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# STRESS RUPTURE BEHAVIOR OF A UNIDIRECTIONALLY SOLIDIFIED N1/N13A1-N13Nb EUTECTIC COMPOSITE

by

Xuan Nguyen-Dinh

#### A Thesis

Presented to the Graduate Faculty

of Lehigh University

in Candidacy for the Degree of Master of Science

in

Metallurgy and Materials Science

Lehigh University

1977

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This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science.

December 17, 1976 (date)

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#### ABSTRACT

The stress rupture behavior of a Ni/Ni<sub>3</sub>Al-Ni<sub>3</sub>Nb (Ni-21.5 wt. % Nb-2.5 wt. % Al) directionally solidified eutectic composite grown at 5 cm/hr was investigated in the temperature range 480 to 950°C.

The microstructure of the aligned eutectic composite consisted of Ni ( $\gamma$ ) lamellae strengthened by Ni<sub>3</sub>Al ( $\gamma$ ) precipitates and reinforced by Ni<sub>3</sub>Nb ( $\delta$ ) lamellae.

An attempt was made to relate the deformation and fracture mechanisms which occurred during stress rupture testing to the three deformation and fracture mechanisms identified by Bertorello, et al in their investigation of the hot tensile properties.

Under short time  $550^{\circ}$ C stress rupture (less than 100 hours), the eutectic composite deforms by slip in the  $\forall$  phase, by twinning in the  $\delta$  phase on  $\{211\}$  type planes and by cracking at the  $\delta$  twin.  $\delta$  matrix interface. Failure was caused by the link up of these cracks and by eutectic grain boundary delamination limited to a region near the fracture surface. Grain boundary delamination was observed to be the result of premature rupture of the brittle  $\forall'$  film at the eutectic grain boundaries which caused transverse cracks to be deflected. This Region I deformation and fracture behavior in stress rupture is similar to Region I tensile deformation and fracture behavior which was found to

operate in the temperature range 20 to 400°C.

At intermediate exposure time at  $550^{\circ}$ C, additional grain boundary delamination occurred away from the fracture surface. Deformation and fracture behavior in this Region II was also observed under tensile conditions from  $400^{\circ}$ C to  $690^{\circ}$ C.

Under long time exposure at  $550^{\circ}$ C cooperative twinning of the  $\chi$  and  $\delta$  phases occurred, resulting in the appearance of two variants of deformation bands. Bertorello, et al have also observed the same deformation behavior during tensile testing between 690°C and 926°C.

Minimum creep rates were measured in the temperature range 480 to 580°C and yielded a value of 380 kjoules/mole for the activation energy of the creep process. The significance of this result was discussed in terms of known activation energy data. It was proposed that the grain boundary delamination process might be rate-controlling during steady-state creep, and therefore would contribute to the creep of the eutectic composite.

Unlike the  $\forall -\delta$  system, at 750°C the stress rupture behavior of  $\forall/\chi'-\delta$  eutectic composite was characterized by a transition from Region I behavior to a void nucleation and void coalescence process.

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The 850°C stress rupture behavior and the long time 950°C stress rupture behavior were characterized by void nucleation and void coalescence, with the

highest creep ductility being related to the highest amount of voids produced during composite deformation.

A longitudinal section of a short time 950°C stress rupture specimen exhibited features characteristic of Region II tensile behavior.

#### CHAPTER I

#### INTRODUCTION

#### 1.1 Introduction to High Temperature Materials.

The development of Ni and Co-base superalloys has played an important role in the increased performance of the aircraft gas turbine (1). These materials derive their high temperature capability by several strengthening mechanisms: solid solution hardening by addition of Ta, Nb, Mo, W, Cr for example; dispersion strengthening by carbides of these elements; and age hardening by precipitation of ordered intermetallic phases such as  $\chi', \chi', \eta$  or S.

Conventional superalloy turbine blades have equiaxed grain structure. At high temperatures, the grain boundaries that are normal to the major stress axis are preferential sites for crack nucleation and propagation which results in the failure of the part. Directional solidification of superalloys provided a means to eliminate these grain boundaries. Indeed, nickel based alloys containing columnar grains and monocrystals were successfully produced by this novel process and were found to have longer creep rupture life, higher creep ductility and better thermal shock resistance than their conventional counterparts (2-5).

