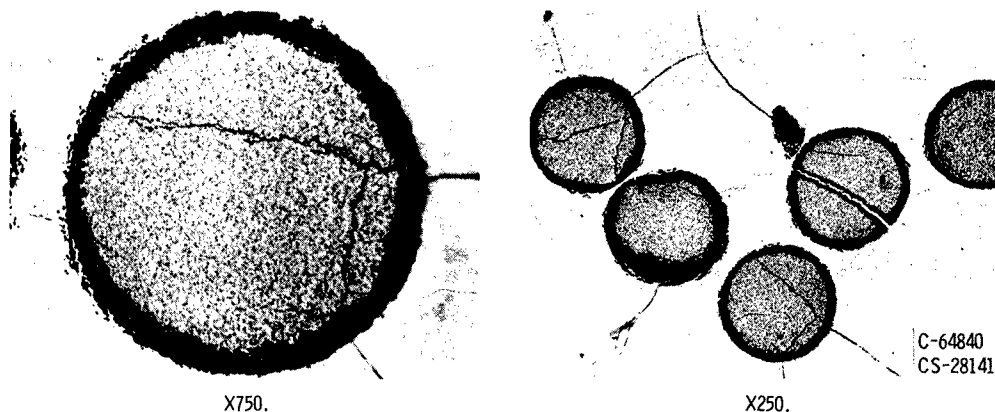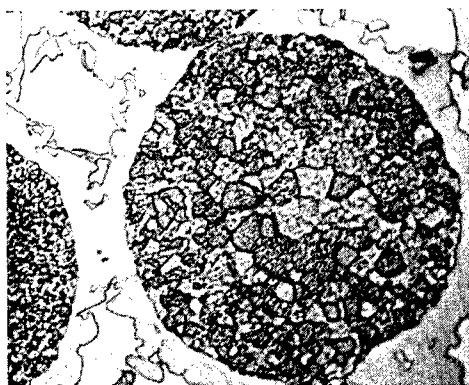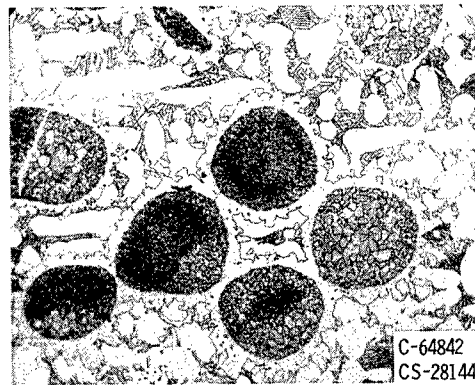

X750. X250.

Figure 27. - Tungsten fibers in columbium-nickel matrix. Transverse section; as infiltrated (ref. 8). (Reduced 50 percent in printing.)

Figure 28. - Tungsten fibers in cobalt base alloy (S-816) matrix. Transverse section; as infiltrated (ref. 8). (Reduced 50 percent in printing.)
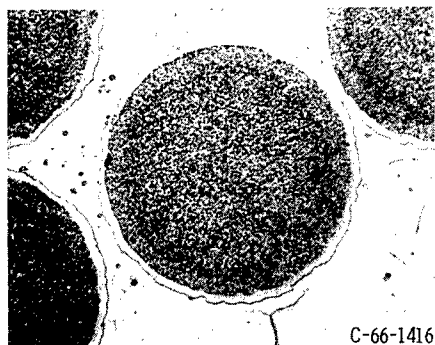

Figure 29. - Tungsten-fiber-reinforced nickel composite showing slight reaction zone as result of solid-state sintering.

matrix. The reaction that occurred at the periphery of the fiber apparently had not recrystallized the fiber (ref. 8). In this case, however, there appears to have been stress cracking in the matrix and a penetration of these cracks into the fibers. In fact, the stresses were so great that in one case (photograph on the right) the fiber itself was completely severed. Figure 28 shows a cross-sectional microstructure of some tungsten fibers embedded in a cobalt-alloy matrix (S-816) (ref. 8). The liquid matrix that was used in an attempt to infiltrate the fibers partially dissolved the fiber and recrystallized the undissolved portion of the fiber. Furthermore, the matrix became highly alloyed with the constituents of the fiber. To minimize such reactivity between the fiber and the matrix, various corrective steps might be taken. For example, where a fairly long time (1 hr) was used for the infiltration, the time could be reduced. Another step might be to form a diffusion barrier of some sort about the periphery of the fiber prior to infiltration. Still another method of avoiding the reaction is illustrated in figure 29, which is from unpublished work of D. W. Petrasek. Tungsten fibers were embedded in powdered nickel, and the consolidation process was accomplished by solid-state sintering of the composite. It should be recalled that small percentages of nickel in a liquid infiltrant had previously done considerable damage to the periphery of tungsten fibers. However, the solid-state sintering of tungsten fibers in a pure nickel matrix caused very little reaction to occur at the periphery of the fiber at the same temperatures that were used for the liquid infiltration studies. In fact, no recrystallization of the fiber was noted.

