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DIRECTIONALLY SOLIDIFIED COMPOSITE SYSTEMS UNDER EVALUATION

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DIRECTIONALLY SOLIDIFIED COMPOSITE SYSTEMS UNDER EVALUATION

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SUMMARY

The directionally solidified eutectic in-situ composites being evaluated for use as turbine materials range from ductile-ductile systems, where both matrix and reinforcement are ductile, to brittle-brittle systems, where both phases are brittle. The alloys most likely to be used in gas turbine engines in the near term are the lamellar ductile-semi ductile alloys gamma prime-delta, Ni_3Al-Ni_3Nb and gamma/gamma prime-delta Ni,Cr,Cb,Al/Ni_3Al-Ni_3Nb and the fibrous ductile-brittle alloys M-MC CoTaC or NiTaC and M-M_7C_3(Co,Cr,Al)-$(Cr,Co)_7C3$. Gamma prime strengthened NiTaC alloys and a (Co,Cr,Al)-$(Co,Cr)_7C3$ have greater tensile strength than the strongest superalloys at temperatures up to about 600° C. The gamma prime-delta and gamma/gamma prime-delta alloys in the Ni,Al,Nb(Cr) systems have greater tensile strength than the superalloys at temperatures greater than 800° C. At low stresses fibrous carbide reinforced eutectic alloys have longer lives at high temperatures than the strongest superalloys. Lamellar delta, Ni_3Nb reinforced eutectic alloys have longer lives at high temperatures than the strongest superalloys at all stresses. The experience currently being gained in designing with the brittle ceramics SiC and Si_3N_4 may eventually be applied to ceramic matrix eutectic in-situ composites. However, the refractory metal fiber reinforced brittle-ductile systems may find acceptance as turbine materials before the ceramic-ceramic brittle-brittle systems.

INTRODUCTION

I have the impression that aircraft engine designers are a conservative lot and that the materials engineers or metallurgists who provide the materials and data with which they work are not much less conservative. However, over the years designers have learned that they could safely use cast alloys for turbine blades even though "castings" was a dirty word in many engine companies and engineering schools. The turbine blade and vane casting alloys with which gas turbine designers have become comfortable are all based on a ductile Ni or Co face centered cubic matrix (ref. 1). At least the matrix is ductile when the blades are installed in the engines.

Despite their conservatism designers and metallurgists have responded to the pressure to run engines hotter and hotter. As a consequence they have considered alloys having higher and higher use temperatures but possessing less and less ductility. This trend is reflected in figure 1 which shows that as use temperatures have increased, ductility has decreased (ref. 2). Indeed, the high pay-off in terms of material temperature that would result from using highly refractory and corrosion resistant, but brittle ceramics, and the large sums of money that are being invested by government and industry to develop ceramic turbine materials (ref. 3) have lent a certain respectability to research in brittle turbine materials. The design experience gained with materials like SiC or Si_3N_4 may well pave the way for use of other materials which at one time would have been rejected out of hand because of low ductility.

Long time strength and oxidation resistance are the prime requirements for a turbine blade or vane. Ductility itself, as well as impact strength and fatigue strength both of which are dependent on strength and ductility, are also important. For rotating blades, strength must of course be related to density to give a true picture of an alloy's worth.

The search for materials with greater rupture-strength at high temperatures to meet the needs of advanced engines has led to directionally solidified eutectic alloys which are also referred to as in-situ composites. It is hoped, as this new technology develops, that this class of materials will provide the basis for turbines with material temperatures far above any now possible. Directionally solidified composites include materials that have a wide range of properties. They range from materials that are highly ductile to those that are entirely brittle. Most are highly anisotropic; some are relatively insensitive to the direction of applied stress. Some can be processed by current foundry practices; some will require new technology if they are to be grown to shape.

The purpose of this paper is to review various types of high temperature in-situ composites and to consider which offer the most potential for future development. I shall categorize some of the systems that have been investigated according to the ductility of the component phases. The categories range from ductile-ductile to brittle-brittle. I shall consider examples in each category with particular emphasis on systems which now look attractive for use in gas turbine engines, touching on microstructure, mechanical properties, and process problems; and then conclude with some prognostication on the direction of future development of directionally solidified composites for gas turbines.

DEFINITIONS

The materials we are considering exhibit "normal" eutectic microstructures in the sense that their growth is coupled (ref. 4). The reinforcing fibers or lamellae are formed by cooperative growth with the matrix under conditions of plane front solidification. The classification system for high temperature in-situ composites shown in Table I was suggested by E. R. Thompson and is based on the room temperature mechanical behavior of the component phases of the eutectics. The categories are ductile-ductile, ductile-semiductile, ductile-brittle, brittle-ductile and brittle-brittle. The first word applies to the matrix and the second to the reinforcing phase. I am indebted to Alan Lawley for many of the entries which appear in Table I which were first presented in table II of reference 5 where the categories are based on the types of phases present (e.g., metal, intermetallic compound, ceramic).

Strictly speaking a brittle material is one which behaves in a linearly elastic manner to failure. A nominally ductile material undergoes considerable plastic deformation before failure. In some of the systems reviewed the degree of brittleness or ductility of the phases is known only qualitatively. Within any one system, alloy purity or alloy additions made to improve strength may have a profound effect on the ductility.

To categorize the eutectics listed precisely, it would be necessary to examine fractured specimens at high magnification to determine whether the phases present had failed in a ductile or a brittle fashion. Certainly the ductility of the in-situ composite depends on the disposition of the phases. While a matrix phase may be ductile in its own right, it may show little strain at fracture when restrained by adjacent lamellae of a reinforcing phase. Or, conversely, a phase which may be brittle in the bulk, may deform plastically in a composite structure as does delta, Ni_3Nb by twinning. In some instances a phase has been found to be brittle by one investigator and ductile by another. This difference may be the result of differences in purity in the starting materials or the effect of alloying additions.

In this present compilation of eutectics we are, of course, discussing only alloys that have been considered because of their potential high temperature use. "High temperature" means different things to different people. For this paper its definition is implicit in Table I in that none of the materials listed melt at less than 1150° C and only two at less than 1200° C.

SYSTEMS UNDER EVALUATION

As might be expected the trend in eutectic alloy development has been from the simple to the more complex, and the goal has been to achieve ever higher use temperatures. Early work was with low melting alloys that could be handled in relatively simple furnaces. Now investigators are studying systems that cannot be contained in a crucible and can be melted only with great difficulty. Early work was also directed at binary alloys. Almost all current work is with more complex systems to improve particular properties.

In the following sections of this report example systems of each of the categories listed in Table I will be discussed, with emphasis on these alloys or alloy types that are of most interest as turbine materials.

Ductile-Ductile

Relatively few high temperature ductile-ductile systems have been investigated and none have been investigated in great depth. The reinforcing phase is usually a refractory metal. In the Ni-W eutectic (ref. 6) W fibers reinforce a Ni matrix. This system is highly ductile with an elongation of 45 percent at room temperature but only moderate tensile strength, 830 MN/m^2 (120 ksi) (ref. 7). Its composition, Ni-45W w/o (ref. 8), would not be expected to be oxidation resistant, and would require additions of aluminum or chromium to make it so, as well as to reduce its density.

Such a modified alloy is being investigated (ref. 9). It also consists of a face centered cubic matrix and a body centered cubic reinforcing phase. The composition of this γ-α alloy is Ni-0.1Al-37.0Cr-10Co-18W. With a fully lamellar structure, this alloy has a tensile strength of 424 MN/m^2 (61.5 ksi) and elongation of 22.4 percent at 1093° C. Its oxidation resistance in cyclic exposure at 1100° C was not quite as good as Hastelloy X.

Another ductile-ductile system is based on a gamma-prime matrix. It is Ni_3Al-Mo (ref. 7). Preliminary data from F. D. Lemkey, UARL, show that a γ'-α alloy Ni-8Al-27Mo is superior to one of the strongest conventionally cast superalloys, NASA-TRW VIA, in tensile strength at most temperatures and also superior in rupture life. At room temperature it has an elongation of 20 percent.

Although this simple alloy has excellent properties, its very simplicity suggests that its properties could be improved by the addition of other elements. The faceting of its Mo fibers indicate that the microstructure would be resistant to coarsening at high temperatures (ref. 10).

An alloy which has not been explored in detail is Ni_3Al-Ni_7Zr_2 (refs. 7 and 11). It is mentioned here only because of the interesting kinking mode of deformation in compression of the two lamellar intermetallic phases at room temperature. It has a tensile strength of over 414 MN/m^2 (60 ksi) at 1093° C. No room temperature tensile properties were reported.

Ductile-Semiductile

The ductile-semiductile systems discussed here are all based on the reinforcement of a ductile matrix phase by the semiductile intermetallic phase Ni_3Nb. In the Ni-Ni_3Nb eutectic (refs. 7, 12, 13, 14) it has been shown that the deformation of Ni_3Nb is accomplished by twinning on the (211) planes of delta. The gamma Ni deforms by both slip and twinning (ref. 13). The modes of deformation of this alloy result in fractures (ref. 12) where necking of the Ni lamellae occurs in conjunction with both twinning and cracking of the delta.

The Ni-Ni_3Nb system is neither strong nor oxidation resistant enough to be considered as a turbine material. Therefore, other systems reinforced with delta have been developed to provide the properties required.

One of these is the gamma prime-delta system, where Ni_3Al has been substituted for gamma and another is gamma/gamma prime-delta, where the gamma phase has been strengthened by the precipitation of gamma prime. In both of these systems the delta deforms plastically to a limited degree by twinning as it does in the simpler Ni-Ni_3Nb alloy. Figure 2 shows the microstructure of gamma prime-delta (ref. 15) and figure 3 shows the microstructure of gamma/gamma prime-delta (ref. 16).

Gamma prime-delta - The pseudo-binary eutectic gamma prime-delta, Ni_3Al-Ni_3Nb was one of the first alloys in the ductile-semiductile category that received significant attention as a turbine blade alloy (ref. 15). It has a lamellar structure with approximately 44 v/o of the semi-ductile delta Ni_3Nb in a matrix of ductile gamma prime Ni_3Al. Its composition is Ni-23.1Nb-4.4Al.

As may be seen in figure 4 which shows the ultimate tensile of Ni_3Al-Ni_3Nb over a range of temperatures, it has greater tensile strength at temperatures above 900° C than several other promising eutectic alloys. This figure presents the effect of temperature on the tensile strength of those eutectic alloy systems that have received the most attention and which are most likely to be used in gas turbine engines. For comparison the properties of two strong superalloys, directionally solidified MAR-M200 and conventionally cast NASA-TRW VIA are shown.