One crucial problem of conventional and directionally solidified superalloys is the resolutioning of the

strengthening phases upon exposure at high temperatures. From the standpoint of the engine designer, engine performance can be increased by increasing turbine inlet temperature. The phase stability problem concomitant to a good retention of strength and ductility seems to be solved in directionally solidified eutectics.

In the early sixties, Kraft and his co-workers (6-10) discovered that in-situ composites can be produced by unidirectionally solidifying binary eutectics. Coupled plane front growth, established under specific conditions of growth rate, temperature gradient at the solid-liquid interface and purity of the elements, resulted in an aligned lamellar or fibrous biphase anisotropic structure which exhibited a composite behavior. Unlike in man-made composites, microstructures of directionally solidified binary eutectics exhibited thermal stability when exposed to temperatures close to their melting points, due to the presence of low energy interfaces established under growth conditions approaching equilibrium (11-17).

Subsequently, coupled plane front growth was successfully achieved for other binary and pseudo-binary eutectics, for off-eutectic compositions which allowed variation of the volume fraction of the phases, and for monovariant eutectics which permitted variation of the volume fraction and composition of the phases (18-28).

Nevertheless, multivariant, multicomponent

eutectic systems (29-41) appeared to be the short term answer to the requirements of the turbine designer since the increased flexibility in varying the volume fraction and the chemistry of the phases provided a means to produce materials whose matrices can be strengthened by mechanisms similar to those found in conventional superalloys.

#### 1.2 Objectives of the Current Investigation.

In an extensive study, Gangloff and Hertzberg (24) identified three deformation and fracture mechanisms which govern the hot tensile and creep behavior of the unidirectionally solidified Ni-Ni<sub>3</sub>Nb eutectic composite.

In tensile testing between  $25^{\circ}$ C and  $600^{\circ}$ C and in short time  $600^{\circ}$ C stress rupture testing, the eutectic composite deforms by twinning of the  $\delta_{,}(Ni_{3}Nb)$  phase on  $\{211\}$  type planes and by twin boundary cracking. Fracture occurs by the coalescence of sub-critical  $\delta$ phase cracks.

Cooperative twinning of the  $\delta$  and  $\delta$  phases was found to operate in tension between  $600^{\circ}$ C and  $875^{\circ}$ C, during long time creep exposure at  $600^{\circ}$ C and short time creep testing at  $750^{\circ}$ C. Coalescense of sub-critical cracks along transgranular bands of  $\delta$  and  $\delta$  twins promoted fracture of the eutectic composite.

Under  $875^{\circ}C$  and  $1000^{\circ}C$  tensile conditions and upon long time creep exposure at  $750^{\circ}C$ , deformation was caused by void nucleation at lamellae ends and eutectic 6. grain boundaries while fracture was due to void coalescence into transgranular cracks.

It was concluded that, in the unidirectionally solidified Ni-Ni<sub>3</sub>Nb eutectic composite, there existed a time-dependent transition of the deformation and fracture processes in creep which was analogous to a temperature-dependent transition in tensile deformation and fracture, the transition in creep occurring at a shorter time as the testing temperature was increased.

In a recent study of the hot tensile properties of a  $\frac{1}{6}$  (Ni-21.5 wt% Nb-2.5 wt% Al) eutectic composite grown at 5 cm/hr., Bertorello et al (40) identified three regions of deformation and fracture behavior. In Region I, from 20°C to 400°C, deformation of the eutectic composite was caused by twinning and cracking of the  $\delta$ phase, and by slip in the  $\frac{1}{6}$  phase. This deformation behavior was similar to that observed at low temperatures in the  $\frac{1}{6}$   $\frac{5}{6}$  eutectic composite.

Furthermore, it was found that extensive grain boundary delamination occurred in a region near the fracture surface. The investigators proposed that premature rupture of the brittle  $\delta'$  film at the eutectic grain boundaries caused the transverse cracks to be deflected. Grain boundary delamination was found at the fracture surface and along the gage length of specimens tested in the temperature range from  $400^{\circ}$ C to  $690^{\circ}$ C.

In this Region II tensile deformation mode, it was found that the considerable ductility of 27% observed at  $550^{\circ}$ C could be related to the previously recognized ductility minimum of  $\gamma'$  and its key role in the delamination process. It was suggested that the critical temperature range 600-750°C associated with the ductility minimum of  $\gamma'$  strengthened superalloys could be depressed by addition of niobium.

