# Superalloy Eutectic Composites With The VI A Refractory Elements - Cr, Mo and W

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## Abstract

With the progress achieved in $\gamma/\gamma'$-TaC and $\gamma/\gamma'$-$\delta$ eutectic superalloys, the eventual application of eutectic composites seems assured. While work on these systems is moving toward actual turbine hardware manufacture, work on exploratory new systems is underway to develop still further advanced composite superalloys.

Systems receiving considerable study now involve as the prime ingredients Ni, Al and the VI A refractory elements Cr, Mo and W. The phases of interest which may form from the liquid are $\gamma$ (fcc solid solution), $\gamma'$ (ordered fcc, $Ni_3Al$), $\beta$(CsCl structure, NiAl) and $\alpha$(bcc solid solution). Almost every combination of two phases from among these four possible phases has been studied in the range of the eutectic composition. Examples include $\gamma+\alpha$(Ni/Cr, Ni/W, Ni,Al/Mo), $\gamma'+\alpha$ ($Ni_3Al$/Mo), $\beta+\alpha$ (NiAl/Cr, NiAl/Mo,) and, with additions of Fe, $\gamma+\beta$ (Ni,Fe,Cr/(Ni,Fe)Al).

The region of interest in each of the ternary systems is bounded by $\gamma+\alpha$, $\beta+\alpha$, $\gamma+\gamma'$ and $\gamma'+\beta$ two-phase fields. The first three fields tend to form by eutectic reactions, and the last field by a peritectic reaction. However, the two phase field contained within these boundaries can be either $\gamma'+\alpha$ or $\gamma+\beta$. For NiAlW, $\gamma+\beta$ is observed, while for NiAlMo, $\gamma'+\alpha$ is observed. In the system NiAlCr, $\gamma+\beta$ is formed from the liquid, but is replaced at lower temperatures by $\gamma'+\alpha$.

By appropriate adjustment of the levels of Cr, Mo, and W in Ni, Al base superalloys, it has been possible to select eutectics which fall into either the $\gamma+\alpha$, $\gamma'+\alpha$ or $\gamma+\beta$ two-phase fields. Furthermore, other elements have been added. Elements such as Co and Fe tend to destabilize $\gamma'$ formation, but show extensive solubility in $\gamma$ and in $\beta$ through replacement of Ni. These additions tend to suppress the $\gamma'+\alpha$ phase field in favor of the $\gamma+\beta$ field. Elements such as Ti, Ta and Nb show extensive solubility in $\gamma'$ through replacement of Ni. These elements have very low solubility in $\beta$, and therefore tend to suppress the $\gamma+\beta$ field in favor of $\gamma'+\alpha$.

Phase equilibria will be reviewed for the eutectic fields involving $\gamma+\alpha$, $\beta+\alpha$ and $\gamma+\gamma'$. Emphasis of this presentation will focus on the phase equilibria involved in selection between $\gamma'+\alpha$ and $\gamma+\beta$ phase fields, as a function of eutectic chemistry. The resulting variations in eutectic morphology, phase volume fraction and eutectic behavior will be discussed.

## Introduction

Improvements in turbine blade and vane materials have come through combinations of new chemistries and new processing technologies (1, 2). The newest class of high-temperature metallic materials known as in situ composites, or directionally solidified eutectics, depend on such a combination. (3,4) Composite properties can be achieved by coupling a high strength reinforcing phase and a ductile matrix in arrays that range from fibrous to lamellar. Since the arrays are formed during solidification, morphological stability against long time high temperature service is possible. This is an improvement over conventional superalloys strengthened by precipitation where increasing temperatures lead to loss in strength due to dissolution of the precipitate (5).

Two aligned eutectic classes are closest to actual engine application. Receiving the greatest attention are the TaC fiber eutectics, with either a Ni-base or Co-base superalloy matrix (i.e., NiTaC-13, TaC-33), (6-9) and the lamellar $\gamma/\gamma'$-$\delta$ ($Ni_3Nb$) eutectics (10,11). Each of these eutectics is considered to offer substantial advantage over current superalloy behavior in terms of long time, high temperature, high stress service. Because it is expected that they represent the beginning of a series of new eutectic materials, exploratory studies continue to develop even better alloys. (12-15)

Eutectics are likely to prove most useful for specific hot section components of turbine engines, primarily blades or buckets and vanes or nozzles. The service environments for these components differ considerably, so that eventually different eutectic alloys will be used in each of the applications. Not only do required properties vary from blade to vane applications, but the environment for a blade in a jet aircraft engine creates different demands on the materials system than is the case for a blade in a marine turbine or in an industrial gas turbine. (16) At present, eutectics are under development principally in response to aircraft turbine requirements. This is due to the cost of alloy development and to the materials and processes involved in producing directionally solidified eutectics. Once the potential of eutectics is fulfilled in jet engine turbines, it seems almost certain that similar alloys will find application in other turbine environments.