At low and high temperatures, strength of gamma prime-delta increases with decreasing lamellar spacing according to a Hall-Petch relation. Its strength is therefore sensitive to growth rate since the lamellar spacing decreases with increasing growth rate. This alloy is highly anisotropic and has considerably less strength in the transverse direction than it does parallel to the growth direction (ref. 17). For example, as shown in figure 5, up to 800° C its transverse tensile strength is less than one-third the longitudinal strength (ref. 17). When bent transversely, the alloy is brittle at room temperature, but ductile at 1093° C (ref. 15).

To improve ductility several other Ni-Nb-Al alloys have been developed. Examples of room temperature stress-strain curves for these alloys in figure 6 show their greater strain to fracture. These alloys include: a hypo-eutectic gamma prime delta with a lower, 32 v/o, volume fraction of delta (ref. 17); a ternary gamma-gamma prime-delta eutectic which solidifies in three phases (ref. 18); and gamma/gamma prime-delta, a bivarient eutectic which solidifies in two phases, gamma and delta (ref. 16). The bivariant $\gamma/\gamma' - \delta$ alloy will be discussed in more detail under a separate heading.

Despite its low ductility the gamma prime-delta eutectic shows surprising impact strength. At room temperature its notched Charpy impact strength, shown in figure 7, is about 2.7 joules (2 ft-lb) and is comparable to cast superalloys Mar M-302 or Mar M-200. At 1000° C the impact strength is over 20 joules (14.7 ft-lb) (ref. 19). During impact fracture cracks are diverted by longitudinal splitting of lamellar interfaces and grain boundaries (ref. 7).

The rupture strength of gamma prime-delta and other alloys of interest as turbine blades or vanes is shown in figure 8 plotted against the Larson-Miller time-temperature parameter. Up to a parameter of 55, which is equivalent to 1000 hours at 1050° C the gamma prime-delta is superior to the other eutectic alloys shown.

Gamma/gamma prime-delta - In the Ni-Nb-Cr-Al system a series of alloys have been identified (ref. 16) which show promise of meeting the requirements for gas turbine engine blade materials. These alloys called gamma/gamma prime-delta solidify as lamellae of gamma (Ni,Cr,Al) and of delta (Ni_3Nb). On cooling a precipitate of gamma prime Ni_3Al forms in the gamma, face centered cubic solid solution matrix. Hence, the name gamma/gamma prime-delta. They fall in the ductile-semi-ductile class. One of the alloys in this series that has received particular attention has the nominal composition Ni-20.0 w/o Nb-6.0 w/o Cr-2.5 w/o Al. This alloy is a natural outgrowth of the alloy Ni-Ni_3Nb (ref. 12). Although Ni-Ni_3Nb has considerable strength at high temperature, the matrix is precipitation strengthened only slightly by Widmanstatten delta. The gamma matrix of the gamma/gamma prime-delta alloys is strengthened principally by the precipitation of gamma prime in the manner of nickel base superalloys. There is also opportunity for precipitation of gamma double prime, a tetragonal form of Ni_3Nb as well as delta Ni_3Nb (ref. 20). The alloy Ni-20Nb-6Cr-2.5Al has a density of 8.58 gm/cm, a liquidus of 1261° C, and a solidus of 1243° C. The volume percent of delta lamellae is 37.3 percent. A characteristic of this alloy helpful in growing long turbine blades is that during solidification the composition along the length of an ingot remains relatively constant, with the chromium and aluminum increasing only slightly from beginning to the end (ref. 16).

Figure 4 shows the tensile strength of gamma/gamma prime-delta as a function of temperature. Its strength is superior to that of two of the strongest available superalloys over almost the entire temperature range. Its ductility as shown in figure 6 is considerably greater than the ductility of gamma prime-delta. Its elongation of 2 to 5 percent at room temperature increases with temperature. Its tensile anisotropy as shown in figure 5 is also less marked than that of gamma prime-delta. The impact strength of gamma/gamma prime-delta also increases with temperature as shown figure 7 (ref. 21).

Figure 8 shows the rupture strength of gamma/gamma prime-delta in comparison to that of gamma prime-delta and several other eutectic alloys. Although gamma/gamma prime-delta has less rupture strength than gamma prime-delta, it is being considered more seriously as a turbine blade alloy because of its greater ductility and its greater oxidation resistance. Its rupture life and ductility at the temperature and stress that might be expected in a turbine blade root attachment are shown in figure 9, taken from reference 22. The ability to control properties through control of growth conditions is evident in this figure which shows the effect of growth rate on strength and ductility. As growth rate increases for a given gradient the structure becomes more cellular and rupture life decreases but ductility increases. It should be possible to optimize the structure in a turbine blade, with a ductile cellular structure in the root which solidifies more rapidly because of its proximity to a cooled stool, and with a strong fully aligned structure in the airfoil.

Work is being pursued to evaluate and improve the shear and transverse properties of this alloy. To do this addition of minor elements such as Zr, B, and C are being made to alter grain boundaries (ref. 23) and the effect of growth parameters on properties is being considered.

Ductile-Brittle

In the ductile-brittle category all the examples listed in Table I are based on a ductile gamma matrix and a brittle second phase. In one example, Ni-Cr, chromium rich lamellae failed at room temperature in a typical brittle manner while the nickel rich phase was ductile (ref. 24). In two other systems the reinforcing second phase lamallae are intermetallic compounds. In the $Ni-Ni_3Ti$ system (ref. 25) brittle fracture of the Ni_3Ti plates occurs. The $NbCo_3$ in the $Co-NbCo_3$ system is also lamellar and the intermetallic phase is also brittle at room temperature (ref. 26). Probably because of their low oxidation resistance and brittle behavior neither intermetallic system is being considered for turbine use.

In the other examples of ductile-brittle systems the brittle second phase is a carbide. The M-MC systems (ref. 27) have been intensively studied in this country and abroad. However, the gamma-M_7C_3 alloys have also attracted considerable attention (refs. 28, 29, and 30). Alloys from both of these systems are being considered as turbine blade or vane materials.

M-MC - The general family of a gamma matrix containing mono carbide fibers has been widely studied (ref. 27) and has many attractive properties. The early work was directed at pseudo binary eutectics of cobalt or nickel and a mono carbide of a group V element (refs. 31 and 32). Following the trend observed in most systems that have been studied intensively, alloying additions were made to improve particular properties. The M-MC alloys have exhibited great flexibility in accepting alloy additions to the matrix or to the fibers. Chromium or aluminum were added to increase oxidation resistance (ref. 27). In nickel base alloys aluminum was also added for the purpose of strengthening the matrix with gamma prime precipitate (ref. 33). Solid solution strengthening additions such as tungsten and molybdenum were also made (ref. 34). Where the matrix was cobalt-based, additions of nickel and iron have been made to stabilize the fcc structure (ref. 34). The fibers which are nominally TaC in most of the alloys studied, are brittle at room temperature (ref. 35). In some alloys the fibers have been modified by the addition of other group V and group IV elements (ref. 34).

The effectiveness of additions of nickel and chromium in suppressing the HCP-FCC transform in a CoCr-TaC alloy has been shown by differential dilatometry, figure 10 (ref. 27). Such stabilization eliminates repeated phase transformations during cyclic heating which have been shown to be deleterious to subsequent stress rupture properties of Co, Cr-TaC alloys (ref. 34).

Even some stabilized cobalt base alloys have shown a loss of stress rupture properties as a result of thermal cycling. Both the alloy Co-50-BW3 Co-15.7Cr-9.5Ni-3.0W-12Ta-0.77C) (ref. 34) and Co-25Ni-15Cr-13TaC (ref. 36) showed a loss of stress-rupture life as a result of thermal cycling to 1095° C. At low stresses 138 to 172 MN/m^2 (20 to 25 ksi) and high temperatures there was an improvement in the rupture properties of Co-50B3W with the first 500 thermal cycles followed by a decrease with increasing numbers of cycles (ref. 34). The same trends were observed in Co-25Ni-15Cr-13TaC (ref. 36). The TaC fibers in these alloys became increasingly serrated as the number of cycles increased (fig. 11). The loss in rupture life may be due both to the serration of the fibers (ref. 35) and to an increase in the number of subgrains and recrystallization of the matrix (ref. 37).

Bibring (ref. 27) has reported that there was no detectable degradation in specimens of CoTaC-3 cycled up to 1000° C. He suggests that at higher temperatures the variation in solubility may provide a driving force for diffusion and presumably cause deterioration of the fibers. This conjecture was born out by the work of Dunlevey (ref. 36) who showed that higher temperatures had a more potent effect. For example, only 500 cycles to 1230° C degraded the rupture life of a CoCrNi-TaC eutectic as much as 5000 cycles to 1095° C. It is further supported in the study by Breinan, et al. (ref. 38), who reported on microstructural changes in a number of alloys cycled to temperatures up to 1120° C. Neither gamma prime-delta nor gamma/gamma prime-delta showed any degradation of the reinforcing lamellae after 3000 cycles to 1120° C. The damage to carbide fiber reinforced alloys was related to the maximum exposure temperature. Ni-NbC appeared damaged when cycled 3000 times between 260° and 1010° C but not when cycled between 150° and 870° C. (CoC-Ni)-TaC was relatively undamaged after 3000 cycles to 1000° C, but showed severe damage when cycled 3000 times to 1120° C.

A nickel base alloy (Ni-10Cr-5Al-1Ti-14.9Ta-1.1C) was reported (ref. 33) to have its fiber strength at 1000° C reduced by the nucleation on the monocarbide fibers, during deformation, of needles believed to be $M_{23}C_6$. Cracking of the MC fiber in association with the $M_{23}C_6$ precipitate is shown in figure 12.

The M-MC alloys have been shown to have excellent stress rupture properties (see fig. 8). The alloy Co-20Cr-10Ni-TaC has greater strength than even $\gamma' - \delta$ at Larson-Miller parameters above 55. The alloy Ni-TaC-13 has greater strength than $\gamma/\gamma' - \delta$, at least up to a L-M parameter of about 53 (ref. 39). Another virtue of the M-MC alloys is their high strength transverse to the growth direction. For example, in COTAC-3 the transverse tensile strength at room temperature was 87 percent of the longitudinal strength and at 800° C, 56 percent (ref. 27). The transverse elongation was 10 percent of the longitudinal elongation at room temperature and 130 percent at 800° C. High impact strength has also been reported for M-MC alloys. Over the temperature range from 20° to 1000° C notched specimens of the complex Co,Cr,Ni-TaC composite (COTAC-3) resisted fracture in impact tests (ref. 27). As stated previously the M-MC alloys are particularly attractive because of the great alloying flexibility they have shown. The matrix phases have varied from single elements to highly sophisticated superalloys. The microstructure of the complex alloy Ni-TaC-13 is shown in figure 13 (ref. 40). The microstructural flexibility of the M-MC alloys suggests growth potential to develop new alloys.