Solid-state sintering has also been utilized successfully by other investigators.
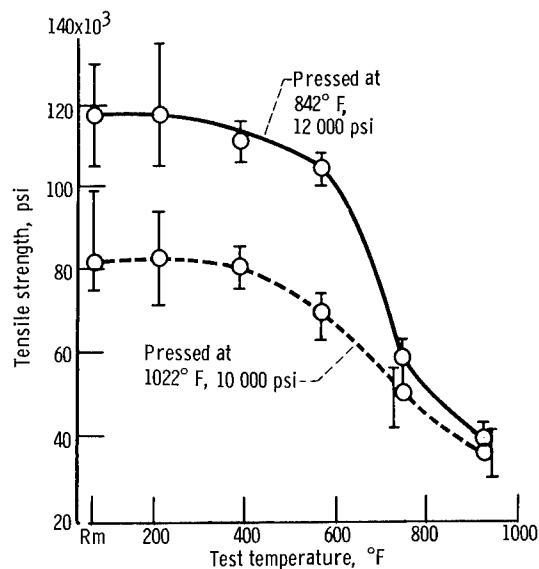
Figure 30. - Ultimate tensile strengths of silica-fiber-reinforced aluminum composites as function of hot-pressing conditions and test temperatures. Continuous silica fibers (ref. 13).
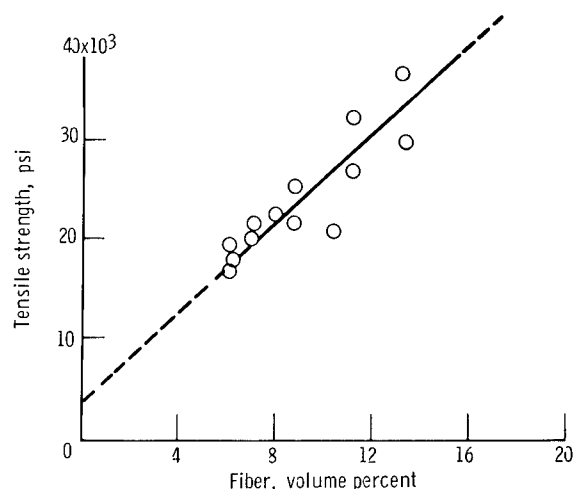
Figure 31. - Strength as function of composition for stainless-steel fibers in aluminum matrix. Fiber diameter, 0.002 inch (ref. 6).
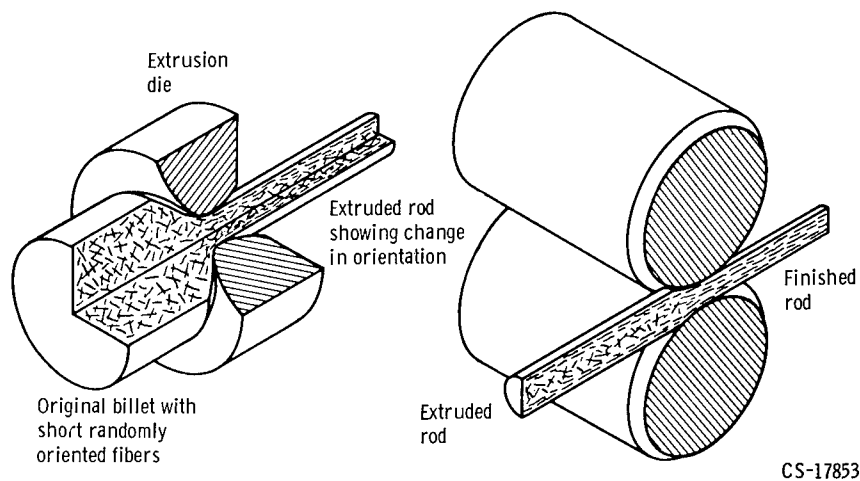
Figure 32. - Fabrication of fiber-reinforced metal matrix composites by extrusion and rolling. Discontinuous fibers (ref. 14).

Cratchley and Baker (see fig. 30), for example, coated silicon dioxide fibers with aluminum by passing the fiber through a molten metal bead (ref. 13). They then packed the beads in a hot press die and consolidated the composite by using pressure and temperature. The composites so produced had very high ultimate tensile strengths at room and elevated temperatures. Stress-carrying capacities of these composites were approximately 120 000 pounds per square inch, and high strengths were maintained to over 300° C. Cratchley, in an earlier work (ref. 6), had embedded stainless steel fibers in an aluminum matrix by a similar hot pressing process. Such materials could be utilized as engineering materials (see fig. 31).

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Figure 31 shows a plot of the tensile strength against volume percent fibers for one of the types of composites made by Cratchley. The fibers in this case were 2-mil fibers that were 1/2 inch long. The stainless steel contained 18 percent chromium, 9 percent nickel, and 0.6 percent titanium. The fibers in these composites ranged from approximately 6 to 14 volume percent. An extrapolation of the curve presented by Cratchley would extend to a value of tensile strength equivalent to the strengths of the fibers that he utilized (fig. 31). The data then appeared to obey a law-of-mixtures relation. This was true even though there may have been a very minor reaction at the interface between the fiber and the matrix. The procedures utilized, however, eliminated deleterious effects from an interface reaction, if there were any. In some work done by Jech, Weber, and Schwope (ref. 14), powder-metallurgy processes were combined with mechanical working to produce molybdenum-reinforced titanium-alloy composites. Figure 32 illustrates the working methods utilized to produce these composites.

Fibers that were 0.1 to 0.25 inch long were blended with either titanium powder or a titanium alloy powder (titanium - 6 percent aluminum - 4 percent vanadium). The powder-wire blends were cold-pressed into billets containing randomly oriented molybdenum fibers, which were vacuum sintered and extruded. The extrusion partially oriented the fibers as shown (fig. 32). The resulting rods were then hot-rolled. The rolling process oriented the fibers considerably; in fact, the fibers were elongated to as much as 6 inches in the final composite and reduced from 10 to 2 mils in diameter. Tensile strengths, as functions of temperature and volume percent fiber, are given in figure 33 for molybdenum-reinforced titanium-alloy-matrix composites. At all temperature indi-
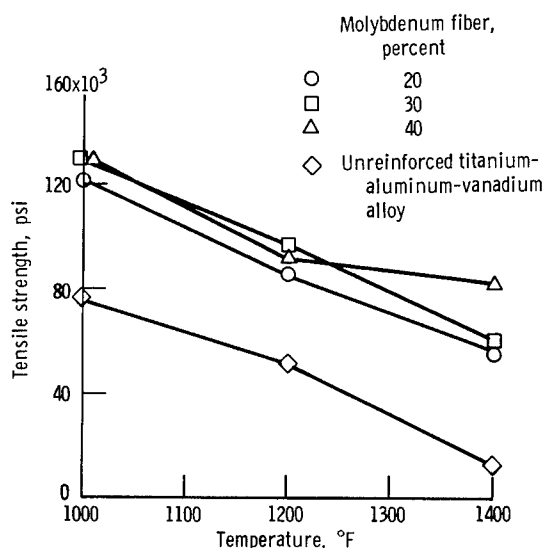