Conventional superalloys now in use rely heavily upon Al for two functions, strength from $\gamma'$ precipitation, and oxidation resistance from formation of an adherent oxide layer. To meet the advanced powerplant goals of higher temperature and longer lifetime service to attain better fuel economy, improved performance and less maintenance, new Ni-base materials will almost certainly contain Al as a key ingredient. The refractory elements Cr, Mo and W form a number of different eutectics with Ni, Al-base alloys, and some of these are the most promising superalloy composites currently being investigated. The purpose of this paper is to describe the physical metallurgy of several of these systems and their potential for application.

## Phase Equilibria for the Ternary Systems

To facilitate later discussion of alloying element influence, a description of phase equilibria for each ternary system, NiAlCr, NiAlMo and NiAlW will be helpful. Reactions from the liquid state will receive most attention, although mention will be made of some of the solid state reactions.

All three of the ternary systems contain several phases in common:

(a) The face centered cubic (fcc) Ni-base $\gamma$ solid solution has substantial solubility for Al, Cr, Mo and W, and also for Co and Fe (17-21). Elements such as Ta, Ti and Nb also have extended solubility, but the solubility is decreased considerably at lower temperatures if Al is present ( 22-24).

(b) The ordered fcc ($L1_2$) $\gamma'$ phase is based on $Ni_3Al$. (17) This phase has a narrow variation about stoichiometry and limited solubility for Cr, Mo and W. (25,26) Solubility decreases in $\gamma'$ along the VI A column of the periodic table from Cr to W. Other elements, such as Ti, Ta and Nb, show substantial solubility in $\gamma'$ (by replacing Al), and help strengthen that phase (25-28).

(c) The intermetallic phase $\beta$, based on the CsCl structure, is nominally NiAl, but stoichiometric variations range from $NiAl_{.4}$ to $NiAl_{1.3}$. (17) The discussion will pertain primarily to $NiAl_{.4}$ to NiAl, since equilibrium in the Ni-base region is the subject of this paper. The $\beta$ phase has limited solubility for Cr, Mo and W, varying from about 10 a/o for Cr to the order of 1 a/o for W. (13,23,29) Solubility limits for elements such as Ti, Ta and Nb are also quite low. (30) For elements such as Co and Fe, substitution for Ni allows extensive solubility. (24,30,31) For example, CoAl and NiAl are completely isomorphous.

(d) The remaining phase of interest is the body-centered cubic (bcc) $\alpha$ solid solution of Cr, Mo and W. (17,23) The $\alpha$ phase has about 10 a/o or more solubility for Al, but the solubility for Ni is only about 1 a/o. Solubility of Ni in Cr is greater above 700°C, reaching 32 a/o at the $\gamma$+$\alpha$ eutectic at 1345°C. Complete solubility between Cr, Mo and W is observed at high temperatures, but two $\alpha$ phases form in the CrW system due to a miscibility gap at 1495°C and below. The solubilities of Co and Fe in $\alpha$ are similar to that of Ni, except for the extensive solubility of Fe in Cr. (17,21)

In the present discussion, the region of interest for each of the three ternary systems is bounded by the Ni-refractory binary, the Ni-NiAl portion of the Ni-Al binary, and the NiAl-refractory pseudobinary diagrams. Around the periphery of this triangular region of each system, several eutectic reactions occur:

$$L \rightarrow \gamma + \gamma'$$
$$L \rightarrow \gamma + \alpha$$
$$L \rightarrow \beta + \alpha$$

The $L \rightarrow \gamma+\alpha$ reaction in the NiAlMo system is displaced into the interior of the triangular region due to the presence of the $\gamma$+NiMo eutectic in the Ni-Mo binary system. (17,23) The equilibrium phase fields at about 1000°C are shown schematically in Figure 1.