Region III tensile deformation behavior, found to operate from  $690^{\circ}$ C to  $926^{\circ}$ C was related to the cooperative twinning of the 7 and 8 phases.

The purposes of the current investigation are threefold:

i) to establish the stress rupture response of the eutectic composite studied by Bertorello et al,
ii) to identify those mechanisms which promote deformation and fracture of the eutectic allow during stress rupture testing, and possibly to relate these mechanisms to those defined by the hot tensile properties investigation, and
iii) to measure the activation energy for the creep process in the temperature range where Region II behavior is operative.

#### CHAPTER II

#### EXPERIMENTAL PROCEDURE

#### 2.1 Melting and Solidification

Solidification procedures employed during this study were similar to techniques described by Gangloff (23) and Mills (26). Niobium of 99.8% purity, purchased in 1 cm. diameter bar was cut into cylinders which were subsequently ultrasonically degreased in methanol. Aluminium of 99.99% purity was cut into 1 cm. cubes, then cleaned by methanol. Nickel, purchased as 3 cm. X 4 cm. X 1.5 cm. slabs was pickled in a solution of 1 part  $H_20$ , 1.5 parts  $H_2S04$ , 2.5 parts  $HNO_3$ , 10 to 30 grams of NaCl and 0.25 part of HF.

Several 2 kg. master heats of composition 21.5 wt % Nb - 2.5 wt % Al - balance Ni were induction melted in an alumina crucible placed in an Inducto vacuum induction melting furnace powered by a 30 kW motor generator. The system was pumped down to 2.66 Pa, then backfilled with argon to provide an inert atmosphere at positive pressure. The niobium was placed at the bottom of the crucible. Power to the crucible coil was gradually increased until melting of the nickel and niobium was achieved. Aluminium was then poured into the melt. After five to ten minutes of homogenization, the power to the crucible coil was switched off. Then

the molten charge was poured into a steel split mold to produce eight round bars 1.25 cm. in diameter by 15 cm. long. Prior to casting, the mold walls were coated with a wash consisting of MgO powder in ethyl alcohol, and placed in a furnace at 200°C.

The as-cast bars were cleaned in the previously described pickling solution, then unidirectionally solidified in 95.5% pure aluminium oxide crucibles under a vacuum purged, positive argon pressure at a growth rate of 5 cm./hr. Solidification was achieved in a vertical zone melter equipped with an induction coil and a water cooled chuck which provided a high thermal gradient (26).

#### 2.2 Stress Rupture Testing

Stress rupture specimens (9.318 cm. diameter, 1.78 cm. gage length) shown in Figure 1 were machined from the unidirectionally solidified bars. When the longitudinal structure was deemed satisfactory upon metallographic examination, the bar was machined to yield two test specimens.

Constant load testing according to ASTM specification E 139-69 was conducted in air on Satec stands and on Dennision stands equipped with Marshall test furnaces. Temperature was controlled to within  $\pm 5^{\circ}$ C by means of two chromel-alumel thermocouples wired to the specimen. Elongation was measured on the fractured halves of the specimen. Testing on the

Satec stands allowed continuous recording of elongation by a system consisting of an extensometer clamped on the shoulders of the specimen, a linear voltage differential transformer (LVDT), a demodulator and a multi-point chart recorder. Because testing was performed in air and oxidation may result in excessive scaling of the extensometer, the previously described system was used for temperatures under 750°C. In several instances, because of the instability of the output signal, elongation was measured on the fractured halves.

#### 2.3 Metallography

Fracture surfaces were plated in a nickel plating bath (5 parts Enplate NI-415 A, 1 part Enplate NI-415 B, 8 parts water) at  $85^{\circ}$ C, then cut longitudinally by electrodischarge machining. Longitudinal sections were mounted in bakelite, polished through 600-grit wet polishing papers, 6 micron-1 micron diamond pastes, 0.3-0.6 micron alumina powders and electropolished in a solution consisting of 37 parts  $H_3P04$ , 56 parts glycerin and 7 parts water, with a voltage of 10 volts accross the electrodes.