$M-M_7C_3$ - A fourth system that has shown promise as a turbine material is the ductile-brittle $M-M_7C_3$ system. It is best known as a cobalt-base alloy $(Co,Cr)-(Cr-Co)_7C_3$ (refs. 28, 29, and 30). The alloy termed 73C has the nominal composition of Co-41.0 w/o Cr-2.4 w/o C, although numerous other alloys along the eutectic trough develop similar structures. The 73C alloy contains 30 v/o of M_7C_3 carbide "fibers." The reinforcing phase is faceted in cross section and irregular in comparison to the well defined fibers observed in many M-MC alloys. A typical microstructure is shown in figure 14 (ref. 41). Because of its higher volume fraction of fibers, the $M-M_7C_3$ alloys tend to be more anisotropic than the fibrous M-MC alloys but less so than the lamellar gamma/gamma prime-delta or gamma prime-delta alloys. Figure 5 compares the longitudinal and transverse tensile strength of 73C at several temperatures (ref. 29).

The anisotropic toughness of $(Co,Cr)-(Cr-Co)_7C_3$ eutectic was studied by Thompson (ref. 42). The load-deflection curves for notched bend specimens tested in three orientations (fig. 15) are strongly dependent on orientation. In all three orientations the work of fracture decreased as interfiber spacing decreased. At a growth rate of approximately 20 cm/hr the Charpy V-notched impact strengths for the A, B, and C orientations were 7.1, 1.36, and 1.15 joules (5.2, 1.0, and 0.85 ft-lb). The A orientation had three times the impact strength of the cast cobalt base alloy MAR-M-302. The M-M_7C_3 alloys are also anisotropic in shear. Figure 16 shows the longitudinal and transverse shear strength of another cobalt base alloy (Co,Cr,Al)-$(Cr,Co)_7C_3$ (ref. 41). This alloy was considered for high temperature fasteners where both types of shear are important. It is less anisotropic in shear than $\gamma' - \delta$ which was also investigated as a fastener material (ref. 41). In both alloys the shear strength is much lower parallel to the growth direction than at right angles to the growth direction and the reinforcing phase.

At temperatures below 700° C the highest tensile strength in eutectic alloys has been observed in a (Co,Cr,Al)-$(Cr,Co)_7C_3$ alloy (see fig. 4). At room temperature its ultimate tensile strength is almost 1370 MN/m^2 (200 ksi). Except at values of the Larson-Miller parameters above 55 (Co,Cr)-$(Cr,Co)_7C_3$ alloy and its aluminum modification grown at 2 cm/hr have rupture strengths somewhat below the superalloy examples, directionally solidified MAR-M-200 and NASA-TRW VIA (see fig. 8). An exception to this is the one point shown for (Co,Cr,Al)-$(Cr,Co)_7C_3$ grown at 50 cm/hr which has strength equivalent to $\gamma/\gamma' - \delta$. The improved properties are attributed to matrix strengthening as a result of reduced fiber spacing at the rapid growth rate.

Nickel-base alloys have also been reinforced with M_7C_3 fibers. In a longitudinal section near the fracture of a room-temperature tensile test the matrix was intact but the fibers were cracked (ref. 7).

The ability to grow the M-M_7C_3 alloys at high rates makes these materials attractive from the processing point of view. In a study of carbide fiber reinforced cobalt-base eutectic alloys it was found that aligned structures could be grown in Co-Cr-C, M-M_7C_3 alloys at 12 to 18 cm/hr while in Co-Cr-TaC alloys alignment was obtained only at growth rates ≤ 0.6 cm/hr (ref. 43).

In the two important carbide reinforced systems just discussed some alloys have microstructural instabilities. These may be due to mismatch in coefficient of thermal expansion, a change in solubility of the reinforcing phase with temperature, a matrix phase transformation, or a phase reaction between the matrix and reinforcement. However, such alloys may nonetheless find use in ground based systems not subject to the repeated cycling of an aircraft engine. Or if the cost of growing aligned eutectics is reduced, they may be used for expendable airborne systems requiring a limited number of cycles.

Brittle-Ductile

The excellent oxidation resistance of ceramic materials would make them an attractive material to the engine designer, if it were not for their lack of ductility and impact resistance. We are concerned that a foreign object might strike one airfoil causing it to fail. This might lead to catastrophic failure by successive impacts on the undamaged blades of the airfoils previously broken off. To reduce the possibility that the failure of one ceramic airfoil in an engine could result in the failure of all the others, ceramic-refractory metal wire composites have been investigated. Synthesized composites of Si_3Ni_4 with W wires have shown substantial increases in impact resistance and resistance to thermal shock (ref. 44). In-situ composites of oxide-refractory metal systems are also being studied (ref. 45). Table I lists some of the combinations that have been examined. In the in-situ composites the refractory metal phase is fibrous and is typically less than 1 μm in diameter (refs. 45, 46, and 47). Although often short, the fibers have high aspect ratios.

One of the few systems for which mechanical properties have been measured is Cr_2O_3-Mo (ref. 45). This eutectic which melts at about 1760° C has 8.6 percent Mo fibers. At room temperature this eutectic had an impact strength 2.5 times as great as that of fine grained alumina. The work of fracture was about 20 times that measured for alumina. Some of this work of fracture must come from the deformation of the Mo fibers. Limited necking in the Mo fibers in the Cr_2O_3-Mo material occurred in room temperature fracture.

The experimental difficulties of growing aligned composites in oxide-metal systems are considerable. Two methods have been used. In one method (ref. 48) the melt is contained within a sintered rod of oxide and metal powder of eutectic composition. Because of the high radiant heat loss from the surface of the rod, the skin of the rod remains below the melting point of the eutectic. The molten zone can travel within the rod to produce an aligned composite. In a second method (ref. 47) presintered oxide-metal powders are sealed in a capsule of the metal to be grown as fibers. The desired structure is produced by induction zone melting. Until these or other processes are developed to produce larger amounts of oxide-metal eutectics, this type of material cannot be fully evaluated. However, the initial results are encouraging and warrant further investigation.

Other brittle-ductile eutectics are alloys that have an intermetallic compound matrix. In micrographs of CoAl-Co strained 4 percent at room temperature (ref. 49) cracks were evident in the brittle CoAl matrix and slip in the Co fibers. Despite the brittleness of the matrix this alloy when directionally solidified has been found to have room temperature tensile strengths as high as 585 MN/m^2 (85 ksi) and elongations as high as 6.2 percent. Another system which has ductile fibers in a brittle matrix is Ni_3Ta-Ni_3Al (ref. 50). Cracking in the brittle Ni_3Ta matrix and fiber-matrix debonding occurred in a tensile test at 700° C. Even the Ni_3Al fibers appeared brittle in SEM micrographs of the fracture at 700° C. The brittle fiber behavior at 700° C was attributed to the ductility minimum of Ni_3Al at this temperature. At higher temperatures this alloy shows greater tensile strength and lower minimum creep rates than IN-100. Although this brittle-ductile system does have some attractive properties at high temperatures, its properties would have to be outstanding for it to be preferred over an eutectic alloy having a ductile matrix.

Just as the ductility of the pseudo-binary $\gamma' - \delta$ Ni_3Al-Ni_3Nb alloy was surpassed by that of a ternary $\gamma - \gamma' - \delta$ eutectic, ductility in the Ni-Ta-Al system is greater in a ternary $\gamma - \gamma' - \delta$ Ni-N_3Al-Ni_3Ta eutectic than in the pseudo-binary (ref. 51). The structure consists of a matrix of gamma phase containing gamma prime precipitates with lamellae of both gamma prime and delta phases. The ternary has even greater tensile strength than the Ni_3Al-Ni_3Nb pseudo-binary, but its rupture strength is less than that of the Ni_3Ta-Ni_3Al pseudo-binary. The most attractive feature of the ternary alloy is its relative insensitivity to growth rate. Tensile strength appears to have a slight maximum in the vicinity of 40 cm/hr. Rupture life decreases slightly with increasing growth rates.

Brittle-Brittle

In the search for materials with ever higher melting points and better oxidation resistance it is natural to turn to ceramic systems. The experience now being gained with SiC and Si_3N_4 should allow designers of engines to consider other brittle materials such as oxide-oxide eutectics. So far the research on oxide-oxide systems has been rather limited and very little mechanical property information is available. Unless ceramic eutectics develop more thermal shock resistance than the boride or oxide systems such as SiB_6 or ZrO_2 and Al_2O_3 that have been studied (ref. 52), the likelihood of their being used as aircraft gas turbine materials is small. Because of the high melting points of ceramic eutectics, it will be difficult to develop shaped molds to contain them. Rather, shapes may be machined from directionally solidified rods or perhaps airfoil cross sections will be grown by techniques such as edge-defined, film-fed growth (EFG) which has already been used to grow Al_2O_3-ZrO_2 (ref. 53).

The Al_2O_3-ZrO_2 eutectic system has also been directionally solidified in a Bridgman furnace and in a gradient furnace (ref. 54). In both instances the charge was contained in a refractory metal crucible. Crack free material was obtained in the gradient furnace; the structure was lamellar and free from the colonies found in Bridgman ingots.

Al_2O_3-ZrO_2 (Y_2O_3) - An example of a brittle-brittle ceramic-ceramic system that has shown properties superior to ceramic materials prepared by conventional techniques is Al_2O_3-ZrO_2 (Y_2O_3) (ref. 55). When directionally solidified by a floating zone technique the eutectic consists of an Al_2O_3 matrix containing a whisker-like reinforcement phase of ZrO_2. Y_2O_3 was used to stabilize the ZrO_2 in the cubic form. Material solidified under plane front conditions at 0.8 cm/hr had a three point flexural strength of approximately 415 MN/m^2 (60 000 psi). Even though the structure was cellular at higher growth speeds the finer structure resulted in greater strength. In figure 17 the three point flexural strength at 1575° C of the eutectics solidified at 2.5 and 5 cm/hr is shown to be over 20 times as great as that of commercial polycrystalline Al_2O_3. Although this material is not as strong at lower temperatures, 1400° C, as hot pressed silicon nitride, it has greater strength at 1575° C (see fig. 18).

