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Strengthening in a DS Casting Ni_3Al Base Alloy IC6

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Abstract. A unidirectionally solidified casting γ' base alloy IC6, with the chemical composition (at.%) 16.3–17.6% Al, 8.0–8.3% Mo, 0.16–0.78% B, Ni balance has been developed for advanced gas turbine blades and vanes. The experimental results show that this alloy has high yield strengths from room temperature to 1100°C, excellent creep resistance at temperatures up to 1100°C. The microstructural observations and analysis indicate that the superior mechanical properties of this alloy may be attributed to solid solution hardening by the large molybdenum addition, second phase strengthening by γ' phase and other minor phases that precipitate in various temperature ranges, the formation of a γ raft structure during creep, and to the existence of high density misfit dislocation networks at γ'/γ interface areas due to a high value of γ'/γ misfit.

Keywords: Ni_3Al, DS casting, strengthening

Abbreviations:

BSE	back scattered electron
DS	unidirectionally solidified
EDS	energy dispersive spectroscopy

1. Introduction

At present, most of the gas turbine blades and vanes of advanced aero-engines are made of nickel base superalloys. With the development of aero-engines for high performance aircraft, the operating temperatures of these parts are constantly increasing, which limits the use of nickel superalloys. In addition, the high density and need to use expensive alloying elements, such as Hf, Ta and Re has caused the search for new materials, some of which are based on intermetallic compounds. As a matrix for high temperature structure alloy, Ni_3Al has some inherent advantages [1–3], such as high melting point, low density, high resistance to oxidation, low coefficient of diffusion, high resistance to elevated temperature creep, and a yield strength that increases with increasing temperature up to 800°C. Many two phase γ'-Ni_3Al base superalloys have been developed during the last 20 years [3–9]. They are either high strength wrought alloys used in the intermediate temperature range of 650 to 900°C or casting superalloys used at 1050 to 1100°C. A high performance casting γ'-base superalloy containing 8.0–8.3 at.% Mo, 16.3–17.6 at.% Al, 0.16–0.78 at.% B, and the balance Ni, named IC6, has been recently developed for the turbine blades and vanes of advanced aero-engines in this institute. In the present paper, strengthening mechanism of alloy IC6 are studied.

2. Experimental techniques

The material used in the present investigation was unidirectionally solidified cast Ni_3Al-base alloy with a composition (at.%) of 16.33Al-8.24Mo-0.26B–75.17Ni. The master alloy

was prepared by a vacuum induction furnace. The columnar grain rods for tensile and creep tests were produced by a rapid solidification method in a commercial DS vacuum induction furnace. The as cast rods were then homogenized in a vacuum furnace at 1260°C for 10 h to produce optimum properties. The room temperature and high temperature tensile tests were carried out on an Instron universal testing machine at a strain rate of 3.3×10^{-4} s^{-1} with the testing temperature controlled within ±3°C. The stress rupture tests were carried out by using constant load creep machines in air at 760 to 1100°C, with the testing temperatures controlled within ±3°C.

The microstructural analysis of various specimens was conducted by optical, scanning, and transmission electron microscopy. The volume fractions of various phases were determined by quantitative image analysis using a Leitz Image Analyzer. The chemical compositions of phases were determined by X-ray energy dispersive spectroscopy (EDS) technique using the polished surface of the specimens in a JXA-840 scanning microanalyzer.

The lattice parameters of γ' and γ phases at room temperature were determined by X-ray diffraction in a IBM-APD10 X-ray diffractometer using CuKa radiation at a voltage of 35 kv and a current of 35 mA. A step scanning method was used, and the scanning rate was 0.6 degrees/min with 0.02 degrees/step.

3. Results and discussion

3.1. Mechanical properties

Tensile tests were carried out at room temperature and in the temperature range of 700 to 1100°C. The results are shown in Table 1. For comparison, the observed values of the tensile yield strength and those of single crystal Ni_3Al and two advanced nickel base single crystal superalloys are shown in Fig. 1. The results in Table 1 and Fig. 1 show that the alloy used in this study has a higher tensile strength and a higher ductility. The tensile yield strength above 700°C is higher than all commercial DS nickel base superalloys and single crystal alloys. Ductility at 760°C, where a minimum in ductility occurs in most γ'/γ two-phase superalloys, is greater than 4%, which is acceptable for many engineering applications. Figure 1 also shows that the yield strengths of the alloys increase with temperature up to about 760 to 800°C and then decrease with increasing temperature, as has been observed in other γ'-base alloys.