#### CHAPTER III

#### PRESENTATION AND DISCUSSION OF RESULTS

#### 3.1 Microstructure and Crystallography

The composition of the alloy chosen for the present study was 21.5 wt % Nb, 2.5 wt % Al and balance Ni. Upon solidification, the liquid separates into two solid phases, the face-centered cubic  $\mathcal{J}$ -Ni solid solution and the orthorhombic  $\delta$ -Ni<sub>3</sub>Nb phase. Figure 2 shows a representative transverse section of the microstructure. Also shown is the presence of  $\mathcal{J}$ -Ni<sub>3</sub>Al precipitates obtained by precipitation from the Ni solid solution upon cooling from the solidus temperature. Lemkey and Thompson (37, 38) have determined that the crystallographic habit of the  $\mathcal{J}/\mathcal{J}$ -S alloys is:

> growth  $[011]_{\gamma}$   $[100]_{\delta}$ interface  $(1\overline{1}1)_{\delta}$   $(010)_{\delta}$

#### 3.2 Stress Rupture Behavior

The testing program is summarized in Table 1. Figure 3 is a stress-time to rupture plot which shows the effect of temperature and initial stress level on the stress rupture life and depicts the expected increase in stress rupture life with decreasing applied stress. A Larson-Miller parameter plot shown in figure 4 compares the results of the present study with those obtained by United Aircraft Research Laboratories for a 7/7.5 alloy (Ni-21.75 wt % Nb-2.55 wt % Al) grown at 38 cm./hr. (The empirical constant in the Larson-Miller Parameter in both cases was assumed to be 20 ). At this stage of our understanding of eutectic composite behavior, it is not certain that the Larson-Miller Parameter is the best comparison criterion; similarly, the material constant in the relation Parameter = T (C + log t) taken as 20, may not represent the best fit for this alloy. As seen in figure 4, the UARL alloy is superior to the alloy prepared for this investigation. It is not surprising to find such a difference since Thompson et al (25) demonstrated that reduction of the Lamellar spacing increased the creep resistance of the eutectic composite. In fact, if the Larson-Miller plot is normalized relative to tensile strength, the two sets of data are in better agreement (figure 5).

It was shown that the minimum creep rate (steady state creep rate) of lamellar eutectic composites (25. 38) follows the relation:

$$\dot{\mathcal{E}}_{ss} = k (6^{n}/T) \exp(-\frac{\Delta H}{RT})$$

where k = material constant

n = creep exponent

 $T = absolute temperature (^{O}K)$ 

 $\Delta H = activation energy (joules/mole)$ 

R = universal gas constant = 8.314 joules/mole-deg. Table 2 gives the minimum creep rate of three specimens tested at the same stress level and at different temperature. Based on the previously described relationship between minimum creep rate and temperature, the temperature compensated creep rate was computed from the data of Table 2 and plotted on a log-log scale with respect to (l/RT), see figure 6. Using a least mean squares method, a straight line was drawn through the data points. The slope of the line yields the activation energy for the creep process and is equal to 380 kjoules/mole.

A value of 349 kjoules/mole for the creep activation energy of  $\frac{3}{3} - \delta$  alloys was reported by Breinan (41). Although the value found during this investigation is in agreement with Breinan's result, it is not possible to ascertain conclusively as to which phase is in fact controlling the creep process in the temperature range 480 to 580°C owing to the paucity of creep activation energy data. For example, the generally accepted activation energy for steady-state creep of pure Ni is 276 kjoules/mole while the values associated with the creep of the intermetallic compounds Ni3Nb and Ni3Al are respectively 657 kjoules/mole and 685 kjoules/mole (47). Furthermore, N13 (Al, Mo) was found to have an activation energy of 446 kjoules/mole (48) whereas creep activation energy data for NigAl containing Nb or Ni<sub>3</sub>Nb containing Al have not been reported yet.

In the hot tensile properties investigation by Bertorello, et al (40), it was found that the 3' film at the eutectic grain boundaries played an important role

during tensile deformation at  $550^{\circ}$ C. It was shown that rupture associated with the 3' film allowed the deflection of transverse cracks to occur along the direction of the major stress axis, resulting in an increase in ductility. The hypothesis that this grain boundary delamination process is rate-controlling during steady-state creep cannot be ruled out; therefore it would be expected that the 3' grain boundary film would contribute to the creep of the eutectic alloy in the  $550^{\circ}$ C temperature range.