Zr_2O_3-Y_2O_3 - The eutectic of ZrO_2-Y_2O_3 has been directionally solidified by a floating molten zone technique (ref. 56). The lamellar spacing growth rate relation follows the frequently observed inverse square root law, $\lambda^2 R = 2.38\times10^{-6}$ cm/hr. However, bend strength appears to be independent of growth speed up to 40 cm/hr. At temperatures up to 1500° C three point bend strength which was about 241 MN/m^2 (35 ksi) at room temperature was almost independent of test temperature. Cracks were deflected by the ZrO_2 lamellae.

Other systems - A number of other oxide-oxide systems have been studied but very few mechanical property data have been reported. In the system $Al_6Si_2O_{13}$ (mullite)-Al_2O_3 two types of structures were obtained depending on the temperature to which the melt was heated (ref. 57). If the materials were heated 50° to 70° C above the liquidus and then solidified, the ingots consisted of more than 50 w/o aligned Al_2O_3 crystals surrounded by a SiO_3-rich glassy matrix. When heated just above the liquidus for less than 1/2-hour, a metastable solid solution of alumina in mullite was the only phase found. Compositions higher in Al_2O_3 than the eutectic solidified with fibers of primary Al_2O_3 in mullite.

The eutectic NiO-CaO has been grown in an aligned lamellar structure in an arc image furnace at rates between 1 and 12 cm/hr (ref. 58). The relation of interlamellar spacing λ and growth rate V is given by $\lambda^5 V$ = constant.

Some metallic systems have been reported to exhibit brittle-brittle behavior at room temperature. For example, in the Fe-Cr-Nb system a pseudo binary eutectic between an intermetallic compound and an Fe-Cr solid solution forms at the nominal composition Fe-13.3Nb-23Cr (ref. 59). Even at the slowest rate used (1 cm/hr) the structure was cellular with faceted fibers within the cells. At room temperature there was very little evidence of any plastic deformation in either phase prior to fracture. At temperatures above 700° C the minor phase exhibited some ductility or pseudo ductility. No fiber reinforcement was observed at low or high temperatures. As with brittle-ductile systems, it is unlikely that any such alloy would be used unless the strength were far superior to that of alloys with a ductile matrix.

CONCLUDING REMARKS

The future direction in which work on eutectic systems will advance depends not only on the alloy designers but also on the engine designers, on the thermodynamacists and on the process engineers. The degree to which engine designers will depart from their requirements for ductility will be influenced by current research with noneutectic brittle ceramics. For example, in the ARPA contract and other programs automotive and land based power gas turbines are being developed with SiC or Si_3N_4 vanes or blades. If this effort proves to be successful, then eutectic systems made up of intermetallic compounds previously not considered because of their low ductility may also become accepted engineering materials.

The thermodynamacists and phase diagram metallurgists will, with the aid of computer calculated phase diagrams, provide information on ternary, quaternary, and higher order systems in order to identify new compositions with potential as turbine materials.

The process engineers will provide furnaces having steeper gradients. This will permit greater flexibility in the choice of alloying additions or alloy purity because it will be possible to maintain aligned structures while increasing the difference between liquidus and solidus. They will also develop mold systems for containing high melting point or reactive materials. New techniques will be developed for making shaped sections. These may be merely improved methods for machining airfoils from cylinders or slabs or, for example, crucible and die materials will be identified which will permit the edge-defined, film-fed, growth (EFG) of airfoil shapes of ceramic or alloy eutectics for which no suitable mold material has been found.

Even without the assistance from these other areas of technology, alloy designers will make advances moving toward more complex alloys to optimize particular properties and maintain an acceptable balance between strength, oxidation resistance, and ductility. It is unlikely that any current eutectic alloy as it is now constituted will be used in engines. However, I am confident that we shall see some modification of a gamma prime-delta, a Co-Cr_7C_3, a CoTaC, a NiTaC, or a gamma/gamma prime-delta in operating gas turbines. We are far enough along so that some variant of the systems already identified will be used in engines. We can also be confident that many new alloys will be developed. Some of these new alloys may well be three phase ternary eutectics rather than the two phase binary eutectics which were our chief concern here.

To speak specifically of eutectic alloy development: in the near term the conservatism of the metallurgists will dictate that the matrix phase is a ductile face centered cubic solid solution. It will probably be nickel based, strengthened by gamma prime precipitates, unless a suitable precipitation hardening mechanism is found for cobalt. If the matrix is cobalt-base, it will be stabilized to the face centered cubic structure.

As process technology is developed for high melting point materials and the design techniques for more brittle materials are developed, eutectic systems involving high melting point intermetallic brittle-brittle systems will be used. Such intermetallic eutectic systems will probably have more ductility than oxide-oxide systems. The brittle-ductile, oxide-refractory metal, systems may well provide the last stepping stone necessary in the path to the use of fully brittle materials. Ultimately, and probably at first in unmanned or ground based vehicles, truly brittle-brittle ceramic systems will be used. Their use in engines which must be thermally cycled is contingent upon such materials showing better resistance to thermal shock than has been observed in bulk oxides studied to date.

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TABLE I

CATEGORIES OF IN-SITU COMPOSITES

Ductile-Ductile

Alloy	Second phase			Melting point, °C	References
	Composition	Form	v/o		
Ni-W	W	Fiber	6	1500	6,7
Ni,Co,Al-Cr,W	Cr,W(α)	Lam[a]			9
Ni_3Al-Mo	Mo	Fiber	26	1306	7
Ni_3Al-Ni_7Zr_2	Ni_7Zr_2	Lam	42	1192	7,11
Ductile-Semiductile					
γ-δ					
Ni-Ni_3Nb	Ni_3Nb,δ	Lam	26	1270	7,12,13,14
γ'-δ					
Ni_3Al-Ni_3Nb	Ni_3Nb,δ	Lam	44	1280	7,15,17,19
Hypoeutectic			32	1280	17
γ/γ'-δ					
Ni,Al-Ni_3Nb	Ni_3Nb,δ	Lam	$\sim$35	1272 to 1274	16,20,22,
Ni,Cr,Al-Ni_3Nb	Ni_3Nb,δ	Lam	$\sim$33	1244 to 1257	16,22,23
Ductile-Brittle					
Co-Co_3Nb	Co_3Nb	Lam	---	1270	26
Ni-Cr	Cr	Lam	23	1345	7,24
Ni-NiBe	NiBe	Lam	38 to 40	1157	7
Ni-Ni_3Ti	Ni_3Ti	Lam	29	$\sim$1300	7,25
M-MC					
Co-TaC	TaC	Fiber	16	1402	7,27,31,32
Ni-TaC	TaC	Fiber	$\sim$10	----	7,27,32,35
M-M_7C_3					
(Co,Cr)-$(Cr,Co)_7C_3$	$(Cr,Co)_7C_3$	Fiber	30	1300	7,28,29,30,42
(Ni,Cr)-Cr_7C_3	Cr_7C_3	Fiber	30	1305	7
Brittle-Ductile					
MO-M					
(ZrO_2,Y_2O_3)-W	W	Fiber	$\sim$6 w/o		48
(HfO_2,Y_2O_3)-W	W		$\sim$12 w/o		48
MgO-W	W				45,47
Cr_2O_3-Re	Re				45,47
-Mo	Mo		8.6	1760	45,47
-W	W				45,47
Ni_3Ta-Ni_3Al	Ni_3Al	Fiber	35	$\sim$1360	7
CoAl-Co	Co	Fiber	35	1400	7,49
Brittle-Brittle					
Al_2O_3-ZrO_2		Lam		1870	53
Al_2O_3-(ZrO_2,Y_2O_3)		Fiber		1890	55
$Al_6Si_2O_{13}$-Al_2O_3				1840	57
NiO-CaO		Lam		1720	58
$MgAl_2O_4$-MgO_2	MgO	Fiber	>50 w/o	1995	56
ZrO_2-Y_2O_3	ZrO_2	Lam	6.38 w/o		56
Fe,Cr-Fe,Cr,Nb	Fe,Cr,Nb	Fiber	22±1	1275±10	59

[a] Lamellar

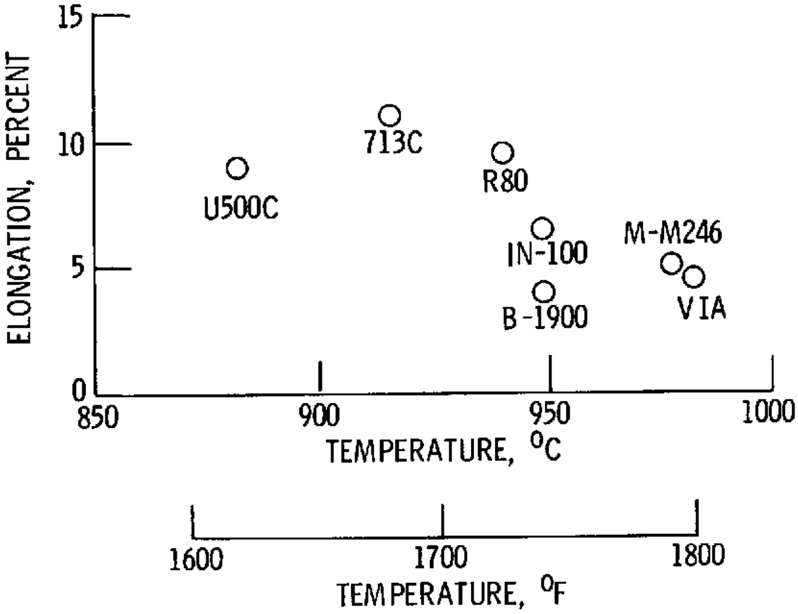
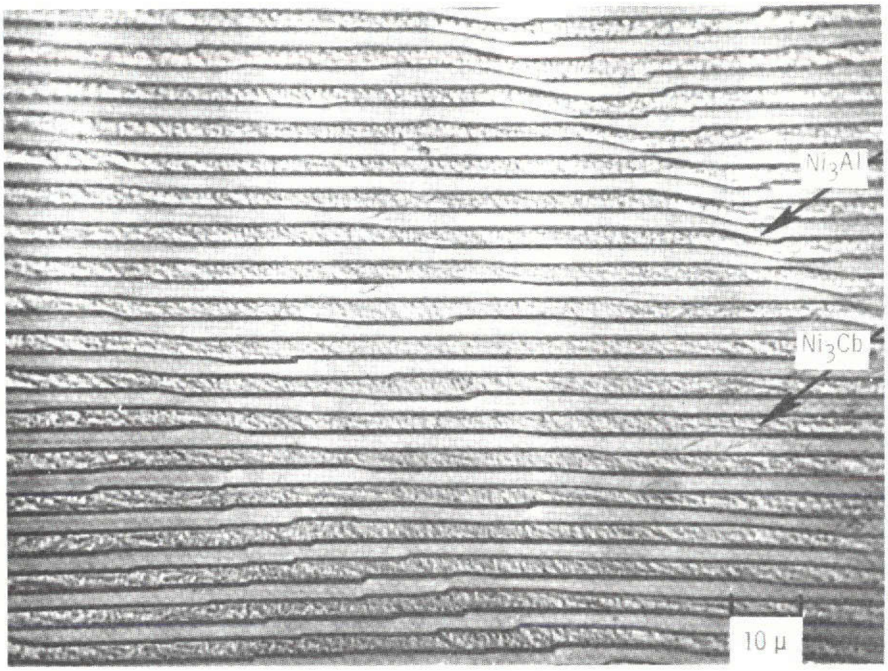


Figure 1. - Relation between tensile ductility at 760° C (1400° F) and use temperature for 1000-hour life at a stress of 138 MN/m^2 (20 ksi) for cast nickel base alloys (data from ref. 2).

