DEVELOPMENT OF A SECOND GENERATION DUCTILE/DUCTILE γ/γ' - α EUTECTIC ALLOY

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ABSTRACT

Ternary monovariant eutectics (γ'/γ - α and γ/γ' - α) in the Ni-Al-Mo system were discovered which consisted of mixtures of three ductile phases exhibiting improved longitudinal and off-axis tensile and creep ductility compared with the first generation eutectic superalloys (i.e. γ/γ' - δ and γ/γ' -MC) currently in PWA, GE, and SNECMA engine test programs. In general, the aligned α -Mo whisker phase of these composite alloys, produced *in situ* by directional solidification, was highly faceted and of square cross section. The crystallographic orientations of the interpenetrating α , bcc and γ/γ' , fcc lattices were found to be rotated about a mutual [100] growth direction by 45 degrees. Furthermore, the γ and γ' lattices were semi-coherent and the {110} planes of α , Mo and the {100} cube planes of γ' were found to form the square interfaces of the whiskers. As a consequence of this preferred crystallography, a low elastic modulus (E) of 19.6×10^6 psi parallel to phase alignment was exhibited. Moreover, the measured thermal expansion coefficients (α) of both the molybdenum and gamma prime phases were smaller than conventional superalloys, indicating that the thermal fatigue resistance of this preferred columnar orientation may be improved due to the reduction of geometrically induced $E \cdot \alpha \cdot \Delta T$ thermal stresses.

For ternary monovariant eutectic γ/γ' - α alloys containing 5.5-6.2 w/o Al, directionally solidified at 3 cm/hr under a liquid thermal gradient of approximately 300°C/cm, a 100°F improvement in creep-rupture strength over D.S. Mar M200 + Hf was demonstrated. Rupture ductility also was greatly improved with respect to d.s. superalloys, i.e. reduction in areas at 1400°F (760°C) of 48-50 percent were observed compared with values of ~15 percent for D.S. Mar M200 + Hf.

The phase equilibria limits of further ternary and quaternary alloying with Co, Fe, Cr, and Ta were investigated. For example, chromium and cobalt additions to ~9 percent by weight to the monovariant eutectic composition Ni-31.0 w/o Mo-6.2 w/o Al produced fully aligned structures under growth conditions identical to the ternary alloy. The effect of these alloy additions on the creep-rupture and corrosion behavior of this promising new turbine blade alloy candidate are discussed.

Introduction

Today's directionally solidified eutectic superalloys offer the potential for significant increases in turbine blade/vane temperature capability (i.e. 100-150°F). This can be compared with the steady improvement rate of perhaps 15°F/yr for conventional superalloys over the past 30 years. The ability to operate at elevated temperature is achieved through the design of multicomponent eutectic alloys where one phase serves as a matrix and the other aligned phase (phases) performs as the reinforcement. In addition to achieving high strength, these *in situ* composite materials exhibit excellent long term microstructural stability at temperatures exceeding 0.9 of their absolute melting temperature. Advanced superalloys are required to operate at these temperatures as well as to resist high applied mechanical stresses and fail, within the expected duration of service, in an acceptable manner.

Today's turbine blades and vanes, which are subjected to careful periodic inspections so that progressive damage is detected, are made of cast superalloys whose matrices are ductile face centered cubic nickel or cobalt. As the trend to run hotter and hotter turbines continues, designers have considered superalloys having greater use temperatures but possessing decreased ductilities. A significant improvement in rupture ductility and thermal fatigue has been shown from the directional solidification of certain superalloys which departs from this trend.⁽¹⁾ These more recent benefits derived from aligned grain boundaries and preferred orientation should also be obtainable in the design of eutectic superalloys of even greater temperature capability.