#### 3.3 Deformation and Fracture Mechanisms

# 3.3.1 Deformation and Fracture in the Low <u>Temperature Range (480°C to 580°C)</u>

It was reported elsewhere (22, 23, 24) that in the  $\delta_{-}\delta$  eutectic composite the intermetallic Ni<sub>3</sub>Nb deforms by twinning on  $\{2il\}$  type planes while the Ni phase slips during deformation of the composite. Figures 7 and 8 give evidence that the same deformation mechanism occurs during exposure up to 700 hours in the temperature range  $480^{\circ}$ C to  $580^{\circ}$ C. Note the slip markings in the  $\delta$  lamellae and the way the cracks propagate at the  $\delta$  twin- $\delta$  matrix interface. Failure of the eutectic occurred by the link up of these cracks and by eutectic grain boundary delamination, as depicted in figures 9 and 10.

Specimen L6-5 which failed after less than 100 hours exposure at 550°C shows grain boundary

delamination at the fracture surface (figure IO a) characteristic of Region I tensile deformation (40).

At intermediate exposure time at  $550^{\circ}$ C, grain boundary delamination occurs also away from the fracture surface (figure 10 b), the extent of which is less than that found on specimens deformed in Region II tensile behavior (40). Bertorello et al proposed that the ductility maximum of 27% obtained during tensile deformation at 550°C could be attributed to premature fractures associated with the 3' grain boundary film at  $550^{\circ}$ C which permitted the structure to deflect transverse cracks by grain boundary delamination. The ductility found on specimen J2-5-2 (about 7%) can be rationalized on the basis of this proposed deformation mechanism.

Figure 11 shows that  $\delta$  phase twin density is approximately equal in both Region I and Region II creep specimens, whereas cracking is more evident in Region II creep specimens.

Under long time exposure at  $550^{\circ}$ C, a transition from Region II deformation behavior to Region III deformation behavior occurred. Longitudinal sections showed two variants of deformation bands (figure 12), consisting of colinear  $\gamma$  and  $\delta$  twins (23, 24, 40). Note that the  $\delta$  twin density has decreased markedly. This cooperative twinning process is quite similar to that found in the  $\gamma$ - $\delta$  eutectic composite after long 16. time 600°C and short time 750°C creep (23, 24). Metallographic observation revealed that cooperative twinning occurred in the  $\sqrt[4]{3'-\delta}$  eutectic composite to a lesser extent than in the  $\sqrt[3]{-\delta}$  eutectic alloy. It is believed that, because of the  $\sqrt[3']$  precipitates, the  $\sqrt[3]$  phase cannot deform readily by twinning in conjunction with the twinning of the  $\delta$  phase.

3.3.2. Deformation and Fracture at 750°C

Under 750°C stress rupture conditions, longitudinal sections of specimens which exhibited a rupture life of less than 100 hours showed that composite deformation occurred by twinning in the  $\delta$  phase and by grain boundary delamination limited to a region near the fracture surface (figure 13). The  $\delta$  phase twins appeared to be thinner, resulting in a drastic decrease in the frequency of cracking at the  $\delta$  twin- $\delta$ matrix interface. Deformation under short time 750°C stress rupture conditions is thus typical of Region I tensile deformation.

The deformation mechanism observed from metallographic examination of long time 750°C stress rupture specimens was characterized by void nucleation at lamellae ends and eutectic grain boundaries. Figure 14 a shows that fracture occurred by the link up of these voids. It is believed that, because of the disruption of the crystallographic relationships at lamellae ends and eutectic grain boundaries, these

sites have a high energy configuration and would become preferred sites for void nucleation during composite deformation.

Metallographic analysis of two specimens creep tested at 760°C which failed after less than 100 hours of exposure revealed that cooperative twinning of the  $\delta$ and  $\gamma$  phases became operative. Near the fracture surface, severe blocky movements of the  $\Im$  and  $\Im$  lamellae produced cracking in the two phases (figures 15 b and 16). This extremely localized deformation mechanism resulted in failure of the composite when these cracks linked up, thereby decreasing the load bearing capability of the specimen. It was noted that away from the fracture surface, deformation of the composite was less severe, with fine bands of colinear  $\gamma$  and  $\delta$  twins being observed (figure 15 a). It is believed that this  $\sqrt[7]{-\delta}$ cooperative twinning deformation mechanism was not induced by a change in the testing temperature from 750°C to 760°C, but rather by some unidentified metallurgical variable.