Stress rupture tests were carried out at 760 to 1100°C at various stress levels, and the results are shown in Table 2. For comparison, the stress rupture lives of some advanced

Table 1. Tensile properties of alloy IC6 with the composition of 16.33Al-8.24Mo-0.26B-Ni balance at.% after 1260°C/10 hours followed by oil quenching.

Testing temp. (°C)	R.T.	700	760	870	1000	1050	1100
σ_{UTS} (MPa)	1115	1040	1170	1075	705	585	465
	1225	1090	1165	1040	720		500
$\sigma_{0.2}$ (MPa)	805	980	1105	975	565	520	385
	780	995	1110	950	640		395
Elongation (%)	14.2	8.5	6.3	11.0	28.1	25.0	44.0
	15.7	8.7	4.2	10.3		28.0	32.4

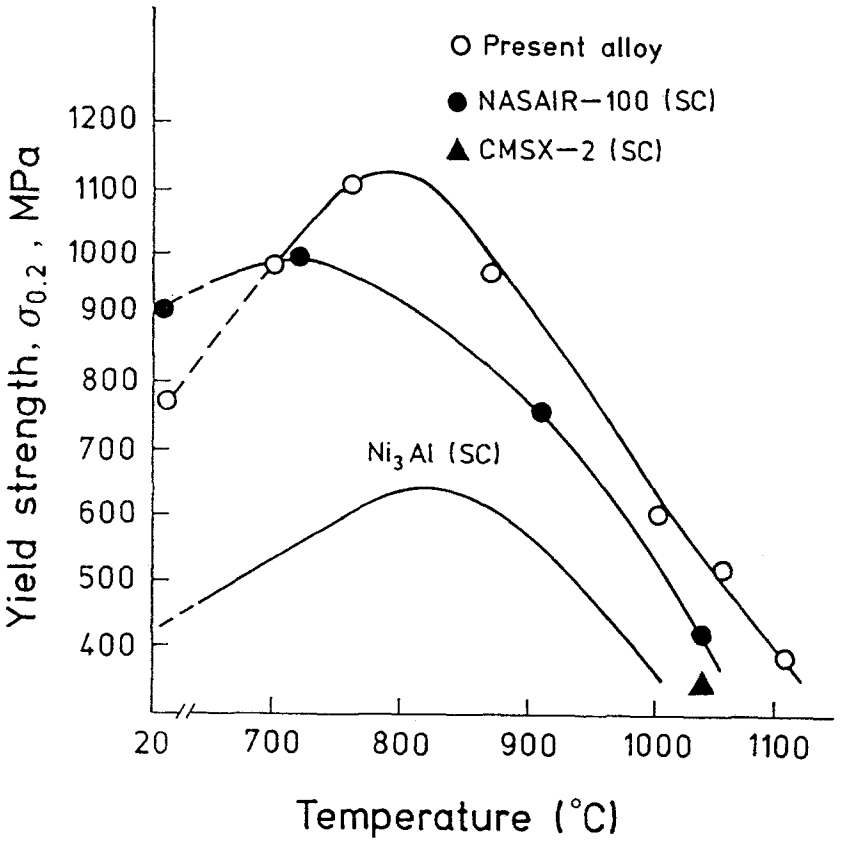

Figure 1. Variation in yield strength of the alloy used in the study, Ni_3Al single crystal, Ni base single crystal alloy CMSX-2, and NASAIR100 with temperature.

