CREEP DEFORMATION OF A 6th GENERATION Ni-BASE SINGLE CRYSTAL SUPERALLOY AT 800 °C and 735 MPa

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Abstract

The latest Ru-bearing 6th generation Ni-base single crystal superalloy TMS-238 developed at National Institute for Materials Science (NIMS) in Japan is a promising candidate for applications as gas turbine blades. Fundamental understanding of the creep deformation processes is necessary for alloy design and performance. In this study, the creep tests of MX-4/PWA 1497 (a 4th generation Ni-base single crystal) and TMS-238 were conducted at 800 °C and 735 MPa along [001]. For creep strain of 0.1%, TMS-238 had a creep time more than sixty times longer than MX-4/PWA 1497. Microstructural observations showed that stacking faults (SFs) sheared both the γ matrix and γ' precipitates in MX-4/PWA 1497, however, SFs sheared only the γ matrix in TMS-238. After creep rupture, a high density of dislocations was observed in MX-4, whereas a number of planar defects were formed in TMS-238. The factors that affect the creep deformation are discussed. The results imply that the stacking fault energy (SFE) of γ matrix in TMS-238 is lower than that in MX-4. Higher additions of Re and Ru are responsible for the lower SFE in TMS-238. The creep deformation processes in the two superalloys are different, primary creep stage and tertiary creep stage are dominant in MX-4 and TMS-238, respectively.

Introduction

Additions of refractory elements, especially Re and Ru, are well known to improve the creep resistance of Ni-base single crystal (SC) superalloys [1]. Recently, a 6th generation Ni-base SC superalloy TMS-238 with high Re and Ru content has been developed at NIMS for applications as gas turbine blades [2]. The temperature capability of TMS-238 to achieve 1000 h of creep life at 137 MPa is approximately 1120 °C. Compared to MX-4/PWA 1497 (hereafter abbreviated as MX-4), a 4th generation SC superalloy, TMS-238 has comparable creep resistance at 900 °C and 1000 °C, but much better creep properties at 800 °C and 1100 °C.

The creep strength of SC superalloys at high temperatures is very important in view of the high gas temperature. However, the creep behavior at relatively low temperatures (750-850 °C) is an essential concern because the rear part of turbine blades such as platform is subjected to high stress level and relatively low temperature.

The mechanism of creep deformation in <001> oriented SC superalloys is sensitive to temperature and applied stress [3]. At high temperatures and low stresses, rafting process is a typical characteristic. At intermediate temperatures and stresses, dislocation activity is mainly restricted in the γ matrix. In the temperature range of 750-850 °C and high stresses, γ' shearing and activation of <112>{111} slip are commonly observed during primary creep, and the creep behavior is strongly dependent on

the crystallography orientation [4, 5]. Especially, $a<112>$ dislocation ribbon shearing both γ and γ' was reported in CMSX-4 [6-9] and DD6 [10].

For better understanding of the enhanced creep strength of TMS-238 at 800 °C, the creep deformation of MX-4 and TMS-238 at 800 °C and 735 MPa was investigated. In our previous paper [11], we reported creep deformation mechanism using alloy samples with a 0.1% creep strain, but now in the present paper, based on the previous work, the deformation throughout the creep until rupture and the whole feature of creep deformation are discussed.

Experimental

The Ni-base SC bars ($\phi 10 \times 130$ mm) of MX-4 and TMS-238 were cast using a directional solidification furnace at NIMS. For both alloys, the crystallography misorientation determined by the Laue back-reflection X-ray technique was within 6° of [001], as shown in Figure 1. The nominal chemical compositions of the two alloys are listed in Table I. Heat treatments were carried out as follows:

MX-4: 1320 °C/10 h/air cooling (AC) + 1121 °C/4 h/AC + 899 °C/4 h/AC

TMS-238: 1345 °C/40 h/AC + 1150 °C/2 h/AC + 900 °C/16 h/AC

The specimens of diameter 4 mm and gauge length 22 mm were machined from the heat treated SC bars. Constant load tensile creep testing was performed in air at 800 °C and 735 MPa. For each alloy, both interrupted creep testing at a strain of 0.1% and creep rupture testing were attained.

Before and after creep testing, the microstructures were examined using transmission electron microscopy (TEM). TEM discs were cut perpendicular to stress axis. Then, the discs of MX-4 were manually ground to 100 μ m and perforated by a twin-jet electropolisher at 20 V/20 mA and -20 °C. The electrolyte consisted of 13% perchloric acid, 80% ethanol and 7% glycerol. The TEM foils of TMS-238 were prepared using Gatan's 656 Dimple Grinder and 691 Precision Ion Polishing System (PIPS) at 5 kV and $\pm 4^\circ$. A Tecnai 20 microscope was operated at 200 kV. Thermo-Calc program using a commercial Ni-RUMN database was employed for calculation.