## 3.3.3 Deformation and Fracture at 850°C and 950°C

Composite deformation under  $850^{\circ}$ C and  $950^{\circ}$ C bear some resemblance to the previously described long time  $750^{\circ}$ C deformation mechanism (figures 17 a and 19 a), with the difference that creep ductility was higher for all test conditions at  $850^{\circ}$ C and for long time  $950^{\circ}$ C creep specimens. The increase in creep ductility was related to an increase in the number of voids as clearly evidenced in figure 19 a.

Metallographic analysis of a short time  $950^{\circ}$ C creep specimen (specimen M5-5-1) showed that composite deformation was qualitatively similar to Region II tensile behavior: the  $\delta$  phase deformed by twinning and cracking (figure 18 b), whereas grain boundary delamination was believed to be the result of the coalescence of small voids produced at eutectic grain boundaries. This type of delamination appears to be different from that found in Region I hot tensile deformation behavior,  $550^{\circ}$ C and short time  $750^{\circ}$ C creep exposure.

Two additional observations were considered important to mention. Longitudinal section of specimen K4-5 crept under 850°C exhibited  $\delta$  Widmanstatten precipitates in the  $\delta/\gamma'$  phase (figure 17 b).  $\delta$ Widmanstatten precipation was previously reported (50) for the  $\chi-\delta$  aligned eutectic as a result of a 1220°C solution treatment and long time static exposure at 800°C.

Metallographic observation gave evidence that long time exposure under high temperature produced coarsening of the  $\delta'$  precipitates (figure 19 b). This observation suggests that, in order for  $\delta/\delta' - \delta$ eutectic alloys to retain their high strength at elevated temperatures, further alloying with elements which would stabilize the Ni<sub>3</sub>Al precipitates will be required. 19.

#### CHAPTER IV

#### CONCLUSIONS

Several conclusions were drawn from this study :

- 1) At 550°C, the stress rupture behavior of the eutectic alloy was characterized by three deformation and fracture mechanisms which were qualitatively similar to the three deformation and fracture mechanisms identified by Bertorello, et al in their investigation of the hot tensile properties. The time-dependent transition in creep and the temperature-dependent transition in tensile deformation suggest that the  $550^{\circ}$ C stress rupture behavior of the  $3/8'-\delta$  eutectic composite bears some similarity with the elevated temperature tensile and creep rupture behavior of the  $3-\delta$  eutectic composite.
- The creep activation energy of the eutectic composite tested between 480°C and 580°C was found to be 380 kjoules/mole.
- 111) Unlike the  $\delta \delta$  system, at  $750^{\circ}$ C the stress rupture behavior of the  $\delta/\delta' - \delta$  eutectic composite was characterized by a transition from Region I behavior to a void nucleation and void coalescence process. At  $760^{\circ}$ C, it was found that 20.

cooperative twinning of the  $\gamma$  phase and  $\delta$  phase became operative. It was hypothesized that this cooperative twinning process was caused by some unidentified metallurgical variable rather than by a change in the testing temperature.

iv) The 850°C stress rupture behavior and the long time 950°C stress rupture behavior were characterized by void nucleation and void coalescence, with the highest creep ductility being related to the highest amount of voids produced during composite deformation.

> A longitudinal section of a short time  $950^{\circ}$ C stress rupture specimen exhibited features characteristic of Region II tensile behavior.

v) The current study showed that the deformation and fracture mechanisms reported by Gangloff and Bertorello et al have been identified in this study. The analogy between a temperaturedependent transition in tensile deformation and fracture, and a time-dependent transition of the deformation and fracture processes in creep which was found for the  $\sqrt[7]{-\delta}$  aligned eutectic was not always found for the  $\sqrt[7]{-\delta}$ directionally solidified eutectic at high

temperatures.