Figure 33. - Tensile strength as function of temperature of molybdenum-fiber-reinforced titanium - 6-percent-aluminum - 4-percent-vanadium alloy (ref. 14).
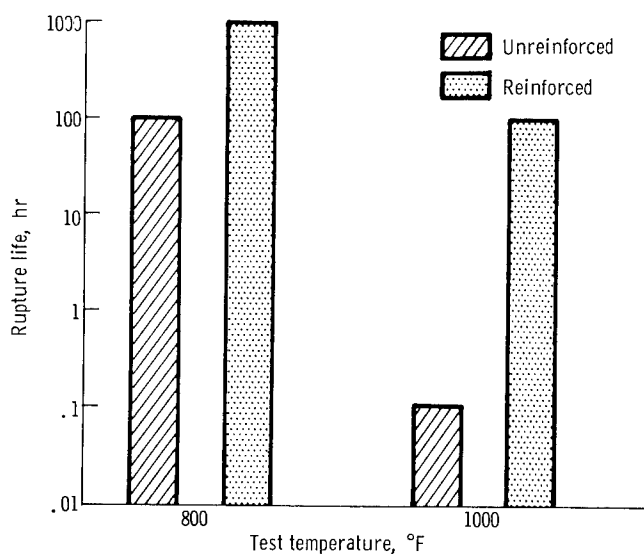

Figure 34. - Stress-rupture life of molybdenum-fiber-reinforced titanium-matrix composites and unreinforced titanium at 20 000 pounds per square inch as function of test temperature. Volume percentage of fibers, 10.
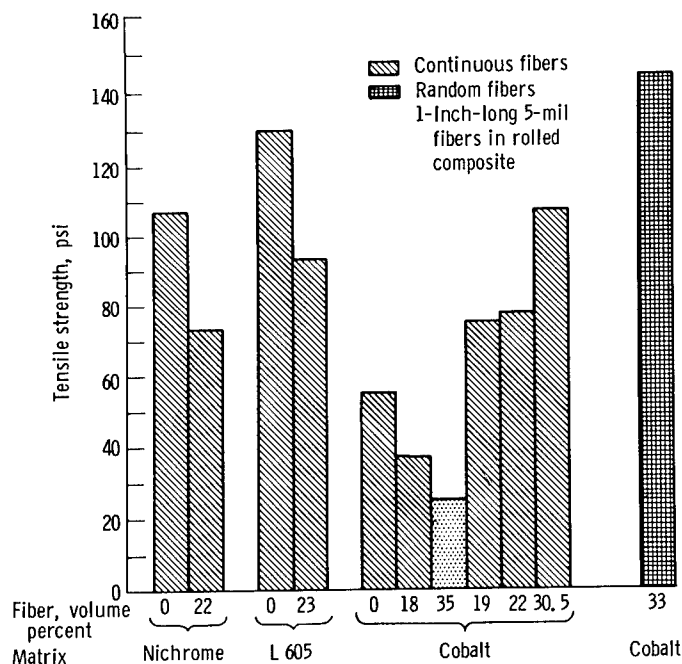
Figure 35. - Room-temperature tensile strengths of tungsten-fiber-reinforced cobalt- or nickel-base matrix composites. Dicontinuous 5- and 10-mil-diameter tungsten fibers; as-hot-pressed condition (ref. 15).

cated, the tensile strength is considerably above that of the unreinforced alloy.

Some stress-rupture data for the molybdenum-reinforced titanium materials were also presented by Jech, Weber, and Schwope in reference 14. The bar graphs (fig. 34) represent rupture lives obtained at 800° and 1000° F at a stress level of 20 000 pounds per square inch. With only 10-volume-percent fibers, the life was increased by a factor of 10 at the lower temperature and by a factor of 1000 at the higher temperature.

Recently, Baskey presented results obtained from numerous experiments on fiber-reinforced superalloys or high-temperature alloy materials (ref. 15). Some of the matrix materials to which tungsten fibers were added as reinforcements were powders of cobalt, L-605, Ni-Cr, and stainless steel. Composites were hot-pressed in some cases and cold-pressed and sintered in others, and some materials were worked subsequent to their initial fabrication. Room-temperature and 2000° F tensile tests were made of these materials. Figure 35 represents some of the results obtained from hot pressed materials. When 10-mil-diameter tungsten fibers were added to a Nichrome matrix, the strengths of the composites at room temperatures were decreased relative to the Nichrome. A similar situation was true for a matrix of L-605. When a pure cobalt matrix was used and a small amount of fibers were added, the properties were lowered. This was true for both continuous and for random fibers. With a larger volume percent of fibers, however, the strengths were increased considerably. When a composite containing 33 volume percent was further rolled or mechanically worked, the strength was increased by an appreciable amount. It should be pointed out that these studies were exploratory in nature, and fabrication problems arose that, in some cases, may have prevented the attainment of expected room-temperature strengths.

Elevated-temperature tensile tests at 2000° F of the same types of materials showed that the composites were much stronger than the matrix materials (fig. 36). This was true even though only a small volume fraction of fibers was used. This represents the second case, where elevated temperature test results for fiber-reinforced materials

Figure 36. - High-temperature tensile strengths of tungsten-fiber-reinforced cobalt- or nickel-base matrix composites. Diameter of tungsten fibers, 10 mils; as-hot-pressed condition; test temperature, 2000° F (ref. 15).