casting DS nickel and Ni_3Al base superalloys are also listed. DS NX188 is an experimental γ'-base superalloy with a chemical composition (wt.%) of Ni-18Mo-8Al-0.05C, which was developed in the early 1970s for gas turbine vanes operating at 1100°C. PWA1422 is a recently developed nickel base superalloy (Ni-0.1C-10Co-12W-5Al-2Ti-1Nb-2Hf-0.02B-0.05Zr) widely used as turbine blade material for advanced aero-engines, and DZ-3 is a high performance DS nickel base superalloy (Ni-0.1C-11Cr-5Co-5W-4.2Mo-5.6Al-2.7Ti) developed in the 1980s at the Beijing Institute of Aeronautical Materials for use as gas turbine blade material. Table 2 shows that the creep resistance of the present Ni_3Al base superalloy at an intermediate temperature of 760°C and at higher temperatures of 1040 and 1100°C is greater than these advanced experimental and commercial DS cast nickel and Ni_3Al base superalloys. The creep resistance of the alloy at 1040 to 1100°C compares favorably with the advanced nickel base singe crystal alloys CMSX-2 (Ni-8Cr-4.6Co-7.9W-0.1Hf) and NASAIR100 (Ni-9Cr-10.5W-1.0 Mo-5.8Al-1.2Ti-3.3Ta). However, the density of the present alloy (7.90 g/cm^3) is about 10% lower than that of CMSX-2 and NASAIR100. Therefore, the density compensated strength of this alloy is higher than these advanced single crystal nickel base alloys, which suggests that the present alloy has good

Table 2. Comparison of stress-rupture lives of the alloy with some advanced DS superalloys.

Testing temp. (°C)	Stress level (MPa)	Stress rupture lives (hours)			
		Present alloy γ' base	DSNX188 γ' base	PWA1422 γ base	DZ-3 γ base
760	804	197, >190	130	100	
950	235	>120			
	255			100	100
980	180	161 ~ 172			
	206			100	100
1040	140	107 ~ 169			
	137			100	100
1100	78.5	>500	30, 39		100
	88.3	>200		42, 44	40, 42
	100	100 ~ 300			

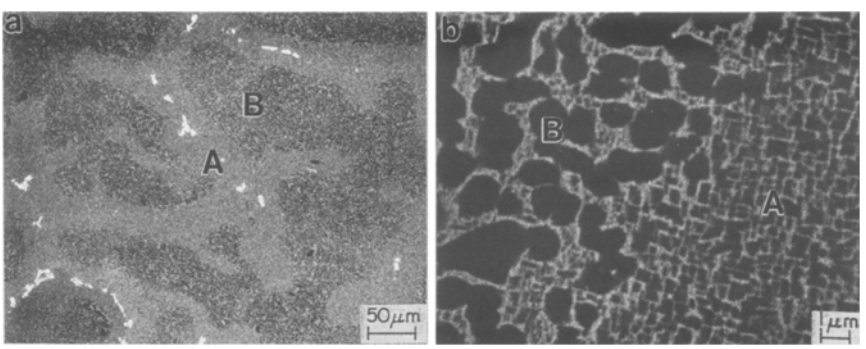

Figure 2. Back scattered electron images of the alloy homogenized at 1260°C for 10 hrs. and oil quenched: (a) polished surface, showing areas A and B, and boride (white particle); (b) higher magnification image of (a).

potential for application in the manufacture of gas turbine components for use at 1000 to 1100°C.

3.2. *Microstructure of the alloy*

Typical microstructure in a specimen homogenized at 1260°C for 10 h followed by oil quenching are shown in Fig. 2, which are the back scattered electron (BSE) images of the alloy obtained by SEM microscopy. The results of EDS analysis indicate that three major phases γ', γ, and boride are present in the homogenization heat treated condition. The white particles in area A in Fig. 2 are borides, the dark phase is γ'-Ni_3 (Al, Mo) with a chemical composition of 74 to 75 at.% Ni, 21 to 22 at.% Al, 3.9 to 4.3 at.% Mo and the grey phase is γ Ni solid solution. Area A is the interdendritic area consisting of fine (0.1 to 0.3 μm) cubic shaped γ' phase with a skeletal network of γ phase around it and white particles of boride. Area B in Fig. 2 is the dendrite arm consisting of γ' phase (1 to 2 μm) that did not dissolve by the homogenization treatment and a $(\gamma' + \gamma)$ two phase area similar