The two most promising eutectic superalloys (i.e. $\gamma/\gamma'-\delta$ and γ/γ' -MC) considered for engine hardware both consist of a strong ductile face centered cubic gamma matrix containing gamma prime, Ni_3Al , precipitates reinforced by aligned more brittle phases. Although possessing excellent stress rupture strength these eutectic superalloys exhibit reduced total elongations due in part to the plastic constraint of the matrix by the less ductile reinforcing phase. To improve the longitudinal and off growth axis ductility the design of a ductile-ductile eutectic superalloy was thought desirable. Relatively few high temperature ductile/ductile systems have been investigated and only the Ni-Cr system⁽²⁾ in any detail. This binary system however exhibited rupture properties considerably inferior to current cast nickel-base superalloys. Other systems based on gamma and gamma prime with the group VI-A bcc metals were considered for further investigation especially the Ni-Al-Mo system as no eutectic reactions between γ' and chromium or tungsten containing phases were indicated from previous equilibria studies of the Ni-Al-Cr⁽³⁾ and Ni-Al-W⁽⁴⁾ systems. Although contradictions in the location of the two phase $\gamma' + \alpha$ and three phase $\gamma' + \gamma + \alpha$ regions within the Ni-Al-Mo system were reported^(5,6,7), preliminary melts indicated a fibrous pseudo-binary eutectic reaction between $\gamma'-\alpha$ at 27 w/o Mo and 8 w/o Al.⁽⁸⁾ At this same point in time strong d.s. γ/γ' alloys solid solution strengthened by molybdenum were found by Maxwell⁽⁹⁾ which exhibited good high temperature strength and stability. These alloys containing 18 w/o Mo and 8 w/o aluminum (NX-188) were considered for turbine vane application. Further strengthening of this heat treatable γ/γ' matrix by increasing the molybdenum content to permit the generation and alignment of α Mo whiskers during directional solidification was undertaken. The purpose of this paper is to describe the compositional paths taken, the resultant unique microstructures observed, and the outstanding high temperature mechanical properties derived from reinforcement of γ/γ' by an aligned ductile phase.

Experimental Procedures

Master melts of the various Ni-Al-Mo alloys investigated were made in new recrystallized alumina crucibles in a Heraeus vacuum induction melting furnace powered by a 30 kW motor generator and pumped by a 25.4 cm (10 in.) vacuum system. The system was exhausted to approximately 10^{-5} torr (0.001 N/m^2) and then back-filled with high purity argon to provide a dynamic 200 liters per hr ($\sim 7 \text{ cfh}$) inert cover at atmospheric pressure. Power to the furnace coil was slowly increased until melting of the nickel, chromium and molybdenum or tungsten charge material was achieved. Aluminum was then added separately to the melt. Subsequently the melt was held at a constant temperature of $\sim 1400^\circ\text{C}$ (2550°F) for a 15-20 min homogenization period prior to pouring into copper chill molds.

Each resulting cast alloy bar was usually directionally solidified vertically within a nominally 1.27 cm (1/2 in.) diameter 99.7 percent recrystallized alumina cylindrical crucible whose wall thickness was nominally 2 mm. Vertical controlled solidification was accomplished in a high gradient apparatus.⁽¹⁰⁾ In this technique a known mass of alloy, typically 200 gms, contained in a both ends open or one end closed cylindrical alumina crucible, was positioned within the induction coil, water spray ring, and constant water level tank. With the impinging water spray, melting was accomplished by inductively coupling with the stationary graphite sleeve. Power requirements were established from empirical trials or experiments instrumented with thermocouples. Controlled freezing commenced by the withdrawal of the Al_2O_3 crucible through the water spray ring. In this setup thermal gradients in the liquid of approximately $250\text{--}350^\circ\text{C/cm}$ were routinely achieved. Excluding small end affected regions the rate of freezing was found to be equal to the velocity of crucible withdrawal over the six to ten inches of specimen length dependent on crucible diameter.

The liquidus and solidus temperatures of certain alloys in the Ni-Al-Mo system were determined using a differential thermal analysis apparatus. Heating and cooling of the sample was accomplished in argon at a rate of 3°C/min . The platinum vs platinum-10 percent rhodium thermocouple was calibrated against a pure silver standard and the accuracy of the temperature measured was within $\pm 2^\circ\text{C}$. The liquidus temperature was normally determined during cooling and the solidus during heating from the respective exothermic and endothermic inflections of the temperature-differential temperature traces.