(a) Transverse section. X500. (b) Longitudinal section. X200.
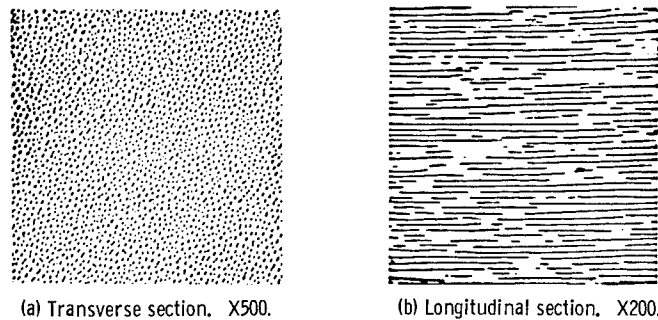
Figure 37. - Microstructure of unidirectionally solidified aluminum - aluminum-nickel (Al-Al$_3$Ni) eutectic specimen (ref. 16). (Reduced 50 percent in printing.)

were relatively better than low-temperature test values. The first case described was that of Petrasek (ref. 9), which also involved tungsten fibers but a different matrix (see figs. 20(a) and (b)). In cases where the test temperatures are greater than the ductile-to-brittle transition temperature of the fibers, it might be expected that one of the main problems associated with the use of tungsten, namely, its brittleness at room temperature, would have been eliminated.

A novel method that has been under investigation to produce composites of an engineering nature is the method of directional solidification of castings. Recently, Ford, Hertzberg, and Lemkey (ref. 16) have published results that show some idealized microstructures produced by casting an aluminum alloy with a fibrous aluminum-nickel intermetallic compound (Al$_3$Ni) in the matrix. Figure 37 shows a cross section and a longitudinal section of such a microstructure. Usually the problem associated with controlling a microstructure is that segregations associated with casting conditions prevent the arrangement of the phases in idealized manner. Also, fiber-like growths, such as those shown in figure 37, will usually not occur without special solidification techniques. The authors (ref. 16) have grown both rods and parallel or lamellar plate-like structures in

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Figure 38. - Stress-strain curve for 0.45-micron-diameter chromium whisker directionally solidified from copper-chromium alloy (ref. 16).

different alloys.

The aforementioned investigators (ref. 16) also have grown fibers of chromium in a chromium-copper eutectic alloy. These fibers have whisker strengths. Figure 38 shows a stress-strain curve for a whisker that was obtained by digesting the copper-rich matrix from such a eutectic. The tensile strength of the whisker exceeded 1 000 000 pounds per square inch. It is too early to speculate how far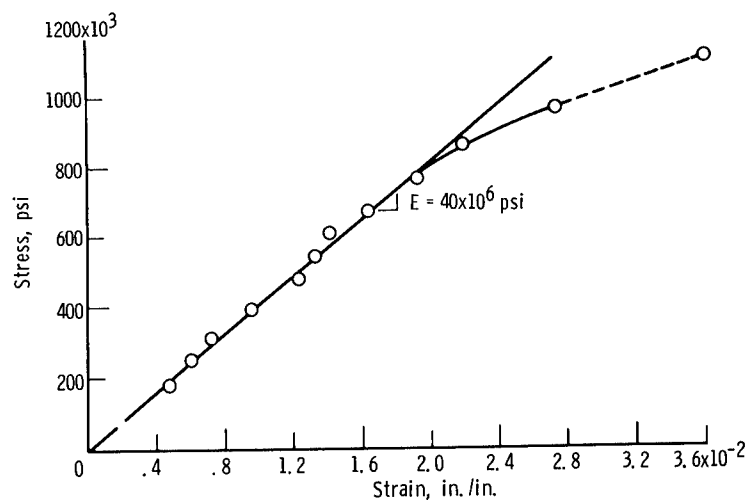 such casting approaches may be varied, but from an academic standpoint as well as a potentially practical one, the work is most interesting.

Finally, some recent work done at the Lewis Research Center in which ceramic and refractory compound fibers were made by extrusion (refs. 17 and unpublished work of R. W. Jech, J. W. Weeton, and R. A. Signorelli) will be discussed briefly. While the composites to be described are not sufficiently developed to be considered useful as engineering materials today, this method shows considerable promise.

In the first of two studies, tungsten was used as a matrix, and refractory oxide or refractory compound powders were elongated to form fibers during the fabrication process. Composites were made by blending tungsten powders with the powdered oxides or refractory compounds. Billets were extruded at a temperature of 4200° F at reduction ratios ranging from 8 to 20. Most of the specimens were extruded at a ratio of 8. This work was done to observe if the compounds were elongated during the extrusion process or reacted with the tungsten, or both. It was suggested in reference 17 that extrusion of fibrous ceramics or refractory compounds in a matrix has several inherent advantages. There is a possibility that fibers so produced may be much stronger than materials produced by conventional methods of sintering or fabrication. Since the fiber surfaces are created in contact with the matrix during extrusion, this process offers a better opportunity to obtain good bonding at the fiber-matrix interface. The matrix acts during the ex-

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(a) Unreinforced tungsten; cross section.

(b) Typical reinforced tungsten; cross section.

(c) Tungsten - 8 volume percent zirconium oxide; longitudinal section.

(d) Tungsten - 8 volume percent yttrium oxide; longitudinal section.

(e) Tungsten - 10 volume percent hafnium oxide; longitudinal section.

(f) Tungsten - 8 volume percent thorium oxide; longitudinal section.

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Figure 39. - Microstructures of extruded tungsten and tungsten composites containing fibered refractory oxides (ref. 17). X500. (Reduced 50 percent in printing.)