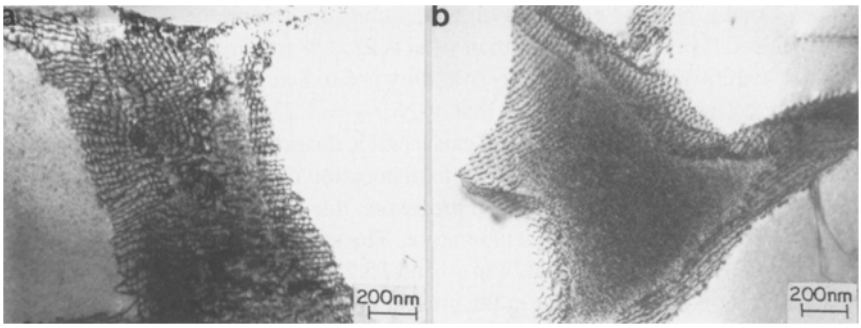

Figure 3. TEM micrographs showing misfit dislocation networks in specimen: (a) homogenized at 1260°C for 10 hrs. and oil quenched, $\bar{B} = [001]$; (b) after creep deformation at 1100°C/95 MPa, 190 hrs., $\bar{B} = [111]$.

to area A. The volume fraction of γ' phase in the present alloy was 75 to 80% as determined by quantitative image analysis.

The lattice parameters of γ' and γ phases, as determined by X-ray diffraction, were found to be 3.5859 and 3.6289 Å, respectively. Therefore, the lattice misfit between the γ' and γ phase is about 1.185%, which is higher than the reported values of lattice misfit in other nickel and Ni_3Al base superalloys. This high value of lattice misfit has been confirmed by the existence of high density misfit dislocations in the area of γ'/γ interfaces, as shown in Fig. 3. Figure 3a was taken from a specimen homogenized at 1260°C for 10 h, followed by oil quenching; Fig. 3b is the microstructure of a creep deformed specimen showing misfit dislocation at the γ'/γ interface. It has been suggested that the generation of these interface dislocations is an irreversible process and that the system stabilizes itself by their creation.

The interface dislocations were found to be primarily edge dislocations. The Burgers vector of these interface dislocations was determined to be primarily $a/2\langle 110\rangle$ type and a very few were $\langle 100\rangle$ type.

3.3. Strengthening mechanism in Alloy IC6

The results of this study suggest that Alloy IC6 exhibits high tensile strengths up to 1100°C and good creep resistance at both the intermediate and high temperature ranges. These properties, like those of any other complex alloy, are the result of a complex interplay of various strengthening mechanisms. In the present alloy, these mechanisms are likely to be (1) solid-solution strengthening by Mo in both γ' and γ phases, (2) second phase strengthening of γ' by 20% volume fraction of γ and other minor phases, (3) the formation of raft structure during high temperature creep, and (4) the presence of misfit dislocations networks at the γ'/γ interface, which can provide an effective barrier to dislocation motion. It is not possible to quantify the exact contribution by each of these mechanisms to the overall strength of the alloy, therefore, their origin and contributions will be discussed only qualitatively.

3.3.1. Solid solution hardening by molybdenum addition. Mo has been widely used as a strengthening element in many commercial nickel base superalloys, because it can strengthen both the γ matrix and the γ' phases. Compared with other refractory elements

such as W, Co, Ta, Nb, and Hf, it has higher solubilities in both the γ and γ' phases. For example, the solubility of molybdenum in γ-Ni is 21 at.% from room temperature to 800°C and 36 at.% at 1260°C. In γ'-Ni_3Al, its solubility is 6 to 8 at.% [10, 11]. Because Mo has a larger atomic radius ($r_{Mo} = 1.3625$ Å) than γ-Ni($r_{Ni} = 1.2325$ Å) but a smaller radius than Al ($r_{Al} = 1.4315$ Å), addition of Mo will cause lattice distortion of both the γ' and γ phases. This distortion can increase the resistance to dislocation motion and will also increase the activation energy for diffusion and creep processes, thus making the microstructure of the alloy relatively more stable at high temperature. The solubilities of Mo in the present alloy were found to be about 5.4 to 5.5 at.% in γ' and 15.2 to 15.9 at.% in the γ phase. Because the volume fraction of the γ' phase in the present alloy is about 80%, it is assumed that the strength of the γ' phase may be the main factor in determining the strength of this alloy. It has been reported [12] that both the 0.2% flow stresses of Ni_3Al at peak temperature and at-196°C increase with an increase in Mo content. The strengthening effect of Mo on the γ' phase in the present Ni-Al-Mo alloy system has been empirically analyzed, and the results illustrate that the room temperature hardness of the γ' phase increases linearly with Mo content. The experimental results in the present study showed that the solution hardening of the alloy is mainly due to the addition of Mo.