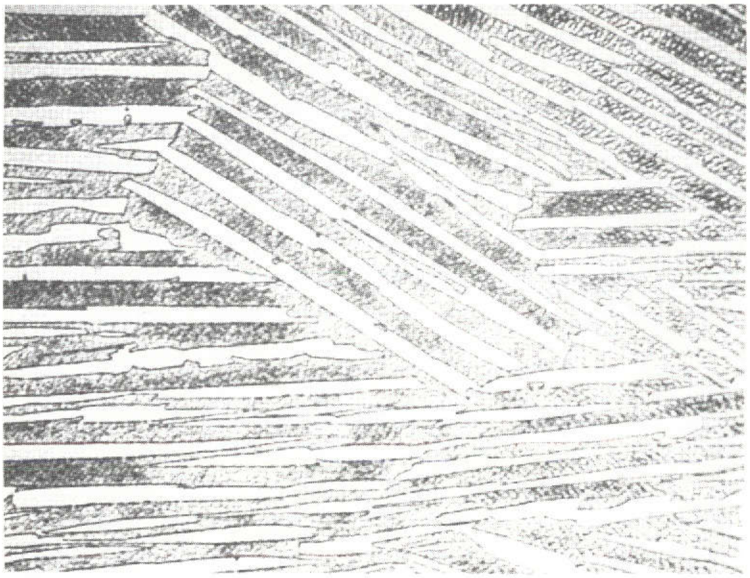


LONGITUDINAL

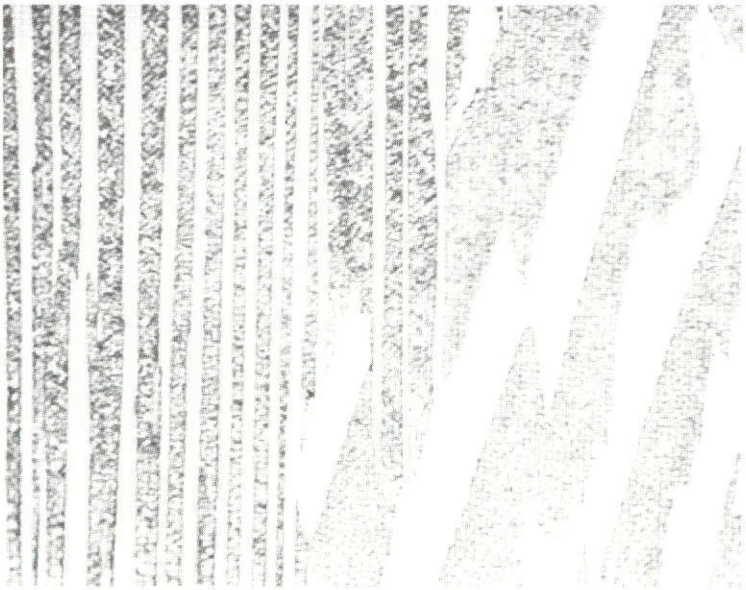
TRANSVERSE

Figure 2. - Microstructures of undirectionally solidified γ'-δ, Ni_3Al - Ni_3Nb; eutectic (courtesy E. R. Thompson).

E-7897



TRANSVERSE



LONGITUDINAL

Figure 3. - Microstructures of directionally solidified bivariant γ/γ'-δ eutectic, Ni 71.5 w/o, Cb 20.0 w/o, Cr 6.0 w/o, Al 2.5 w/o. (courtesy of F. D. Lemkey).

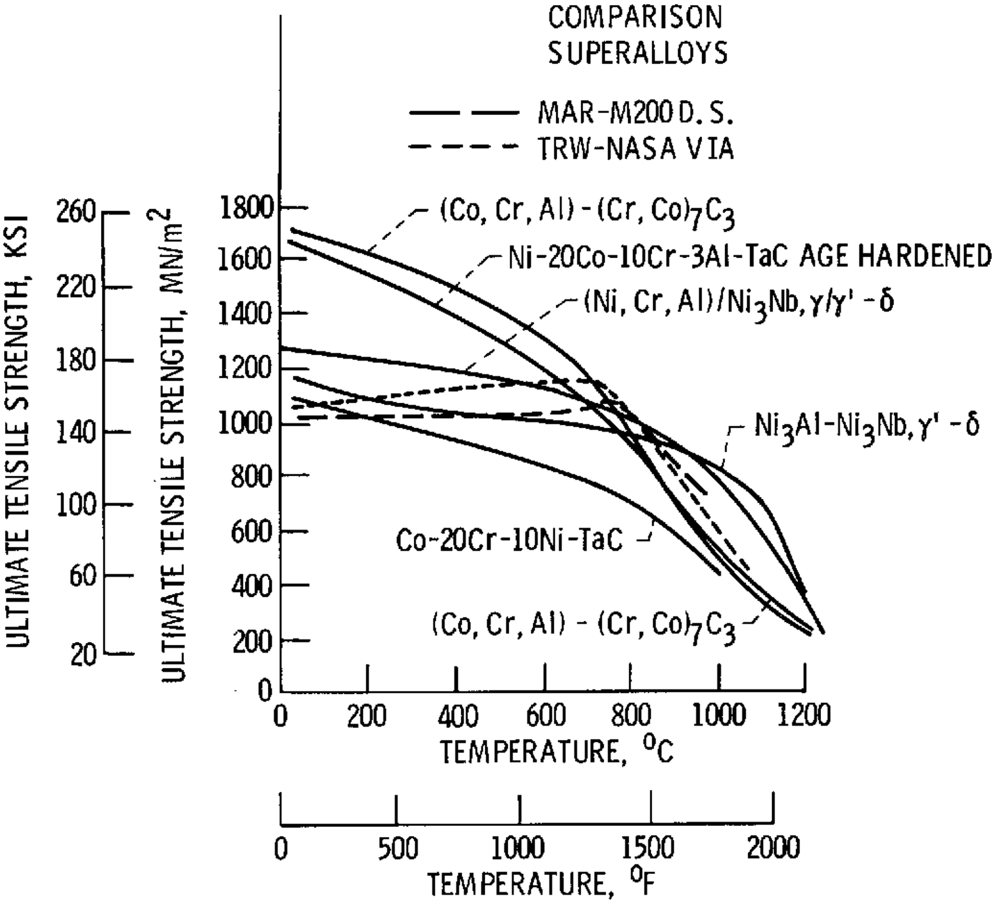


Figure 4. - Effect of temperature on tensile strength of eutectic alloys (after ref. 7).

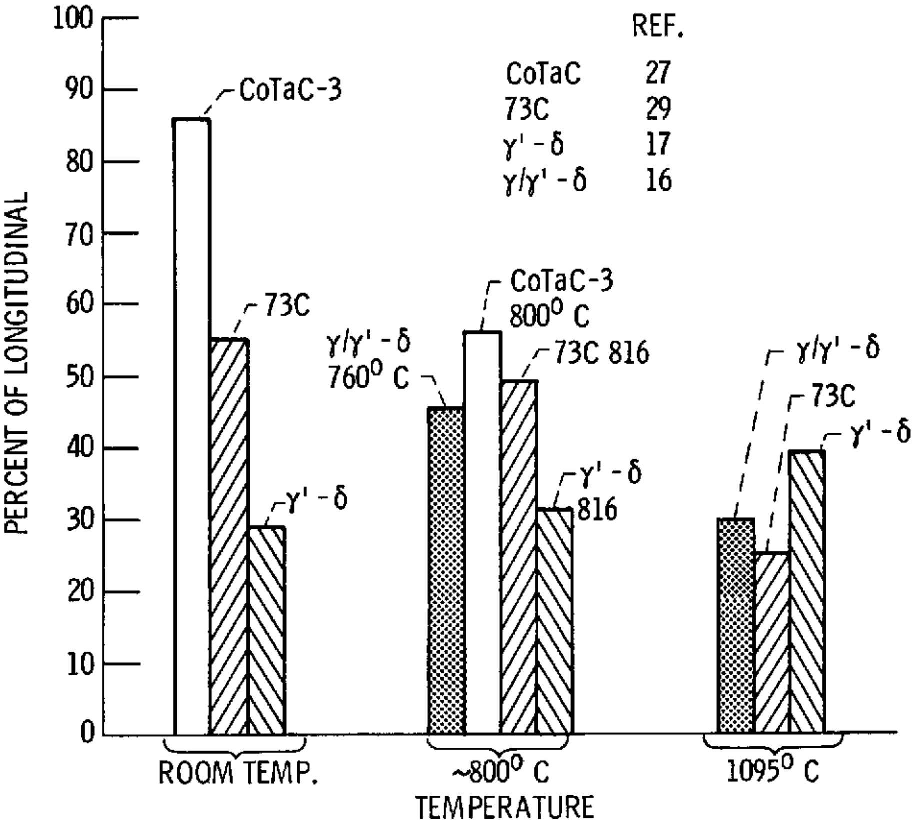


Figure 5. - Comparison of transverse and longitudinal tensile strength.