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Stress for 100-hour rupture life, psi

14 000
12 000
10 000
8 000
6 000
4 000
2 000

CS-33663
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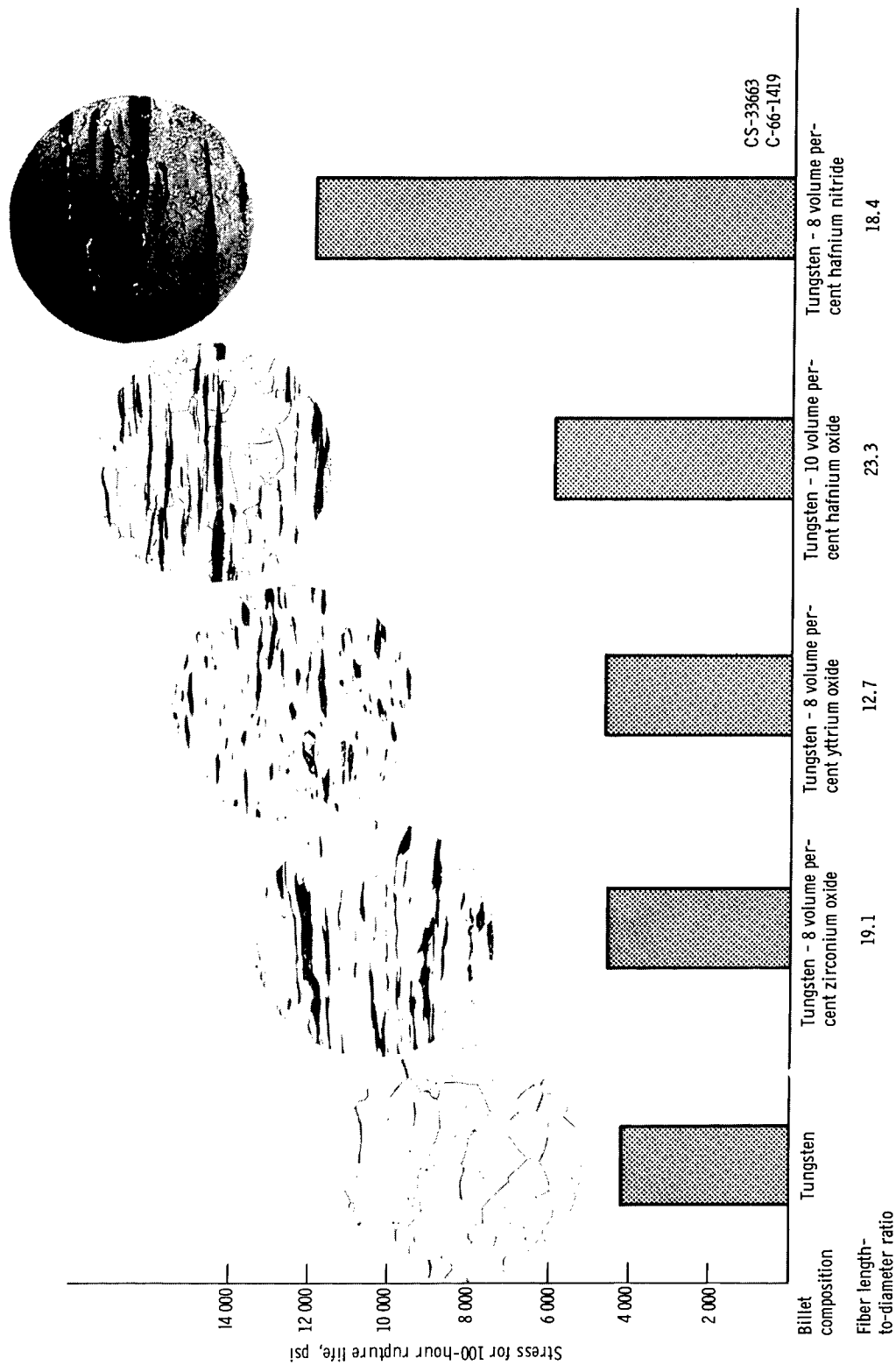| Billet composition	Tungsten	Tungsten - 8 volume percent zirconium oxide	Tungsten - 8 volume percent yttrium oxide	Tungsten - 10 volume percent hafnium oxide	Tungsten - 8 volume percent hafnium nitride
Fiber length-to-diameter ratio		19.1	12.7	23.3	18.4

Figure 40. - Stress-rupture strength of fiber bearing tungsten base composite materials showing microstructures and giving length-to-diameter ratios of fibered ceramics; test temperature, 3000° F (ref. 17). X500. (Reduced 50 percent in printing.)

trusion to distribute the stresses more nearly isostatically around the additive particles, improving the probability of deformation of hard, brittle materials. In addition, fibers made by these methods would not be touched or damaged by handling, nor would they be affected by atmospheres other than the metal in which they are extruded.

Photomicrographs of transverse and longitudinal sections of composites are shown in figure 39 (p. 31). Considerable fibering of oxides is evident in the longitudinal sections (figs. 39(c) to (f)). The degree of fibering also is indicated by the length-to-diameter ratios included in figure 40. These L/D values, determined from photomicrographs, range from 12 to 23. The stresses for rupture in 100 hours at 3000° F are also shown in figure 40 for the different materials. The strength increases due to the fibered zirconia, yttria, and hafnia were slight, since the strength of the final products was only slightly greater than the strength of the tungsten. In the case of the specimen with hafnium nitride, however, the property increase was substantial. At least two effects may have contributed to the strengthening of this composite other than fibering, a dispersion effect and an alloying effect. Ways to determine which of the effects noted is predominant are under study. For a first approach, however, it was felt that the success obtained in actually elongating these materials was significant. It is interesting also that the extrusion process used, including the proper selection of billet container design, permitted an elongation of materials such as carbides and borides.