Results and Discussion

Creep Properties

Figure 2 shows the results of creep testing. The serrated curves are because of the accuracy of strain sensor, i.e. 0.01%. For MX-4 and TMS-238, the time to creep strain of 0.1% was 2.9 h and 195 h, and the creep rupture life was 22.2 h and 1150.4 h, respectively. Though MX-4 had a larger creep strain than TMS-238 after rupture, its rupture life was much shorter than TMS-238. In Figure

2(e) and (f), one can see that most of the strain in MX-4 accumulated at primary creep stage, whereas that occurred at tertiary creep stage for TMS-238. The primary creep stage in TMS-238 is very short. Generally, the creep rate of MX-4 is approximately two orders of magnitude higher than that of TMS-238. It indicates that TMS-238 has much stronger creep strength than MX-4 at 800 °C and 735 MPa.

Figure 1. The patterns of Laue back-reflection using X-ray technique. Red dashed line: incident X-ray; Green: Greninger chart; Red solid line: $\langle 110 \rangle$ axis. For creep rupture testing specimens, the crystallography misorientation of (a) MX-4 and (b) TMS-238 is 5° and 3.5° of $[001]$, respectively.

Initial Microstructures

The initial γ/γ' microstructures of two SC superalloys were investigated, as shown in Figure 3. The average γ' size in MX-4 and TMS-238 was 243 ± 59 nm and 228 ± 50 nm, respectively, while the average γ channel width was 23 ± 6 nm and 26 ± 9 nm, respectively [11]. In general, the two alloys have similar γ' size and γ channel width. The small difference is that MX-4 has a slightly larger γ' size and narrower γ channel width with respect to TMS-238, while TMS-238 has a relatively uniform distribution and regular shape of γ' precipitates. Based on the experimentally determined γ' size and γ channel width, the V_f in MX-4 and TMS-238 at room temperature (RT) was calculated to be 76% and 73%, respectively, assuming the precipitates in a regular cubic shape. Comparing the V_f at RT and 800 °C (see Table I), one finds that the experimental results are in good agreement with the theoretical prediction in view of the stable γ/γ' microstructures at 800 °C. The lattice misfit of MX-4 and TMS-238, predicted using the Alloy Design Program (ADP) developed by NIMS [12], has no significant difference, as seen in Table I.

Shape of Cross Section

After creep testing, the specimen shape of cross section cut perpendicular to stress axis was examined. As seen in Figure 4, the aspect ratio of MX-4 increased with increasing creep strain, i.e. ~ 1.0 for $\epsilon = 0.1\%$ and 1.22 for creep rupture. This indicates that the creep deformation occurred in MX-4 is heterogeneous. In contrast, TMS-238 had a nearly constant aspect ratio, i.e. 1.0, even after creep rupture. It means that the creep deformation in TMS-238 is more homogeneous.

Deformation Microstructures

Creep of MX-4. The typical deformation microstructures of MX-4 are illustrated in Figure 5. For the interrupted specimen, see Figure 5(a), stacking faults (SF) were confirmed to lie on one of $\{111\}$ planes and to shear both the γ matrix and γ' precipitates. The narrow anti-phase boundary (APB) bonded together with two SFs were characterized, as indicated by the white arrows in Figure 5(a). This morphology is in agreement with the $a\langle 112 \rangle$ dislocation ribbon in CMSX-4 crept at 750 °C and 750 MPa reported by Rae et al [6]. The suggested configuration of a dislocation ribbon is: $a/3[11\bar{2}] + \text{SESF} + a/6[11\bar{2}] + \text{APB} + a/6[11\bar{2}] + \text{SISF} + a/3[11\bar{2}]$. The leading partial dislocation $a/3[11\bar{2}]$ shears γ' precipitates and creates an SESF within γ , the trailing partial dislocation $a/6[11\bar{2}]$ remains at γ/γ' interface due to the high energy of APB formed by net shearing of an $a/2[11\bar{2}](a/3[11\bar{2}] + a/6[11\bar{2}])$ dislocation. The preceding of the second $a/6[11\bar{2}]$ partial dislocation on the same slip plane reduces the APB width to a narrow band and creates an SISF followed by the second $a/3[11\bar{2}]$ dislocation. In addition to SFs, few undissociated dislocations in the horizontal γ channels were occasionally observed, as seen in Figure 5(b). After creep rupture, see Figure 5(c), a high density of dislocations was observed.