Tensile specimens were ground from unidirectionally solidified ingots oriented in such a way that the load would be applied parallel to the growth direction. Tensile tests of 1.27 cm (0.5 in.) gage length and 0.36 cm (0.140 in.) diameter samples were conducted in accordance with ASTM specification E21-69 from room temperature to 1235°C (2200°F) in air using a Tinius-Olsen four screw machine at strain rates of 0.01 min^{-1} (elevated temperatures). Temperature, controlled to within $\pm 3^\circ\text{C}$, was monitored during each test. Ultimate strain and reduction in area values were made from measurements of the gage length and area before and after testing.

Stress rupture tests were performed at 1255 and 1310°C (1400 and 1900°F) in air. These tests were performed in accordance with ASTM E139-69 as applicable, with specimens of gage diameter 0.28 cm (0.110 in.) and gage length 1.27 cm (0.50 in.) with 5/16-18 threaded ends. Extension was measured during some tests on specimens containing ridges, and elongation and reduction of area were measured from the fractured halves.

Oxidation test coupons were cut perpendicular to the growth direction and ground and lapped through 600 grit paper and cleaned with trichloroethylene. The specimens were exposed for times up to 30 hrs at 700 and 1000°C in a laboratory box furnace and were weighed before and after exposure. Due to the relatively good behavior of alloys, the $\gamma'/\gamma-\alpha$ specimens were also evaluated in a noncyclic hot corrosion test. Here the specimens were coated with a 0.5 mg cm⁻² Na₂SO₄ and were oxidized in a thermal balance for 22-24 hrs at 1000°C in 1 atmosphere of O₂.

Results and Discussion

Microstructure and Phase Equilibria

To meet this study's objective of developing an isotropically ductile high temperature eutectic superalloy, the pseudo-binary $\gamma'-\alpha$ (Ni₃Al-Mo) eutectic(11) was selected for a compositional and mechanical property characterization and optimization. It was postulated that monovariant eutectic troughs extended from the previously identified $\gamma'-\alpha$ eutectic with decreasing aluminum content to a ternary eutectic and beyond. Numerous compositions that solidify in a coupled manner and result in microduplex composite structures were thus predicted. More than seventy ternary compositions(8) were examined to locate the monovariant eutectic trough, $L \xrightarrow{\Delta T} \gamma'/\gamma + \alpha$, the ternary eutectic $L \rightarrow \gamma + \gamma' + \alpha$, and the monovariant trough $L \xrightarrow{\Delta T} \gamma/\gamma' + \alpha$. The observed liquidus troughs, confirming these postulations, are shown in Fig. 1 projected upon the

1200°C isothermal section(6) for the Ni-NiAl-Mo ternary construction. The locus of compositions freezing according to these reactions were determined from the absence of primary γ , γ' or α phases in directionally solidified specimens polished longitudinally to reveal primary phase compositional segregation during directional solidification. The location of a ternary reaction point at

Ni-31.5 w/o Mo-6.2 w/o Al was found by the extrapolation of the liquidus temperatures, determined by DTA measurement (Table I) of the monovariant troughs. The slopes of the liquidus temperatures over the compositional range of interest, i.e. 1.1-9.0 w/o Al, were shallow thus preventing a more precise determination of the class of the reaction. Similarly the location of the pseudo-binary eutectic was located more closely to ~8.5 w/o aluminum rather than 8.0 w/o as previously determined microstructurally.(11)

Fig. 1. Isothermal section (1200°C) of the Ni-Al-Mo equilibrium diagram including the projected monovariant troughs.