Oxides were added to softer, more ductile matrix materials and extruded (unpublished work of R. W. Jech, J. W. Weeton, and R. A. Signorelli). Figure 41 represents the strength of columbium matrix composites containing fibered zirconium oxide. These results are of a preliminary nature, but the composite products are considerably stronger than columbium. The composites have not reached the strengths obtained by F-48, a typical high-strength alloy of columbium, but as a first approach it is felt that the property increases have been significant.
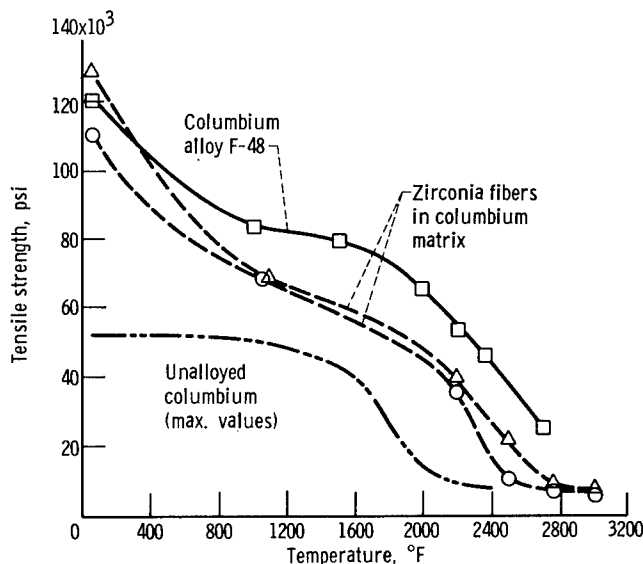

Figure 41. - Tensile strength of extruded ceramic-fiber-reinforced columbium matrix composites as function of temperature.

POTENTIAL OF FIBER-METAL COMPOSITES

The ultimate potential of fiber-reinforced composites will depend in large measure on the ultimate strength or other properties of fibers that might be developed. Here, however, there ⟶
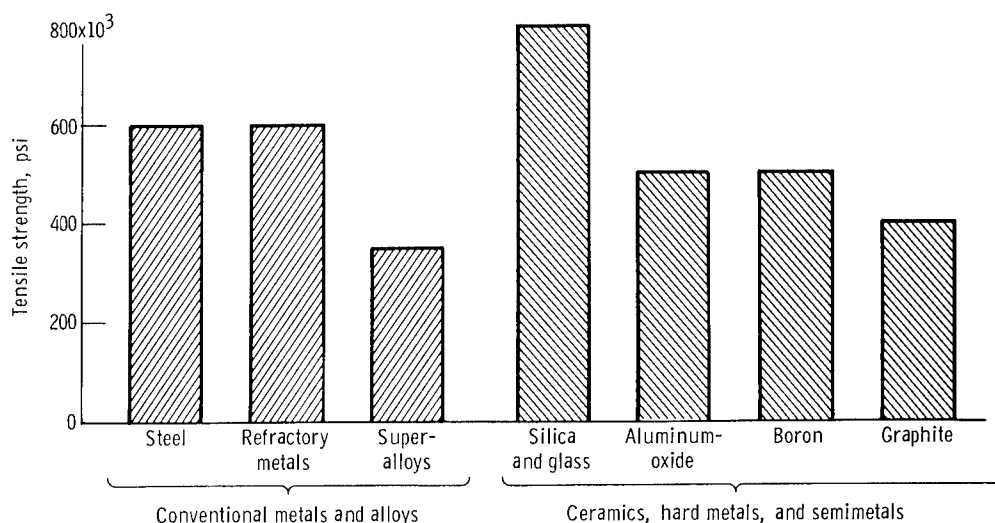
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Figure 42. - Room-temperature tensile strength of polycrystalline and glassy fibers.

are many unknowns. It is not known, for example, what strengths might be produced in such fibrous materials as conventional metals, particularly alloys, or in ceramics, hard metals, or even semimetals such as boron and carbon. Possibly, polycrystalline fibers of different types will be made that will have strengths approaching whisker strengths. Whiskers, of course, are expected to have strengths that are close to the theoretical strength of the materials and possibly will be the maximum strength that will be achieved in a given type of materials.

Actually, several types of fiber materials have been produced that exhibit unusual strength. Figure 42 shows some room-temperature tensile properties of several types of polycrystalline fibers. Conventional metallic alloy fibers are indicated by the three bars at the left in the figure. It is interesting that one of the strongest metallic fibers that has been made is a steel wire that has a strength of about 600 000 pounds per square inch (ref. 18). Numerous superalloy fibers have also been made, and some of these are considerably stronger as wires than they are in bulk form. This is true also at elevated temperatures. The strengths of some superalloy fibers are of the order of 300 000 pounds per square inch (ref. 11). Refractory metal fibers such as tungsten, molybdenum alloys, columbium, and tantalum also have been shown to have high strength. One of the strongest wires that has been produced is tungsten, which has also achieved a strength of 600 000 pounds per square inch (ref. 20). The four bars at the right in figure 42 indicate strengths of some materials other than conventional metals and alloys. These materials may be classified as ceramics, hard metals, and/or semimetals. In materials like silica and glass, for example, strengths of as much as 800 000 pounds per square inch have been achieved (ref. 21). Strengths of 500 000 pounds per square inch have been reported for aluminum oxide fibers by private communication from the Babcock and Wilcox Company. A similar strength has been obtained for some boron fibers (ref. 21). In graphite,

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strengths of as much as 400 000 pounds per square inch have been achieved in unreported work of Union Carbide Corporation, Carbon Products Division.

Under room- or low-temperature test conditions, many different types of fiber products (metals, ceramics, and semimetals) might have strengths that are similar in magnitude and very high. At elevated temperatures, however, depending on the stability of the different materials, the strengths would be widely differing. In all cases, the materials, however, will lose properties as the test temperature is increased. In some cases, a thermal treatment alone causes a degradation of the properties in the materials. The conventional metallic materials indicated in figure 42 owe their properties, in part, to strain energy resulting from mechanical working and, in part, to microconstituents within the materials. A good portion of the strength of the materials then depends on the strain energy retained in the material and the stability of the microstructures. Relative to the conventional metals, it may be generalized that alloys or materials containing precipitates or dispersoids would be expected to lose strength less rapidly with increasing temperature than relatively pure or simple alloys. The materials such as ceramics, hard metals, and semimetals may gain their strength from factors other than stored energy and, thus, may be expected to lose strength less rapidly with increasing temperature. Ceramics, although they often retain strength at high temperatures, usually degrade in properties with increase in temperature.