In addition to the eutectic reactions which bound the triangular regions of interest shown in Figure 1, one of two other eutectic reactions is noted within the interior of each of the triangular regions (13,23,27):

$$L \rightarrow \gamma + \beta$$
$$L \rightarrow \gamma' + \alpha$$

As seen in Figure 1, the $\gamma$+$\beta$ and $\gamma'$+$\alpha$ phase fields do not co-exist, because the presence of one field intervenes between the two phases of the second field. In NiAlW, a $\gamma$+$\beta$ eutectic is formed and the $\gamma$+$\beta$ field is stable at lower temperature. In NiAlMo, a $\gamma'$+$\alpha$ eutectic is formed and the $\gamma'$+$\alpha$ field is stable at lower temperature. However, the situation in NiAlCr is quite different. A $\gamma$+$\beta$ eutectic forms from the liquid, but this phase field is unstable at 1000°C and below, and is replaced by the $\gamma'$+$\alpha$ field shown in Figure 1. (27)

The progression of eutectic reactions in the central portion of the ternary systems seems unusual. From the periodic table of elements, progression down the Cr-Mo-W column might be expected to be more continuous. The NiAlCr system exhibiting $\gamma$+$\beta$ at high temperatures and $\gamma'$+$\alpha$ at low temperatures is followed by $\gamma'$+$\alpha$ at all temperatures for NiAlMo. Thus it might be expected that NiAlW would also contain $\gamma'$+$\alpha$, rather than the observed reversion to a

$\gamma+\beta$ eutectic. However, the behavior is a consequence of the thermodynamic properties of the refractory elements. This has been demonstrated by Kaufman and Nesor who have calculated the phase relations for each of the three systems using available thermodynamic properties, and have correctly predicted the presence of $\gamma'+\alpha$ in NiAlMo and $\gamma+\beta$ in both NiAlCr and NiAlW. (32-35) The predicted phase relations also indicate correctly $\gamma'+\alpha$ field in NiAlCr at lower temperatures. However, the predicted $\gamma'+\alpha$ field in NiAlW at lower temperatures has not been observed. A recent study of the NiAlW system (13,30) verifies the existence of $\gamma+\beta$ at least above 800°C as reported in an earlier finding. (36) However, this work shows that the original diagram had incorrect phase solubility limits for W in the $\gamma$, $\gamma'$ and $\beta$ phases.

## Modifications of Phase Equilibria Through Alloying

The alternation between $\gamma+\beta$ and $\gamma'+\alpha$ eutectics in the ternary systems of Cr, Mo or W with Ni and Al suggests that modification of the systems can be accomplished by additional alloying. For example, substitutions of Mo for Cr will tend to destabilize the $\gamma+\beta$ field and allow the formation of the $\gamma'+\alpha$ eutectic. This is shown schematically in Figure 2. The central diagram is a quaternary isotherm illustrating the location of the two adjacent pseudoternary sections from Cr to Mo to $Ni_3Al$. The NiMo phase and the NiCrMo ternary phases have been omitted for simplicity. (14,17,21) At temperatures below 1000°C, a two phase $\gamma'+\alpha$ field exists from $Ni_3Al$/Cr to $Ni_3Al$/Mo.