#### TABLE 1

CREEP	RUPTURE	DATA
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Specimen#	Temperature	Stress		Time To Rupture	Elongation
	°C	MPa	(ks1)	hrs	(%)
M4-5-1	480	967	(138.5)	718.1	2.6**
15-5-1	4 <b>9</b> 5	911	(130.4)	55•5	5.1**
M1-5-1	530	982	(140.6)	52.9	4.5**
L6-5	547	942	(134.9)	87.7	2.4**
16-5-1	550	840	(120.2)	1055.7	0.7
J2-5-2	553	978	(140.0)	397.9	6.9
M6-5-1	578	967	(138.5)	53.1	4.6**
L4-5-1	550	911	(130.4)	(574)***	-
K2-5	653	868	(124.3)	183.6	1.7**
15-5-2	750	699	(100.0)	13.7	1.3
14-5-1	754	699	(100.0)	52.4	1.2
<b>J1-5-</b> 2	750	629	( 90.0)	22.2	0.9
M2-5	750	586	(83.9)	390.8	0.9**
<b>J3-5-</b> 2	748	562	( 80.4)	200.7	-
K3-5-1	750	543	( 77.8)	247.7	0.9**
J1-5-1	750	524	( 75.0)	261.0	1.2
I4-5-2	760	765	(109.5)	12.3	2.4
J2-5-1	760	638	(91.3)	54.2	0.1
<b>K4-</b> 5	850	350	( 50.1)	105.1	7.0
<b>J7-</b> 5	850	455	(65.2)	29.3	6.0
<b>J3-5-1</b>	930	242	( 34.6)	32.9	0.7
M5-5-1	950	177	(25.3)	12.0	2.0
L5-5-2	950	154	(22.0)	120.2	-
<b>I7-5-2</b>	950	132	( 18.9)	272.6	13.6

\* Third number identified the position of the specimen with respect to the ingot: "1" for the tail, "2" for the head.

\*\* Elongation measured by extensometer.

\*\*\* Test stopped at time reported.

# TABLE 2

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# MINIMUM CREEP RATE DATA

Specimen	Stress		Temperature	Minimum Creep Rate	
	MPa	(Ksi)	- °C	$(x \ 10^{\circ} \ hr^{-1})$	
M4-5-1	967	(138.5)	480	0.498	
M1-5-1	982	(140.6)	530	54.765	
M6-5-1	967	(138.5)	578	458.00	

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Figure 1: Creep Rupture Specimen



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Figure 2: Representative microstructure of a transverse section of a  $1/\gamma'-\delta$  eutectic composite grown at 5 cm/hr. Electropolished. Magnification 1600X.











Figure 6: Temperature compensated minimum creep rates as a function of (1/RT).



Figure 7: Scanning Electron Micrograph of a  $530^{\circ}$ C stress rupture fracture (Specimen M1-5-1) revealing slip markings in a  $\delta$  phase lamella and cracking in a  $\delta$  phase lamella . Magnification 6080X.





Figure 9: Longitudinal fracture profile of a 495°C stress rupture fracture (Specimen I5-5-1). Electropolished. Magnification 100X.



(a)



(b)

Figure 10:

Longitudinal fracture profiles of two 550°C stress rupture specimens. Electropolished.

- (a) Specimen L6-5. Magnification 100X.
- (b) Specimen J2-5-2. Note the higher amount of delamination. Magnification 100X.







- (a) Specimen I4-5-1. Note eutectic grain boundary delamina-tion. Magnification 100X.
- Specimen J1-5-2. Note the fine distribution of  $\delta$  twins in  $\delta$  lamellae. Magnification 1000X. (q)

(q) (a)

Longitudinal section of a long time 750°C stress rupture specimen (Specimen J3-5-2) illustrating that fracture was caused by the link-up of voids near the fracture surface. Modified Marbles etchant. Magnification 80X. Figure 14:



Longitudinal section of a  $760^{\circ}$ C stress rupture specimen showing X-S cooperative twinning (Specimen I4-5-2). Electropolished. Figure 15:

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- Note the fine deformation bands away from the fracture Magnification 500X. surface. (B)
- Note the cracking occurring near the fracture surface. Magnification 1000X. (q)



Figure 16:

: Longitudinal photomicrograph of a 760°C stress rupture specimen (Specimen J2-5-1) depicting severe blocky movement of J and δ lamellae near the fracture surface, producing transgranular cracks. Electropolished. Magnification 1000X.





Figure 18: Longitudinal section of a short time 950°C stress rupture specimen (Specimen M5-5-1).

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- Note cracking in large  $\delta$  lamellae-Modified Marbles etchant. Magnification 80X. (a)
  - (b) Electropolished. Magnification 250X.



- Longitudinal section of a long time 950°C stress rupture specimen (Specimen 17-5-2). Figure 19:
- Note the distribution of large voids near the fracture surface (Compare with figures 14a, 17a and 18a). Modified Marbles etchant. Magnification 80X. (a)
  - Note the coarsening of the v' precipitates. Electropolished. Magnification 1000X. (P)

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