Table I

Differential Thermal Analysis Data for Ni-Al-Mo Alloys

Composition (w/o)	Liquidus Temp (°C)	Solidus Temp (°C)	Reaction
Ni-26.9Mo-9.8Al	1305	1303	$L \xrightarrow{\Delta T} \gamma' / \beta + \alpha$
Ni-27.2Mo-9.0Al	1308	1305	$L \xrightarrow{\Delta T} \gamma' + \alpha$
Ni-28.0Mo-8.0Al	1309	1306	$L \xrightarrow{\Delta T} \gamma' / \gamma + \alpha$
Ni-31.5Mo-6.2Al	1310	1309	$L \xrightarrow{\Delta T} \gamma + \gamma' + \alpha$
Ni-34.6Mo-4.7Al	1313	1306	$L \rightarrow \gamma / \gamma' + \alpha$
Ni-36.8Mo-3.7Al	1313	1306	$L \rightarrow \gamma / \gamma' + \alpha$
Ni-39.7Mo-2.5Al	1318	1311	$L \rightarrow \gamma / \gamma' + \alpha$
Ni-42.3Mo-1.1Al	1322	1314	$L \rightarrow \gamma / \gamma' + \delta$

The microstructure of the directionally solidified γ' - α eutectic alloy consisted of a matrix of γ' , Ni_3Al reinforced by ~26 volume percent of faceted α , Mo whiskers which were primarily square in cross section as shown in Fig. 2. The density of this eutectic was 8.18 gm/cc. As the aluminum content was decreased from the amount required for the pseudo-binary eutectic composition, an increasing amount of γ phase containing precipitates of γ' became apparent between the α fibers which typically were encircled with the γ' phase as shown in Fig. 3. Even for alloys containing appreciable amounts of γ , the γ' phase tended to choose the α interfaces as preferred growth sites. The square or rectangular shaped interfaces indicated a strong interphase crystallographic relationship. The orientation relationships for several directionally solidified alloys including the $\gamma' + \alpha$, $\gamma' / \gamma + \alpha$ and $\gamma / \gamma' + \alpha$ phase regions were determined by transmission electron diffraction. The fcc and bcc lattices were in each case found to be rotated 45° around their [100] directions with respect to each other and are summarized below:

Fig. 2. Electron replica of section transverse to growth of Ni-27 w/o Mo-8 w/o Al (γ' - α)

Fig. 3. Electron replica of section transverse to growth of Ni-31.5 w/o Mo-6.2 w/o Al

Growth Direction	[001] _γ	[001] _γ	[001] _α
Interfaces	(100) _γ	(100) _γ	(110) _α
	(010) _γ	(010) _γ	(110) _α

The faceting exhibited by the α Mo fibers indicated that coarsening should not occur after elevated temperature exposure because of the low energy crystallographic constraint on the interface. After heating several alloys containing 7.8, 6.0 and 4.7 w/o Al to 1100°C for 1000 hrs no change in microstructure was observed attesting to the thermal stability of this unique system. Moreover, since the thermal expansion coefficients (α) of both phases were smaller than conventional superalloys, an improvement in the thermal fatigue resistance of oriented eutectic grains was suggested from the reduction of geometrically induced thermal $E\alpha\Delta T$ stresses.

The microstructure of various ternary Ni-Al-Mo alloys were further examined by quantitative metallographic techniques to measure the volume percentages of the γ , γ' and α phases and their size distribution. Table II⁽¹²⁾ indicates the changes in γ and γ' volume percentage with varying Al and Mo content. It was anticipated that as the monovariant troughs extend to greater Mo contents that the volume fraction of Mo fibers, i.e. 26, might increase. The apparent absence of such a trend can be explained from the increased partitioning of molybdenum atoms to the γ phase as its volume fraction increases with this trough composition movement. Molybdenum atoms were observed to be more soluble in γ than γ' at all temperatures.⁽⁹⁾

Chromium substitutions on an atom basis for molybdenum to the nominal ternary composition, Ni-31.5 w/o Mo-6.2 w/o Al were examined at one weight percent intervals to 9 w/o. For each of these levels (Fig. 4) of chromium modification plane front growth conditions were observed at a constant growth velocity of 3 cm/hr and a thermal gradient in the melt of ~300°C/cm. Chromium was expected to equally partition to both the γ and α phases as it was completely miscible in the former phases and relatively insoluble in γ' . The addition of chromium was also expected to improve the cyclic oxidation resistance of the alloy as will be discussed below. Cobalt and iron substitutions on an atom basis for nickel were examined at intervals to 9 and 5 w/o respectively to examine their effects on the γ' solvus temperature. Plane growth front conditions could be achieved for at least 5 w/o of each transition element substitution under the previously described growth conditions. Tantalum was substituted because of its extensive solubility in all three phases,