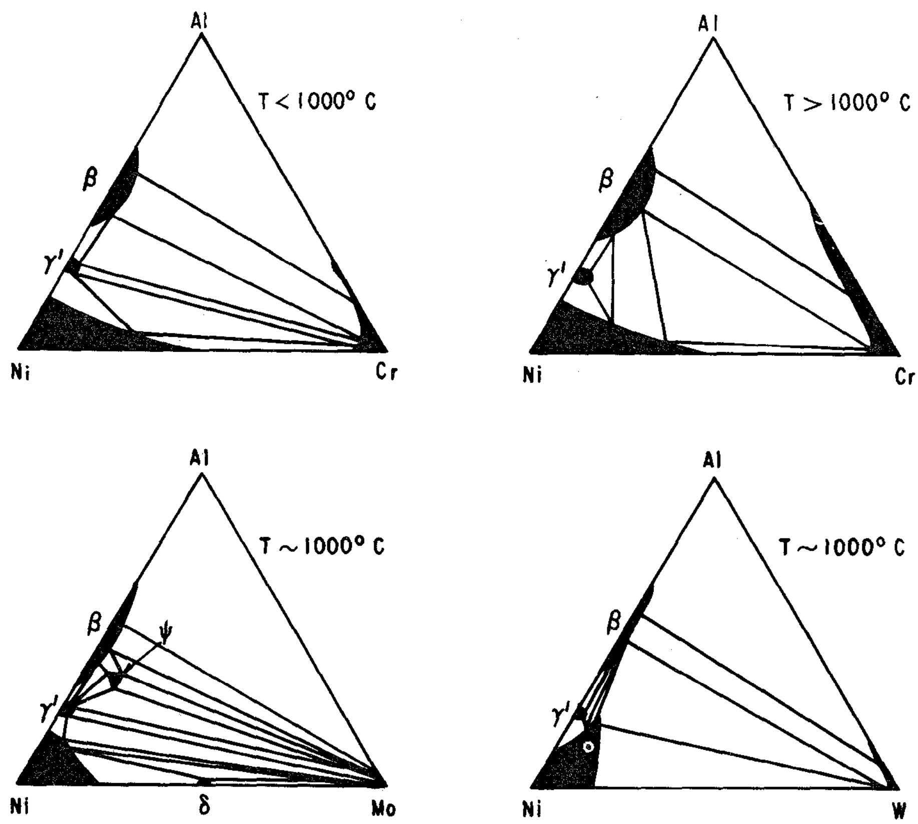


Figure 1: Schematic ternary phase diagrams for the NiAlCr, NiAlMo and NiAlW alloy systems at approximately 1000°C.

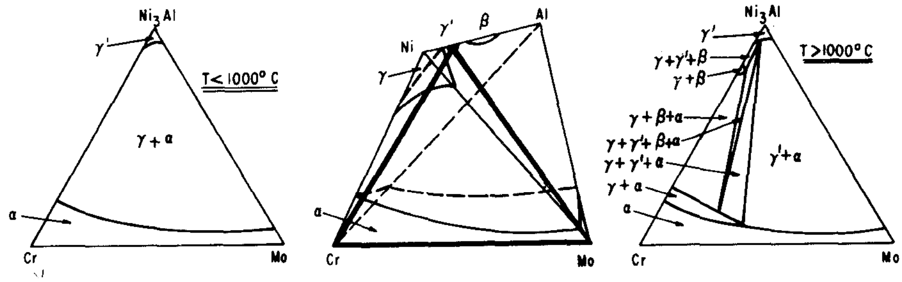


Figure 2: Schematic phase diagram for the ternary section from $Ni_3Al$ to Cr and Mo in the NiAlCrMo quaternary diagram near 1000°C.

However, above 1000°C, the γ+β field is stable for Cr-rich compositions. By partial replacement of Cr by Mo, the temperature for the reaction $\gamma+\beta \rightleftarrows \gamma'+\alpha$ can be increased. With sufficient Mo, the γ+β field can be completely suppressed, so that a stable γ'+α NiAlCrMo eutectic is formed from the liquid.

A parallel description can be offered for the additions of W to the NiAlCr system. Again, for simplicity, the miscibility gap between Cr-base and W-base α phases and the σ phase (14,17) have been omitted. In this case, as is shown in Figure 3, a eutectic reaction produces a γ+β field which is stable above 1000°C. Decreasing the temperature below 1000°C introduces the γ'+α phase field for Cr-rich compositions. Addition of W as a partial replacement for Cr will lower the temperature of the $\gamma+\beta \rightleftarrows \gamma'+\alpha$ reaction. With sufficient W, the γ'+α field can be completely suppressed, and a eutectic composition producing a stable γ+β microstructure can be achieved in the NiAlCrW quaternary system.

The flexibility of γ+β or γ'+α eutectic formation is not limited to substitution among Cr, Mo and W. For example, elements having a high solubility in β and low solubility in γ' will favor formation of γ + β eutectics, that is, will promote diagrams similar to those of Figure 3. Additions of Fe and Co to the NiCrAl system can completely suppress γ' and produce a stable γ+β eutectic. (13) Substantial additions of Fe and Co have a similar influence on the NiMoAl system, so that γ+β eutectics form. Elements having a high solubility in γ' and low solubility in β will have the effect similar to that shown in Figure 2. The γ'+α field will be promoted and the γ+β field will be diminished. Eutectics of γ'+α have been produced in the NiCrAlTi systems. (30) In this case, the correct balance of Ti and Al is necessary to produce the γ'+α structure. If insufficient Ti is used, an unstable γ+β eutectic results. Elements such as Ta or Nb also tend to expand the γ' phase field in the quaternary space to promote formation of a γ'+α eutectic behavior.