3.3.2. Strengthening by second phases. To study the phases present in this alloy at various temperatures, a nickel aluminum quasi-two-phase diagram with a Mo content of 8.2 at.% Mo was constructed. This phase diagram, which is based on a previous study of the Ni-Al-Mo phase equilibria in the temperature range of 600 to 1390°C [13, 14, 15] is illustrated in Fig. 4. According to this diagram, the second phases appearing in the present alloy at different temperatures are likely to be γ-Ni solid solution, α-Mo, δ-NiMo and γ''-Ni_3Mo phases. The α-Mo phase was observed in specimens annealed at 1200 to 1240°C, and its composition (at.%) was determined to be 94Mo-6Ni. The δ-NiMo phase was observed in specimens aged at 900 to 1100°C and creep deformed at 1100°C. The δ phase, the white particles within the grains shown in Fig. 5, forms according to the reaction $\gamma' - \gamma + \delta$-NiMo. A recent work by author showed that δ phase in the present alloy is different from δ phase in Ni-Mo-bi system and has been identified to have a tetragonal crystal structure named Y-NiMo phase with a space group of Cmcm. Although the formation of the Y phase leads to a decrease in the γ phase, which is considered to be an important strengthening phase in this alloy, the dispersive distribution of γ phase particles, as shown in Fig. 5, may also act as a strengthening phase, which will partly compensate for the effect of the decrease in γ phase due to the formation of Y phase. The experimental results show that the tensile yield strength of the alloy decreases only by about 10% when specimens are aged for 200 h at 900 to 1100°C [16]. The very small size, BCT ordered γ''-Ni_3Mo phase and tetragonal structured Ni_2Mo precipitates have been found in as-cast specimen and 700°C aged specimens, as shown in Fig. 6. Therefore, it may be considered that these second phases form in different temperature ranges and will have a corresponding strengthening effect at the temperature of their formation. Therefore, this alloy exhibits excellent mechanical properties over a wide temperature range from room temperature up to 1200°C.

A small amount (<2 Vol.%) of borides is also present after the homogenization heat treatment, as shown in Fig. 2. The presence of the borides improves the grain boundary ductility and tensile strength of the alloy. However, it was observed that any increase in boron content beyond 0.16 at.% has a deleterious effect on creep properties of this alloy [17] because of its brittle tetragonal crystal structure and large size (2 to 3 μm) (Fig. 2). Therefore,