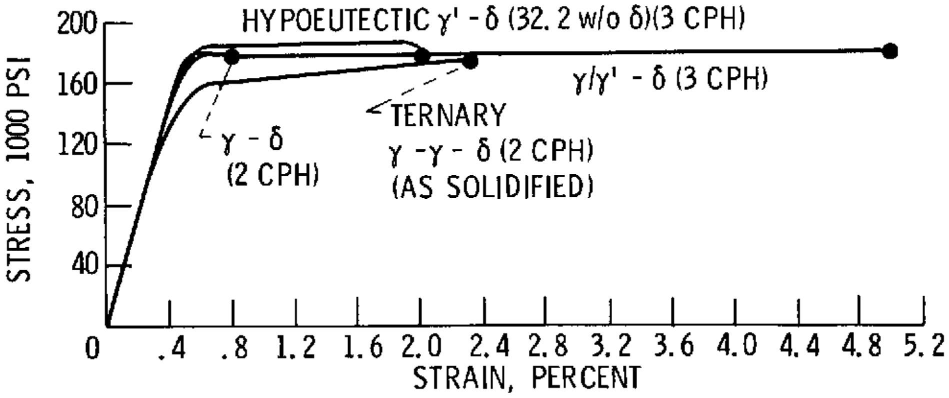


Figure 6. - Room temperature tensile behavior of Ni-Al-Cb eutectic alloys (refs. 16 and 17).

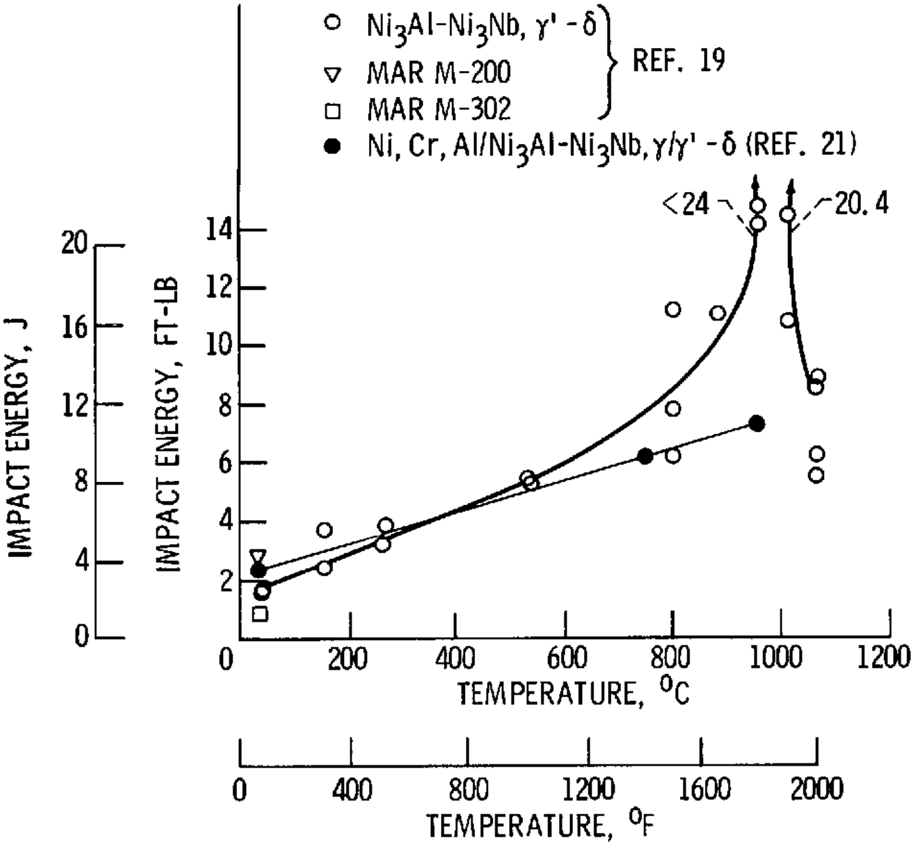


Figure 7. - Charpy impact behavior of γ' - δ and γ/γ' - δ.

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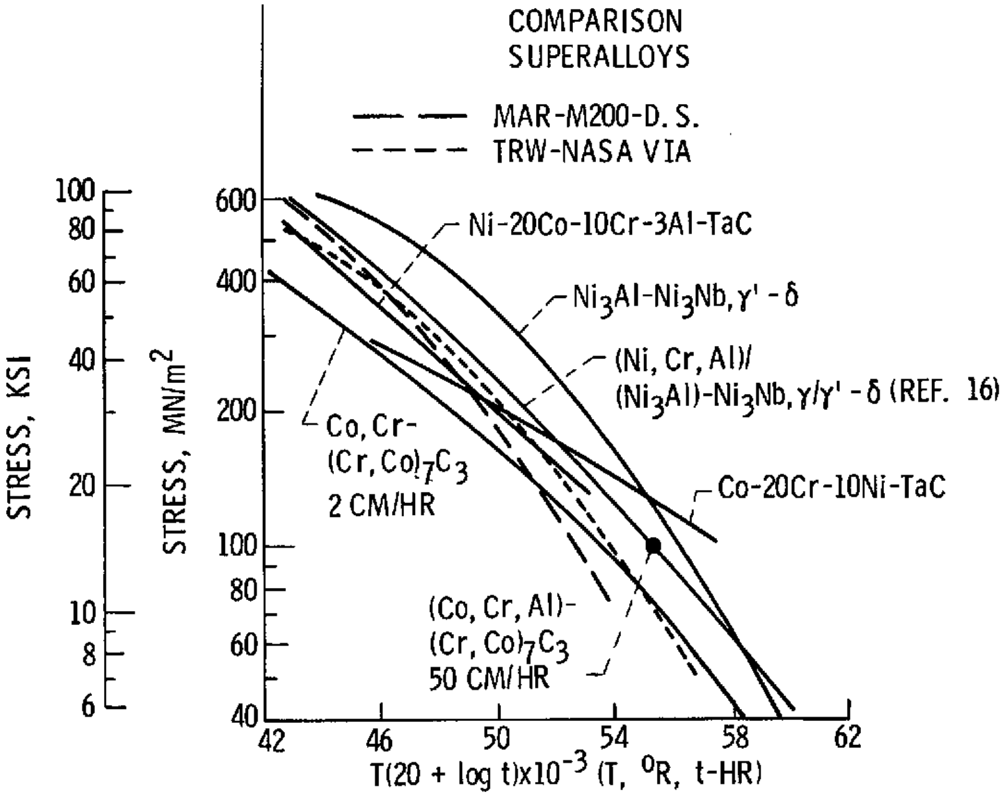


Figure 8. - Stress-rupture comparison of eutectic alloys (after ref. 7).

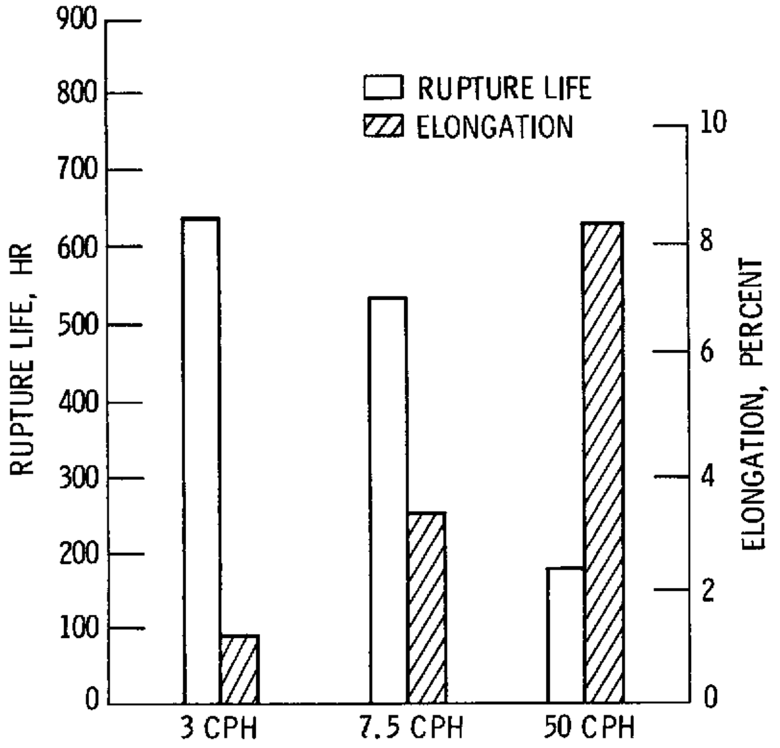


Figure 9. - Creep rupture properties 760° C/ 758 MN/m^2 (1400° F/110 ksi) versus growth rate (ref. 22).

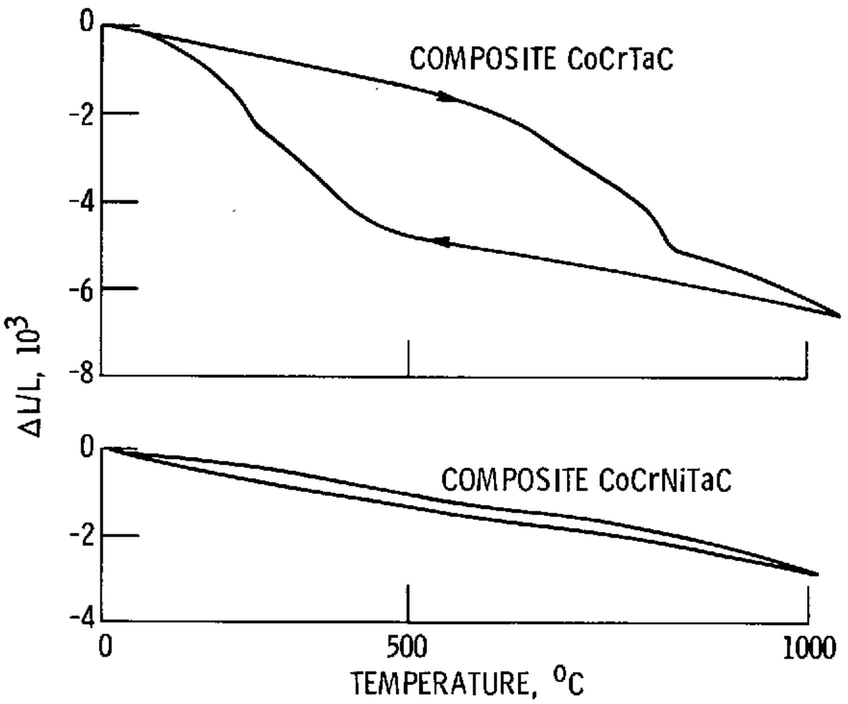
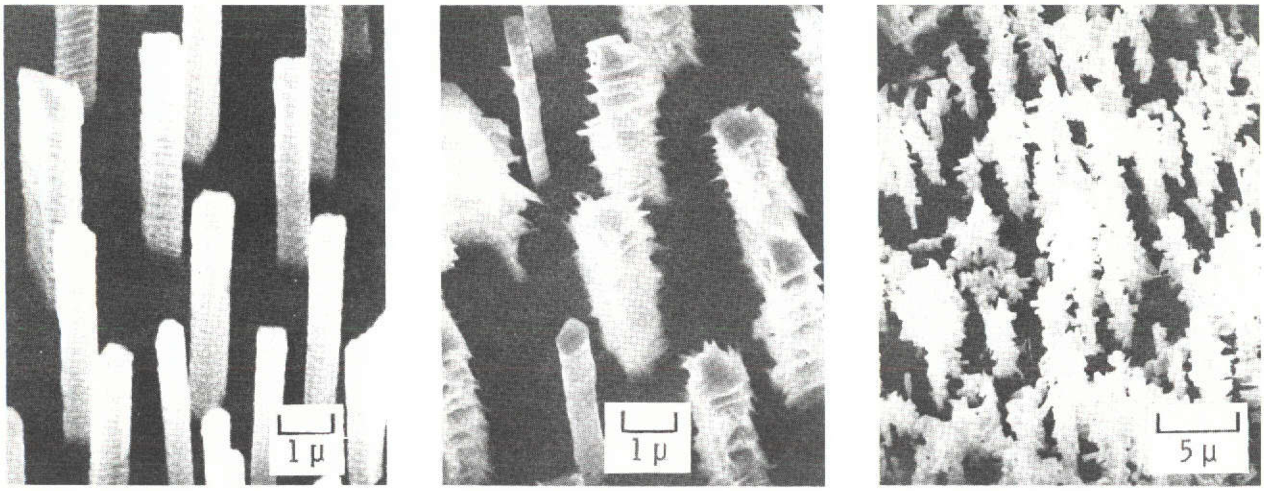