It should be recognized that many of the existing metallic fibers have been made for purposes other than reinforcement of metals. It is believed that it will be necessary to develop new fiber alloys that will be specifically designed for use in composites. Similarly, matrix materials must be alloyed or made of materials that will permit the fiber to remain stable within it. Some fibrous materials should have a capacity for being strengthened in the matrix. Certainly much development work must be done to capitalize fully on the potential strength of metallic fibers. Ceramic, hard metal, and semimetals also will require a great deal of work before fibers can be developed specifically for reinforcement purposes. Some of these products might be developed by novel means, and it is entirely possible that, by reorientating the crystallites of such materials or by orientating the constituents within the materials, completely different products might be produced from those available today. Alloying such materials as ceramics is another method of producing a new generation of materials.

Materials such as metals, semimetals, carbon, and boron tend to react with many metallic matrix materials at high temperatures. In some cases, these reactions might be tolerated, but in others they might have to be avoided. In many cases, it would be ideal if complete heterogeneity could be maintained between the matrix and the fiber. It is conceivable, although not demonstrated as yet, that increases in properties might result from synergistic effects between the fibers and the matrix and permit the production of a composite material with properties far in excess of those that might be predicted from

the strength of the individual materials. Materials such as ceramic (oxide) fibers, which have unusually high negative-free energies of formation, would be expected to be more stable in a given matrix than would materials with lower negative-free energy values. Hard metals or semimetal fibers, because of their reactivity with many matrices, could conceivably be combined with relatively low melting materials, where the exposure temperatures would never become great, or they might require a diffusion barrier or protective layer that would inhibit or prevent reactions between the matrix and fibers in higher use temperature materials. Diffusion barriers between fibers and matrices will be necessary for many fiber-metal systems.

Figure 43 illustrates, schematically, concepts relative to the use of a high-melting-point fiber in a low-melting-point matrix. The figure is based on data for both model and developmental fiber-metal system, namely, (1) the embedding of tungsten fibers in copper and testing at elevated temperatures (ref. 9), (2) the embedding of aluminum oxide or sapphire whiskers in a silver matrix (ref. 22), and (3) the incorporation of glass fibers in aluminum or in aluminum alloys (refs. 23 and 24). Below the scale for the homologous temperature of the matrix, homologous temperatures of a typical fiber are presented. At the melting point of the matrix, at a homologous temperature of 1.0, the fiber in this composite would be at a homologous temperature of only 0.35. The resulting composite would then maintain strengths to very high percentages of the melting point of the matrix. To accomplish this type of strengthening for high-temperature materials, there must be some degree of heterogeneity between the fiber and the matrix material. The strength of the composite, relative to that of the matrix, would of course depend on both the strength of the fibers selected and the volume percent of the fibers utilized and could be more or less than shown in figure 43. Where discontinuous fibers are used, it should be assumed that fibers would be long enough that interfacial bond strengths or matrix shear strengths w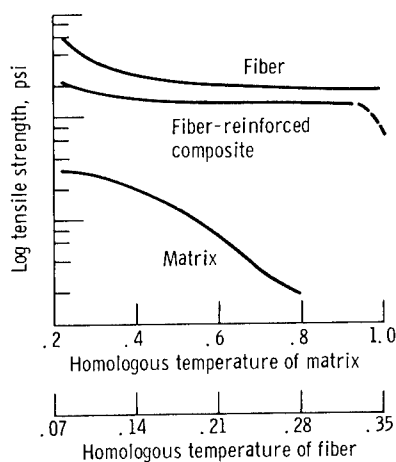ould not be exceeded. For composites to be used at low temperatures, the melting point differential between the fiber and the matrix may not be as important as for high-use temperature composites. Such composites also might be designed by using strength and density considerations rather than melting point or thermal stability relations. Possibly too, fibrous materials might be incorporated in metallic matrices having melting points nearly equal to those of the fibrous materials. Such composites could then be used at temperatures closer to the melting point of the fibers. Some examples of this are aluminum alloys in aluminum, copper alloys in copper, alloy steels in iron and more corrosion-resistant weaker steels, superalloys in steels and more corrosion-resistant alloys, tungsten

Figure 43. - Schematic comparison of tensile strength of composite and composite components as function of homologous temperature.
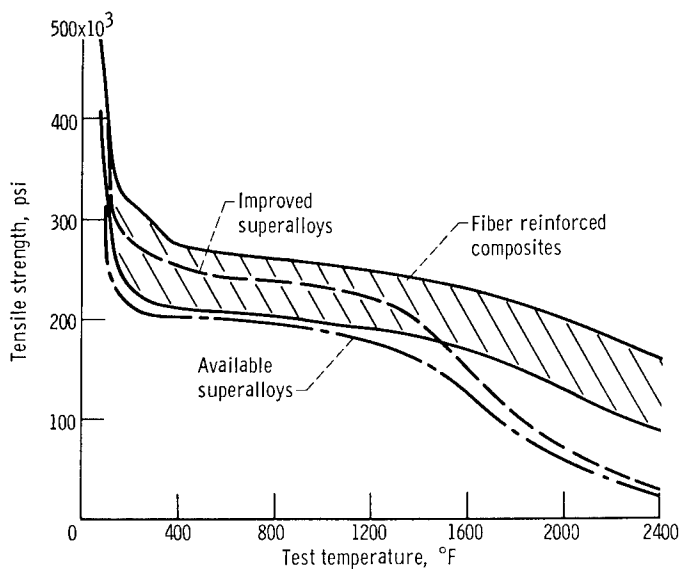
Figure 44. - Potential tensile strengths of fiber-superalloy composites and superalloys.

in tantalum, and ceramics in refractory metals.