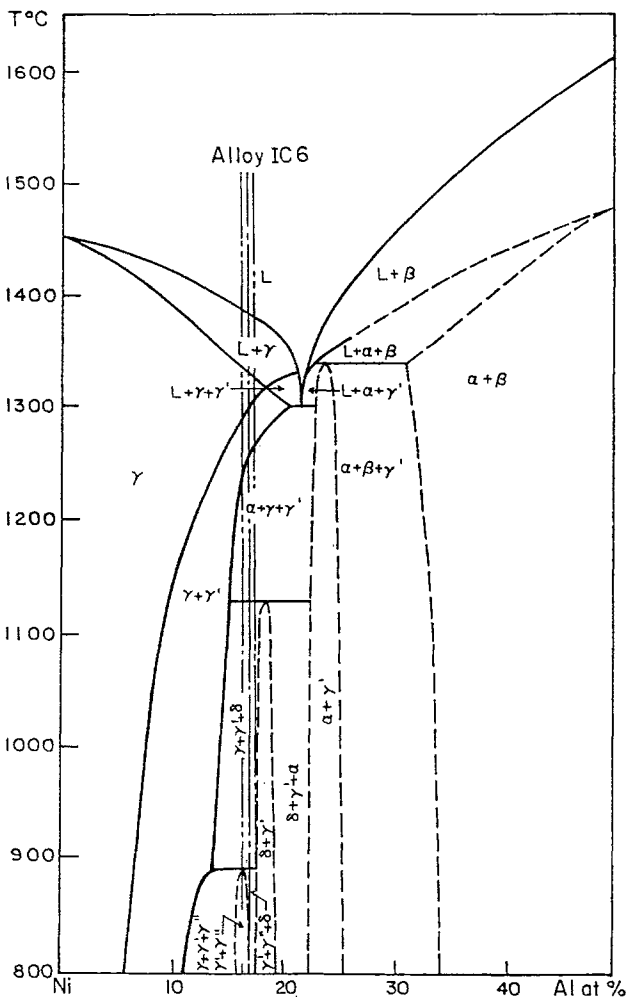

Figure 4. Ni-Al quasi-two phase diagram with Mo content of 8.2 at.% (δ phase in figure was identified to be Y-Ni-Mo phase).

the boron content in this alloy must be controlled to within 0.09 to 0.16 at.% where the amount of boride is less than 2 Vol.%.

The γ phase can cause a significant strengthening of the γ' matrix, as has been reported by Wen et al. [18]. They proposed a model to calculate the critical resolves shear stress, $\Delta\tau$, for an antiphase boundary (APB) dislocation pair to move through the matrix. According to this model, the extra work necessary to pull out dislocation pairs from the precipitate particle is dependent on the difference in the APB energy between the matrix and the precipitate and on the size and volume fraction of the precipitate. The microstructures in Figs. 2 and 3 show that the size of the γ phase is about 200 to 250 Å, which is comparable to the width of the APB dislocation pairs observed in the tensile and creep deformed specimens of the present alloy. This fine distribution of γ phase with a volume fraction of 0.20 can have a significant influence on the overall strength of the material.

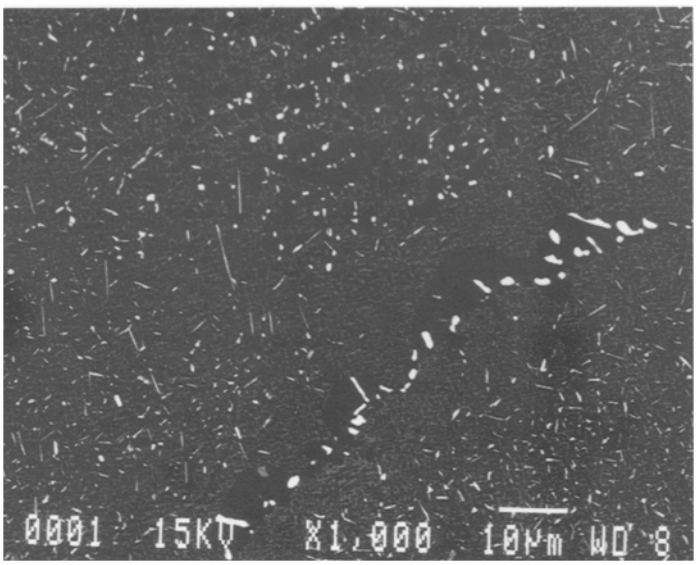

Figure 5. Y-Ni-Mo precipitates in alloy IC6 aged at 1000°C for 100 hrs.