Table II

Volume Percentages α , γ' and γ in Various Ni-Al-Mo-Cr Alloys

Composition w/o	α (Mo)	γ' (Ni ₃ Al)	γ (Ni)
Ni-34.6Mo-4.7Al	27	48	25
Ni-31.5Mo-6.2Al	25	59	16
Ni-31.5Mo-6.2Al	25	64	11
Ni-31.0Mo-7.0Al	29	62	9
Ni-28Mo-7.4Al	26	60	14
Ni-31Mo-6.2Al-2Cr	27	59	14
Ni-29.5Mo-6.2Al-3Cr	23	57	20
Ni-29Mo-6.2Al-6Cr	19	60	21
Ni-27.5Mo-6.2Al-9Cr	32	45	23

γ , γ' , α , and to examine its solid solution strengthening effects. The presence of over five weight percent Ta destroyed the faceted α , Mo phase interfaces but aligned coupled growth was observed to 7 w/o Ta at a growth velocity of 3 cm/hr and a liquid gradient in the melt of $\sim 300^\circ\text{C}/\text{cm}$.

Mechanical Properties

The longitudinal ultimate tensile strength as a function of temperature for γ' - α specimens directionally solidified at rates of 2 and 3 cm/hr is shown in Fig. 5. The strength of this eutectic compares quite favorably with current d.s. superalloys and ductility values observed further exceed the comparison superalloys. The room temperature specimens failed with little necking and much crystallographic cracking after uniform elongation while the elevated temperature tests exhibited considerable necking. The results (Fig. 5) indicate a minimum in ductility ($\sim 18\%$) near 760°C (1400°F) typical to that experienced in current γ' precipitate hardened superalloys. The tensile strength of $\gamma'/\gamma-\alpha$, Ni-27 w/o Mo-7.6 w/o Al was additionally measured for specimens machined at various angles to the growth axis of a $1\frac{1}{2}$ diameter bar directionally solidified at 1 cm/hr under a gradient in the melt of $\sim 70^\circ\text{C}/\text{cm}$. The results of these tests at 760, 982, and 1083°C (1400 , 1800 and 2000°F) are presented in Fig. 6 and indicate a greater degree of strength isotropy for this system than that found in the $\gamma/\gamma'-\delta$ system. (13)

The molybdenum fibers of room temperature tensile specimens exhibiting over twenty percent elongation were observed to be unbroken except at the fracture surface. To examine further this notable ductility, molybdenum fibers were extracted from γ' - α using a refluxing 10 percent alcoholic bromine

Fig. 4. Electron replica of section transverse to growth of Ni-29 w/o Mo-6.2 w/o Al-9Cr ($\gamma'/\gamma-\alpha$)

Fig. 5. Temperature dependence of ultimate tensile strength and elongation for γ' - α (Ni-27.0 w/o Mo-8.0 w/o Al)

Fig. 6. Off axis temperature dependence of ultimate tensile strength of $\gamma'/\gamma-\alpha$ Ni-27 w/o Mo-7.6 w/o Al

solution for 4 hrs. The resulting molybdenum fibers were bent into circular loops and progressively tightened. The fibers displayed considerable plastic deformation (kinking) after 2 percent elastic strain was imposed as measured by their loop radius.

The creep rupture strengths of various ternary γ' - α , γ'/γ - α and γ/γ' - α alloys were measured in air to 1100°C and were found to be strongly dependent on aluminum content and phase alignment and spacing.⁽⁸⁾ The optimum aluminum content resided near 5 w/o aluminum. When β (NiAl) or δ (NiMo) precipitates form within the γ' and γ phases, respectively, a drastic reduction in rupture life results contrary to the improvement achieved from γ/γ' precipitates. The effect of increasing growth velocity and/or decreased thermal gradient was apparent in decreased rupture lives and was associated with cellular microstructures. A Larson-Miller parameter vs rupture stress curve of γ'/γ - α and γ/γ' - α alloys containing between 5.5 to 7.4 w/o Al, which were directionally solidified at 3 cm/hr and under a gradient in the melt of ~300°C/cm is presented in Fig. 7. A