Figure 10. - Differential dilatometry of a Co, Cr-TaC and Co, Cr, Ni-TaC (COTAC-3) composite (ref. 27).



(a) 200 CYCLES. (b) 2000 CYCLES. (c) 5000 CYCLES.

Figure 11. - Structure of Co-15Cr-25Ni-TaC cycled between 427° and 1093° C (ref. 36).

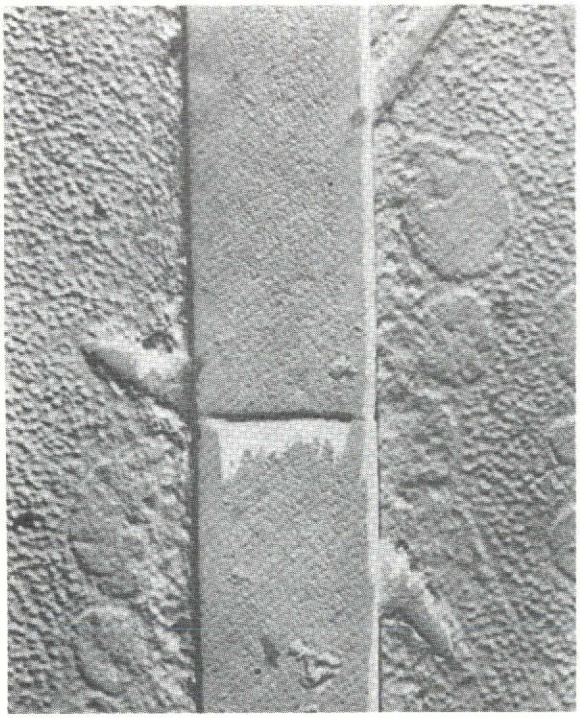


Figure 12. - Replica electron micrograph of γ-γ' matrix eutectic alloy deformed to 0.64 percent permanent strain at 1000° C (ref. 33). X23 000.

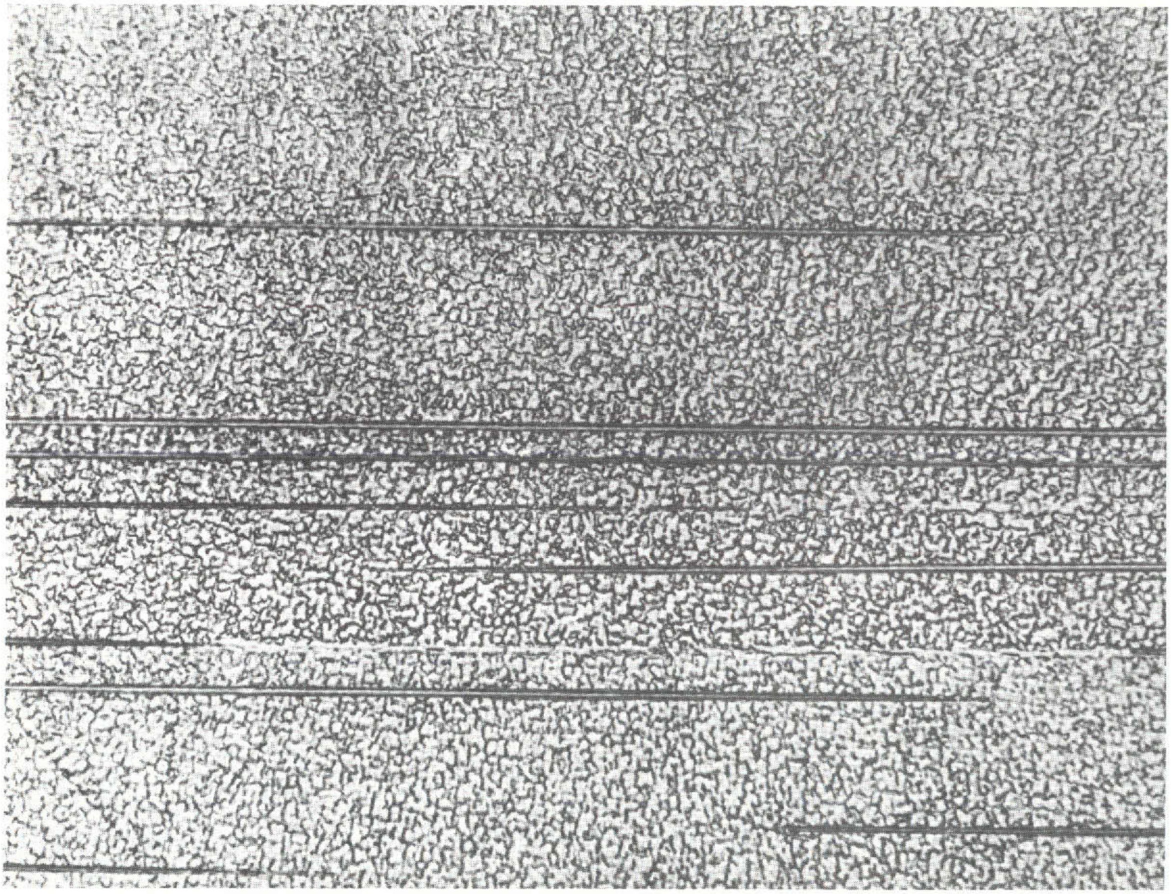
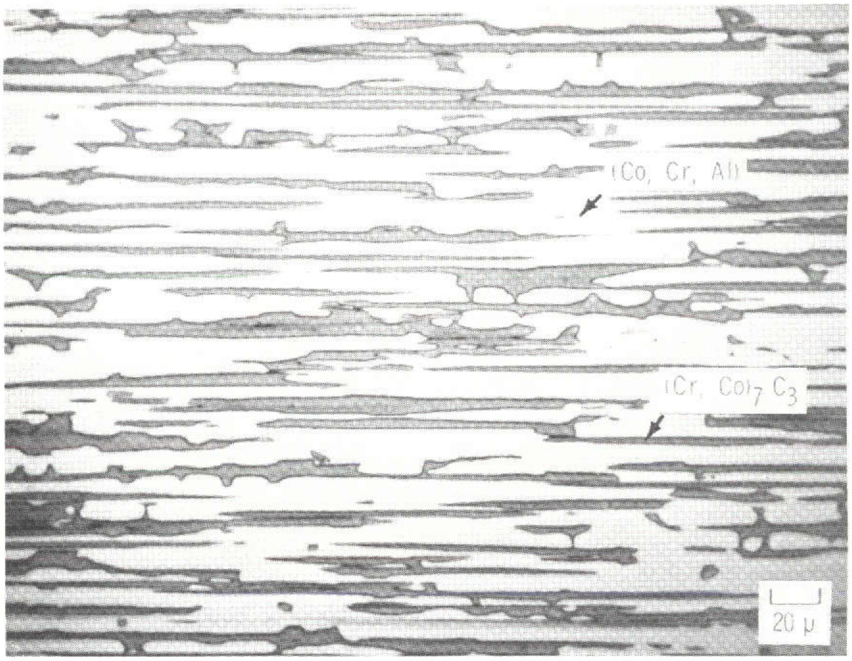
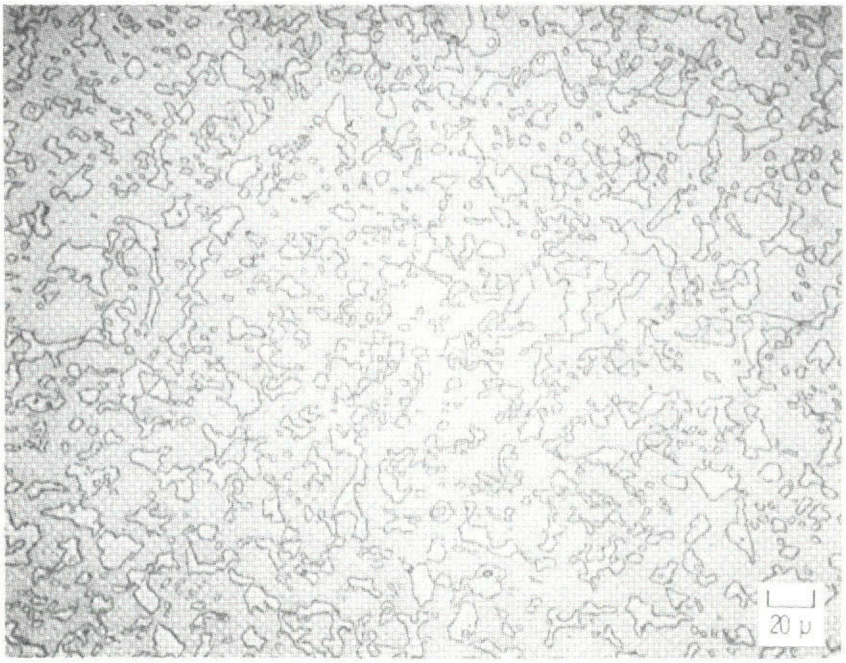


Figure 13. - As-grown NiTaC-13; longitudinal (ref. 40). X750.