An attempt will be made to illustrate some of the elevated-temperature potentials of composites and alloys. Figure 44 illustrates the elevated-temperature tensile strength potential of fiber-reinforced superalloys compared with superalloys. It should be emphasized that, even though the curves that are presented pertain to superalloy systems, similar sets of curves could also be drawn for other matrix systems. For example, similar sets could be drawn for aluminum, copper, or refractory metal base materials. If the plots of the types shown in figure 44 were made by using a homologous temperature scale, the stress-carrying capacities at given homologous temperatures of many of the different materials would be of the same general magnitude. The comparisons of figure 44 then, because they cover an actual-use temperature range of great interest, more graphically illustrate the relative potentials of the different types of materials.

The solid curves in figure 44 represent the strengths of superalloys and should be considered a basis for comparison. The lower of these curves represents strength of alloys presently developed. The upper curve represents possible improvements in strengths of superalloys and is based on an arbitrary 25-percent stress increase over the values shown in the lower curve. To reinforce a superalloy matrix with the objective of improving its strength, it is necessary to add fibers stronger than the superalloy.

Typical fibers that would have strengths in excess of those of the superalloys in these temperature ranges would be refractory metal fibers, some of which exist today, and possibly ceramic fibers. The superalloys shown in figure 44 by the area within the dashed lines could have strengths shown by the shaded area if reinforced by fibers. The lower values of the shaded area represent composites containing fibers only slightly stronger than the matrix. Low-strength composites also would be obtained with low volume percentages of high-strength fibers. The upper portion of the shaded area represents composites containing stronger fibers. The strength values could be very substantial, ranging from as much as 500 000 pounds per square inch at room temperature to 200 000 pounds per square inch at 2000° F. Although the values indicated seem high, particularly for the higher temperatures, such composites could be produced from high-strength
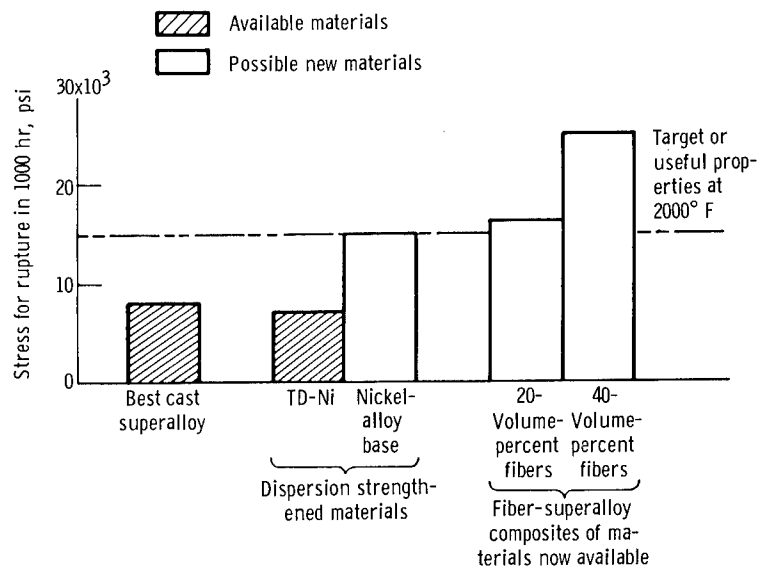
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Figure 45. - Composites for stress-rupture applications possible in near future.

fibers that exist today. If stronger fibers specifically designed for reinforced composite matrices were developed, composite strengths could be higher than the upper values indicated. All the aforementioned strengths are based on the assumption that no deleterious reactions between the fiber and the matrix would occur during processing or testing.

Superalloys are used more for creep-rupture or long time applications than for tensile applications. Stress-rupture studies shown earlier (ref. 12) suggest that the rupture properties of highly worked fibers are superior to those of similar materials in bulk or sheet form. It has also been suggested that the stress-rupture properties of a composite can be related to the rupture properties of the matrix and fibers, as has previously been shown (refs. 1 to 4) for tensile properties (law of mixtures). If the full potential stress-rupture strength of fibers can be utilized, in a composite, fiber-reinforced superalloys may be considerably superior to conventional superalloys.

Some long time, high-temperature (i.e., stress rupture) possibilities for use of fibers are illustrated in figure 45. Here, a hypothetical design condition has been selected. A composite with mechanical properties such as those desired, namely, 15 000 pounds per square inch, 1000 hours, and 2000° F, would be most desirable for air-breathing gas turbines, for example, if they could be made oxidation resistant. The best superalloy strengths today are about 8000 pounds per square inch; wrought products are even less strong. Commercially produced thoria dispersion strengthened nickel has a strength of 7000 pounds per square inch. Other nickel or superalloy base dispersion-strengthened materials are expected from several sources that will meet the 15 000-pound-per-square-inch goal. With known fiber and matrix materials available today, the high strengths shown at the right in figure 45 are possible.

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SUMMARY

The law-of-mixtures relation represents the behavior of some fiber-metal composites at room temperature and elevated temperature for tensile and stress-rupture application. Creep-resistant high-strength composites appear to have great promise for a wide temperature range. Some attempts have been made to produce practical engineering materials utilizing fundamental as well as developmental concepts. Encouraging results have been obtained in the production of these materials. These results, coupled with the potentials indicated from more fundamental programs, suggest that exceedingly strong fiber-metal composites will be produced in the future. These fundamental studies should further encourage the development of practical engineering materials. However exciting the results envisioned may be, even higher strength materials may be possible if fibers and matrix materials designed for composites are developed.

Lewis Research Center,
    National Aeronautics and Space Administration,
        Cleveland, Ohio, March 29, 1966.

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End

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