~38°C (100°F) advantage in metal temperature capability is noted compared with the current state-of-the-art directionally solidified superalloy Mar M200 + Hf. Furthermore, from examination of the rupture ductility as measured in area reduction, the 760°C (1400°F) intermediate temperature % RA was of the order of 48% compared with 15% for D.S. Mar M200 + Hf. The combination of strength and ductility throughout the temperature interval RT to 1100°C (2012°F) exhibited by this simple three element system is indeed truly remarkable.

Fig. 7. Larson-Miller parameter - rupture curves of γ'/γ - α , γ/γ' - α and D.S. Mar M200 + Hf

The effect of chromium modifications on the creep rupture strength of the γ'/γ - α alloy containing 6.2 w/o aluminum was studied most extensively. Additions up to 3 w/o Cr (open circle point denotes 3 w/o Cr, Fig. 7) resulted in no appreciable change in rupture life measured at 1037°C (1900°F) and 30 ksi. However, at chromium levels above 3 w/o, a marked decrease in rupture life was noted as shown in Fig. 8. From examination of longitudinal sections through the fractured specimens, an accelerated coarsening of the α , Mo whiskers was observed for those specimens containing more than 3 w/o Cr. The coarsening resulted in reduced α , Mo whisker length and decreased aspect ratio (l/d) and may be associated with a rapid interface diffusion grooving mechanism.⁽¹⁴⁾

Oxidation and Sulfidation Properties

A preliminary evaluation⁽¹⁵⁾ of the oxidation behavior of various γ'/γ - α alloys was made on specimens representative of the transverse microstructure (perpendicular to the growth direction) wherein α , Mo fibers present a

continuous path to the surface being oxidized. A cyclic test of the γ'/γ - α alloys was conducted at 1000°C with specimens of each alloy exposed for 15, 2 hr cycles. The results are presented in Fig. 9. The oxide formed on the base alloy was primarily NiO and was largely nonadherent. The oxide formed on the chromium modified alloys was more adherent and Al_2O_3 rich. Some spalling of NiO, formed during the transient oxidation period was observed on both of these alloys. The oxide formed on the silicon modified alloy, while primarily NiO, was much more adherent than that formed on the base alloy, Ni-31.5 w/o Mo-6.2 w/o Al. It would appear therefore that additions of as little as 3 w/o Cr and substitution of Si to promote the formation of λ $\text{Ni}_2\text{Mo}_3\text{Si}$, are beneficial to the cyclic oxidation behavior.

Typical results obtained in the isothermal hot corrosion testing of γ'/γ - α alloys at 1000°C are illustrated in Fig. 10. These results obtained for the ternary Ni-Al-Mo alloy and a silicon modified alloy are presented. For all practical purposes, the weight change curves for the chromium modified alloys were identical to that of the ternary Ni-Al-Mo alloy. Replacement of some of the aluminum with silicon appears to result in some improvement in the hot corrosion behavior of the alloy but chromium addition to 9 w/o had little or no effect. A cyclic hot corrosion test was also performed at 900°C (1650°F)⁽⁸⁾ and was planned to continue for up to several hundred cycles. The results obtained in this test are consistent with those shown in Fig. 10. The cycling causes the thick, Ni-rich oxide formed at temperature to spall on cooling, resulting in large weight losses. Again silicon was found to produce beneficial effects, while chromium was ineffective.

Fig. 9. Cyclic oxidation behavior of γ'/γ - α alloys at 1000°C in air

Fig. 8. Effect of chromium content on rupture life (1900°F, 30 ksi) of γ'/γ - α alloys containing 6.2 w/o Al

Fig. 10. Hot corrosion behavior of Ni-31.5Mo-6.2Al (γ'/γ - α) (O) and Ni-29Mo-2.75Si-2.5Al (γ/γ' - λ) (Δ) at 1000°C

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