Alloying with quaternary additions can be used to alter eutectics other than γ+β and γ'+α in the Ni, Al refractory element systems. For example, Fe added to the Ni-W γ+α eutectic allows substantial reduction in the W necessary

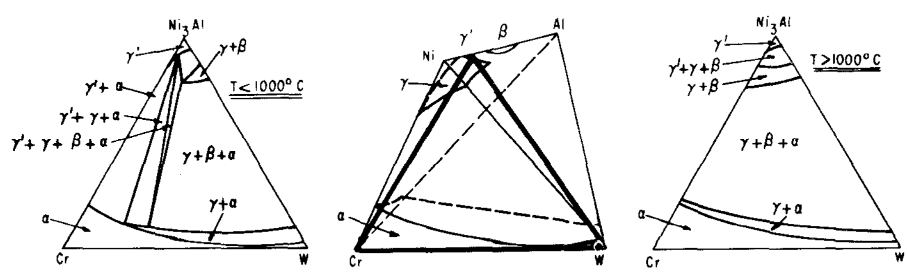


Figure 3: Schematic phase diagram for the ternary section from $Ni_3Al$ to Cr and W in the NiAlCrW quaternary diagram near 1000°C.

to produce the eutectic. (30). The fcc form of Fe dissolves about 1 a/o W, compared to 17.5 a/o W in Ni. Addition of Fe results in a drastic reduction of W solubility in $\gamma$(Ni,Fe) without changing the nature of the eutectic reaction. Substantial reductions in density result. In terms of a strength to density ratio, the Fe-containing alloys are much superior to Ni-W at low temperatures, and equivalent at temperatures of about 1100°C.

Another example of eutectic modification is noted by Walter and Cline. (37,38) The NiAl-Cr $\beta$+$\alpha$ eutectic consists of circular cross-section Cr rods in a NiAl matrix. Additions of Mo change interfacial misfit, producing a faceted square cross-section rod. Additions of as little as 5 a/o Fe result in even a more dramatic change in structure. (13) The eutectic turns "inside out", that is, the Cr, Fe $\alpha$ phase becomes the matrix surrounding $\beta$(Ni,Fe)Al fibers. This microstructural alteration offers the potential for transforming the $\beta$+$\alpha$ system from a brittle matrix to a ductile matrix situation. The plane of compositions in the NiAlCrFe quaternary system that produces the $\beta$+$\alpha$ eutectic structure passes through the quaternary in such a way that as the Fe content is increased, the Al content is decreased. The decrease in Al results in a similar decrease in the volume fraction of the $\beta$ phase, to such a degree that the $\alpha$ phase becomes the matrix.

## Eutectic Structures and Properties

The morphology of the binary eutectics of Ni with the refractory elements Cr, Mo and W have been treated elsewhere, and will be reviewed only briefly here. The Ni-Cr $\gamma$+$\alpha$ system and its modifications with W and Mo are lamellar, (14,39) while $\gamma$+$\alpha$ eutectics in the Ni-W system and its modifications with Fe and Mo form a microstructure of mixed fibers and blades. (30,40) The volume fraction of $\alpha$W is less than 0.10, so that a strong interfacial energy effect is likely to be operative to form the blade morphology. In the Ni-Mo