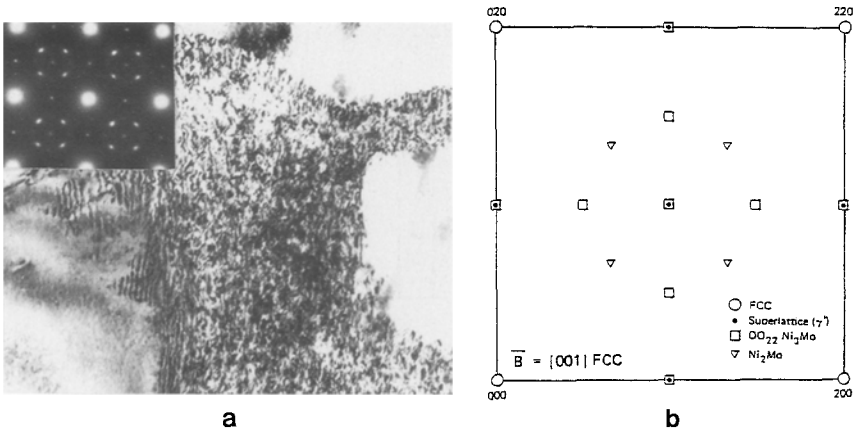

Figure 6. γ''-Ni_3Mo, Ni_2Mo precipitates in as-cast alloy: (a) bright field electron micrograph (taken with $\bar{B} = [001]$), (b) schematic diagram of (b) showing the existence of γ'' and Ni_2Mo.

3.3.3. Strengthening by γ raft structure during creep. The microstructure of this alloy was observed to change significantly during high temperature creep. Figure 7 is the back scattered electron images after creep deformation at 1100°C and 100 MPa for 30, 90, which shows that the skeletally distributed γ phase that is present after the homogenization heat treatment changed to a raft morphology, which is normal to the applied stress in the interdendrite area, and changed to isolated γ islands in the dendrite arm area. The γ rafts were found to form during the first stage of creep and were stable during the secondary creep stage. From the BSE images, the size of the γ rafts was measured to be about 0.2 to 0.3 μm in thickness and 3 to 5 μm in length. It is believed that, as a barrier to dislocation

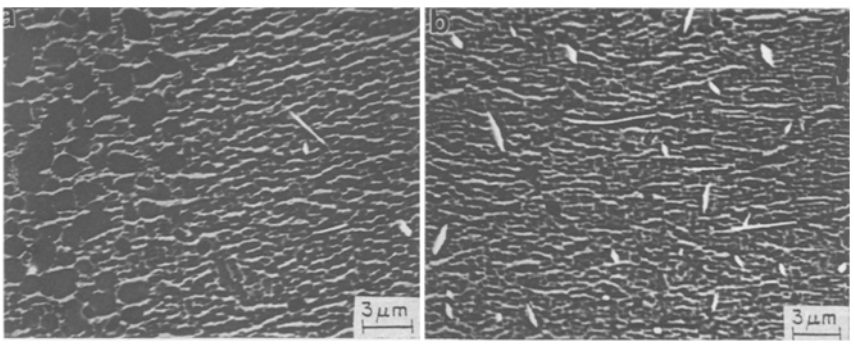

Figure 7. BSE microstructure after creep deformation under 1100°C/100 MPa for (a) 30 hrs., (b) 90 hrs., showing γ raft structure and Y-phase.

motion, the raft structure is more effective than the separated γ particles dispersed in the γ' matrix, because the raft structure may more effectively prohibit the moving dislocations from climbing over the second-phase particles. Consequently, the dislocations move mainly by gliding through from γ to γ' or γ' to γ phase.

3.3.4. Strengthening by misfit dislocations. The high yield strengths and creep resistance of this alloy also may be attributed to the high density of misfit dislocation networks at the γ'/γ interface areas due to the high value of the γ'/γ lattice misfit, δ, as shown in Fig. 3. It is obvious that the high density of the misfit dislocation networks can prohibit or retard the movement of dislocations from γ' to γ or γ to γ' and hence strengthen the alloy. These regularly arranged dislocations may be considered as predislocation pileups at the γ'/γ interfaces and can be effective barriers to dislocation motion. It was observed that, even in highly deformed specimens from both tensile and creep tests, the density of moving dislocations in the interface area was very low compared to that observed in the γ' or phase, and numerous dislocation pileups were observed at the γ'/γ interface [15].

4. Conclusions

The high yield strengths from room temperature up to 1100°C and excellent creep resistance at both intermediate and high temperatures of the present γ'-base superalloy indicate that this alloy is a potential material for turbine blades and vanes of advanced aero-engines. Its superior mechanical properties can be attributed to (1) solid solution hardening by Mo, (2) second phase hardening by γ, (3) strengthening by the formation of γ raft structure during high temperature deformation, and (4) strengthening by the present misfit dislocations at the γ'/γ interfaces.

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