E-7897



LONGITUDINAL



TRANSVERSE

Figure 14. - Microstructures of undirectionally solidified (Co, Cr, Al) - (Cr, Co)$_7$ C$_3$ eutectic (ref 41).

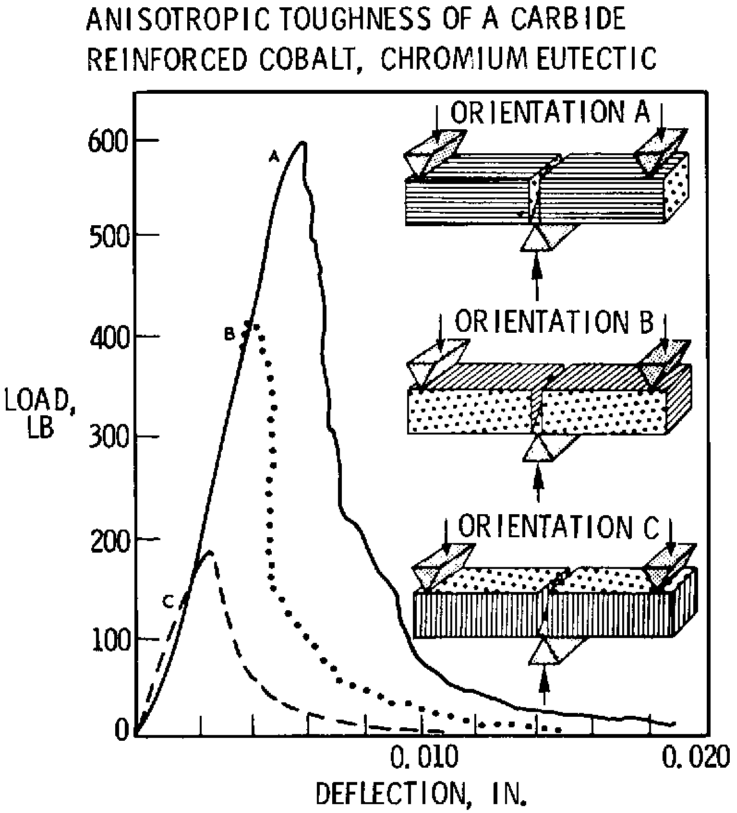


Figure 15. - Load-deflection curves for different orientations of the (Co, Cr)-(Cr, Co)$_7$C$_3$ eutectic grown at 3 cm/hr (ref. 42).

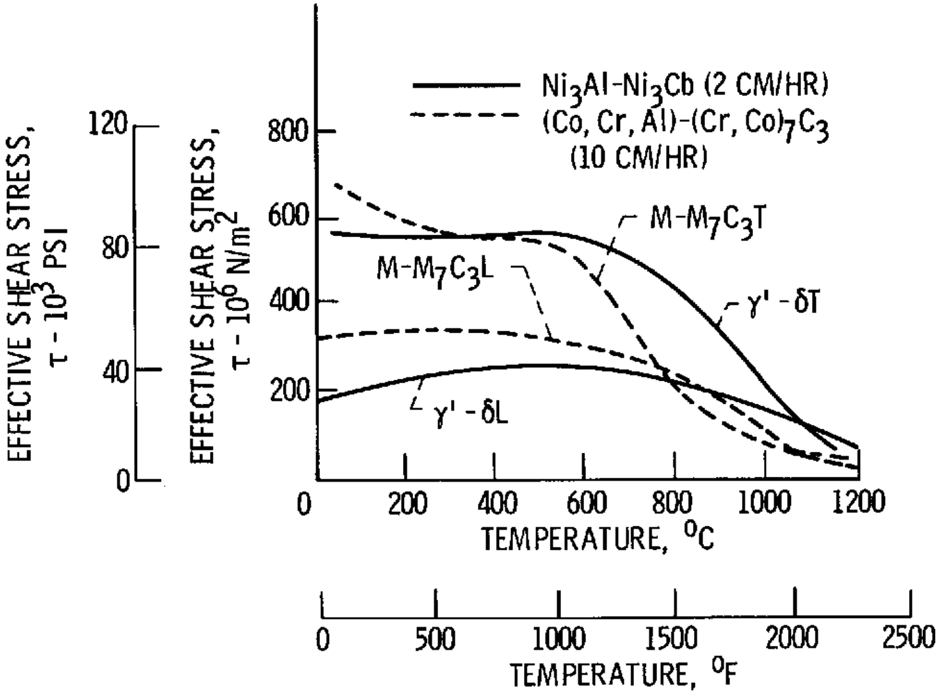


Figure 16. - Temperature dependence of transverse and longitudinal shear strength (ref. 41).

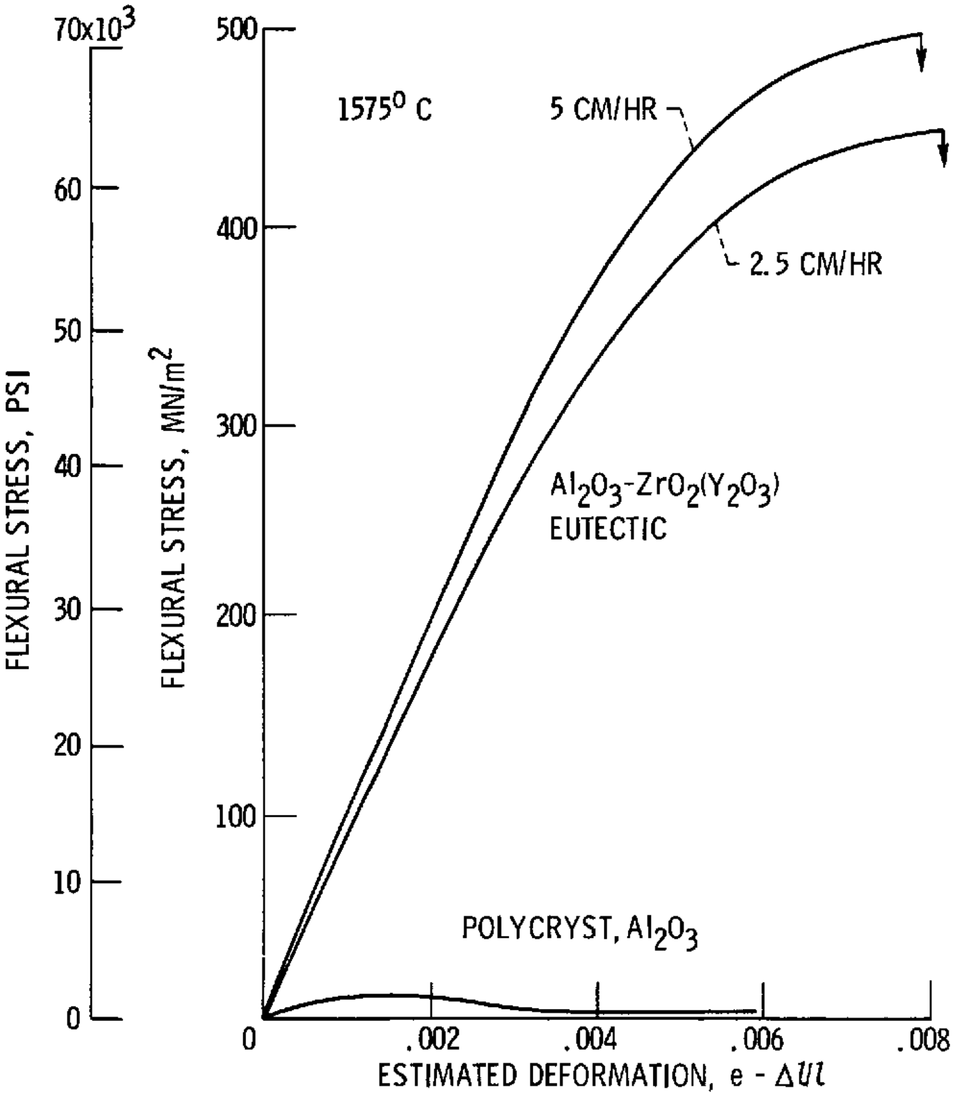


Figure 17. - Three-point bend strength Al_2O_3-ZrO_2(Y_2O_3) eutectic and polycryst, Al_2O_3 (ref. 55).

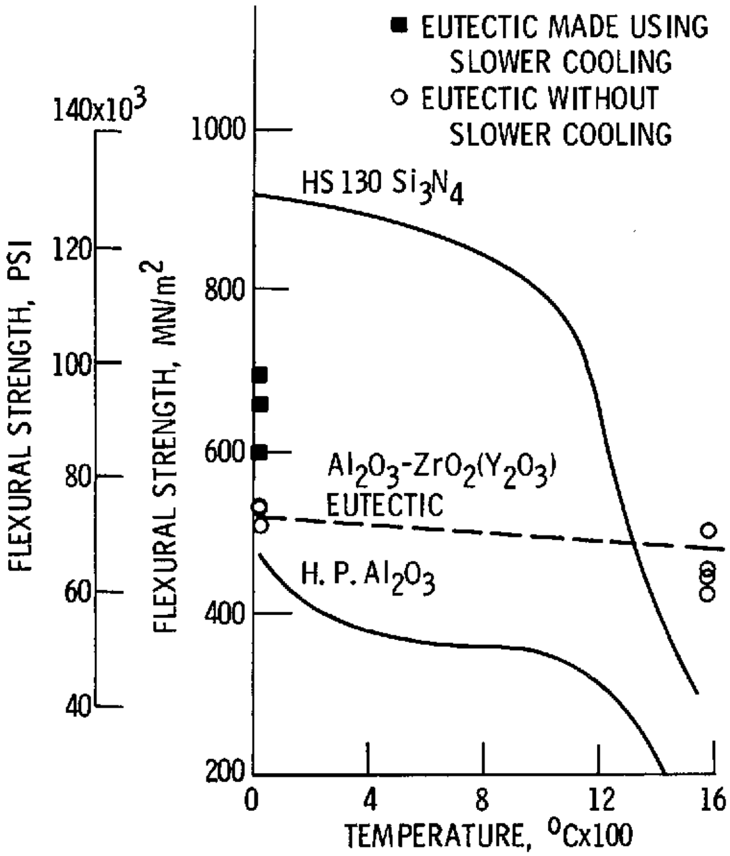


Figure 18. - Strength of unidirectionally solidified Al_2O_3-ZrO_2(Y_2O_3) eutectic compared with hot-pressed alumina, and silicon nitride (ref. 55).