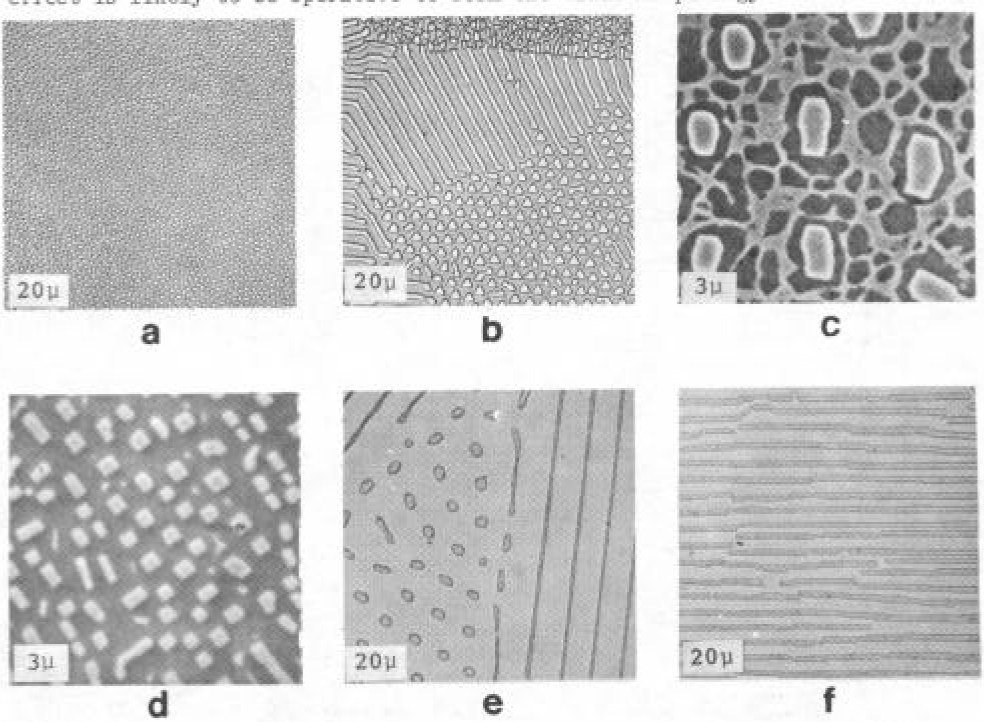


Figure 4: Microstructural variation in Ni, Al, VI A (Cr, Mo, W) eutectics: a) $\beta$+$\alpha$, NiAlCr; b) $\beta$+$\alpha$, NiAlCrMo; c) $\gamma/\gamma'$+$\alpha$, NiAlMo; d) $\gamma'$+$\alpha$, NiAlMo; e) $\gamma$+$\beta$, NiAlW; f) $\gamma$+$\beta$, NiAlCrFe.

system, $\gamma+\alpha$ does not form, but rather a $\gamma$+NiMo eutectic results. (17) The structure of this eutectic is irregular lamellar, as nearly equal fractions of the phases are present, but the phases are quite serpentine and intertwined, rather than alternating planar sheets. (41)

The eutectic composites in the Ni,Al+refractory element systems exhibit a wide range of microstructures, and several of the different morphologies are illustrated in Figure 4. As is expected, the systems with low volume fraction of the second phase tend to form rods. The rods can be round or square in cross-section, depending on the lattice relationships between the phases. For more strongly faceted situations, the rods may elongate in one dimension to become more blade-like. In the extreme, a low volume fraction phase may form a lamellar morphology, as in the $\gamma+\beta$ NiAlW system, with less than .08 volume fraction of $\beta$. As has been mentioned already, structural modifications are possible, such as the inverted $\beta+\alpha$ structures that result from additions of Fe to the NiAlCr ternary system. As with conventional superalloys, it is also possible to produce solid state precipitation, as with the $\gamma/\gamma'$ **matrix** of $\gamma/\gamma'+\alpha$ Mo.

A wide range of behavior also is observed for the different properties of interest. Solidification temperatures vary from 1500°C for the Ni-W binary eutectic to 1285°C for some compositions in the $\gamma+\beta$ NiAlCr eutectic region. Densities vary by nearly a factor of 2, from 6.3 g/cc for NiAlCr $\beta+\alpha$, to 11.3 g/cc for Ni-W. For turbine blades or buckets, density can be a major consideration, while for vanes and nozzles, it may be less important. Oxidation behavior also exhibits wide variation from eutectic to eutectic. Best behavior to date is seen in NiAlCr $\beta+\alpha$ and NiAlCrFe $\gamma+\beta$ systems. This is expected, since these alloys are very similar in composition to some of the most advanced NiCrAlY coatings being developed for Ni-base superalloy protection. (42) What is surprising is that $\gamma+\beta$ eutectics based on NiAlW, containing no Cr, are nearly as oxidation resistant at temperatures of 1100-1150°C. Alloys in the NiAlMo $\gamma/\gamma'+\alpha$ eutectics are less oxidation resistant, but this behavior can be improved significantly through alloying additions. (13)

Stationary parts of the turbine, such as vanes, are subjected to low stresses in service, primarily due to constraint in their mounting. The surface temperature of the vane can be quite high so that localized melting may occur. A certain amount of resistance to creep at these extreme temperatures is necessary to avoid "burn-out" of the vane or gross distortion and bowing. Thermal fatigue also may be critical. Vane alloys are subjected to severe oxidation environments, and must have exceptional resistance to oxidation or be coatable. Rotating blades, on the other hand, operate at lower temperatures but are subjected to high centrifugal forces. Internal cooling passages can give rise to stringent demands on material resistance to cyclic loading and to thermal fatigue. A detailed description of the eutectics discussed here in terms of all of the mechanical properties requirements for blades and vanes is beyond the scope of this presentation. Only a general treatment of the eutectic properties will be given.

A comparison of 1100°C ultimate tensile strengths is shown in Figure 5. The current best eutectics, NiTaC-13 and $\gamma/\gamma'-\delta$, are compared to the strongest eutectic alloys produced in the $\gamma/\gamma'+\alpha$Mo, $\beta+\alpha$Cr, $\gamma+\beta$ and $\gamma+\alpha$W systems. Density-normalized strengths are also indicated. On a strength basis, only $\gamma/\gamma'+\alpha$ compares favorably with the two systems being considered as potential turbine blade materials. Even after adjustment for density differences this is true, although the $\beta+\alpha$ and $\gamma+\beta$ systems hold some promise. The $\gamma+\alpha$W system pays a severe penalty as a result of its high density. For vane applications, the oxidation resistance of this alloy makes it unlikely to find use. The solidification temperatures of $\gamma/\gamma'+\alpha$ eutectics rule against their use as vane materials. However, both $\gamma+\beta$ and $\beta+\alpha$ eutectics look quite promising in terms of strength, oxidation resistance and solidification temperature. Low temperature ductility may limit the usefulness of $\beta+\alpha$. For reference, dispersion strengthened Ni-base alloys and cast Co-base alloys now considered as vane materials exhibit $\sim$100MN/m$^2$ 1100°C tensile strength.

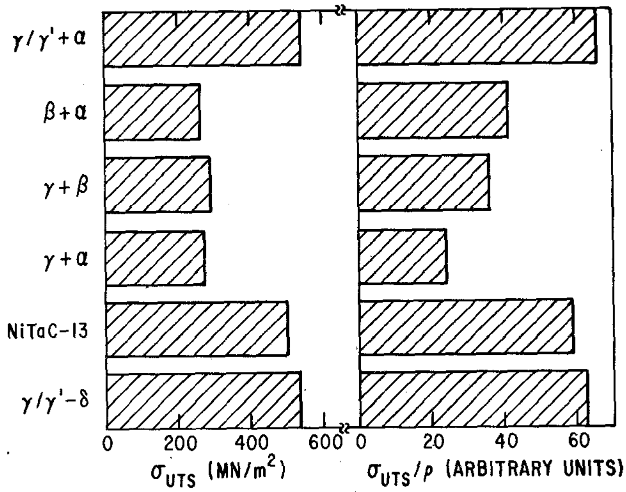


Figure 5: Ultimate tensile strength and density-reduced ultimate tensile strength comparisons at 1100°C for several Ni, Al-refractory element systems relative to NiTaC-13 and γ/γ'-δ

Of course, complete characterization of behavior under simulated service environment is necessary to fully evaluate the eutectic systems discussed here. Much of this evaluation is in progress, and improvements in behavior through alloy modification are still being made. The γ/γ'+α eutectics appear to be likely alternative materials for turbine blade application, and the γ+β and β+α systems are being pursued as possible turbine vane alloys.

## Summary

Eutectics in the Ni, Al - refractory element (Cr, Mo, W) systems offer the potential for high temperature turbine blade and vane applications. The ternary phase diagrams for NiAlCr, NiAlMo and NiAlW can be manipulated to alter the eutectic phases and morphologies by simple quaternary alloying modifications. The resultant combination of chemistry and structure can be controlled to produce behavior well-matched to hot section component materials requirements. Growth of these alloys in terms of higher temperature applications should be possible, in view of the simplicity of present chemistries and the ease with which aligned structures can be produced, relative to eutectics now in commercial development.

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