Creep of TMS-238. Figure 6 shows the deformation microstructures in TMS-238. For interrupted specimen [11], as seen in Figure 6(a), viewing along $[001]$, many defects with

Table I Nominal Chemical Compositions (wt.%), Lattice Misfit (δ , %), and Volume Fraction (V_f) of γ' Precipitates in MX-4 and TMS-238 [11].

Alloy	Ni	Co	Cr	Mo	W	Al	Ta	Re	Ru	Hf	V_f^a	δ^a
MX-4	Bal.	16.5	2.0	2.0	6.0	5.6	8.3	6.0	3.0	0.15	0.74	-0.16
TMS-238	Bal.	6.5	4.6	1.1	4.0	5.9	7.6	6.4	5.0	0.1	0.75	-0.17

^aVolume fraction of γ' precipitates and lattice misfit are calculated using the ADP. The V_f and δ listed are the value of equilibrium state at 800 °C.

Figure 2. Creep curves of MX-4 (a) and (c) and TMS-238 (b) and (d) at 800 °C/735 MPa. Interrupted testing with a creep strain of 0.1% [11]: (a) and (b); Creep rupture: (c) and (d). (e) and (f) showing creep rate as a function of strain in MX-4 and TMS-238, respectively.

Figure 3. Initial microstructures of MX-4 (a) and TMS-238 (b) [11]. Beam direction (BD) is along [001].

Figure 4. Shape of the cross section cut perpendicular to stress axis. The aspect ratio of MX-4 increased with increasing creep strain, i.e. ~ 1.0 for $\epsilon = 0.1\%$ and 1.22 for creep rupture. In contrast, TMS-238 had a nearly constant aspect ratio, i.e. 1.0, even after creep rupture.

straight linear contrast were evidenced, and they were absent in the area including only γ' precipitates and no γ matrix. It implies that the defects are formed in the γ matrix. Selected area diffraction (SAD) pattern along [011], the inset in Figure 6(b), shows clear streaks in two directions and no extra reflections except those from the matrix and γ' , which is a characteristic of SFs. Note that the SAD is slightly away from exact zone axis [011] due to the tilt limitation of microscope. Using the small objective aperture to include part of the streaks in one direction, the dark

field image was attained, as demonstrated in Figure 6(c). SFs were revealed to present in both horizontal and vertical γ channels. In Figure 6(d), SFs lying on four variants of the $\{111\}$ planes are visible as BD is along [011]. Under this orientation, $(\bar{1}11)$ and $(11\bar{1})$, marked as 2 and 4, were edge-on, whereas (111) and $(1\bar{1}\bar{1})$, marked as 1 and 3, were inclined. The typical projection width of a SF on the inclined (111) or $(1\bar{1}\bar{1})$ was 20 nm. It was deduced that the SF width on (111) or $(1\bar{1}\bar{1})$ was 21 nm. Therefore, the thickness of γ layer that the SF sheared was around 17 nm, which is quite reasonable in view of the 26 ± 9 nm γ channel width in TMS-238. Compared to MX-4, the SFs in TMS-238 do not shear the γ' precipitates. After creep rupture, see Figure 6(e), large numbers of interconnected planar defects were present. Apart from these planar defects, some tangled dislocations were also observed.

TEM observations show that the creep mechanisms in MX-4 and TMS-238 are different. In MX-4, the shearing of γ' precipitates results in higher creep rate with respect to TMS-238 and occurs on one primary slip plane. As creep deformation proceeds, the aspect ratio of cross section increases due to heterogeneous deformation. The activation of $a\langle 112 \rangle \{111\}$ slip during primary creep has been commonly observed in many SC superalloys at relatively low temperatures ($T/T_m < 0.6$) and high stresses. For the interrupted specimen of TMS-238, see Figure 6(a) to (d), SFs were revealed to be confined in only the γ matrix and to lie on four variant of $\{111\}$ planes, which makes the creep deformation homogeneous. SFs shearing of only the γ matrix remarkably decreases the creep rate in TMS-238. For SC superalloys, this is an unusual creep mechanism at 800 °C/ 735 MPa. Similar deformation processes were reported in Ru-containing SC superalloys with high Co and Re contents during high temperature creep at 950 °C/ 290 MPa [1]. A high density of SFs in the γ matrix enhances the creep resistance. First, it is because of the

Figure 5. Micrographs showing the deformation microstructures of MX-4. Interrupted testing [11]: (a) and (b); creep rupture: (c). (a) SF shearing both the γ matrix and γ' precipitates. APB presented between two SFs, as indicated by the white arrows. BD is along [011]. (b) Few undissociated dislocations in the horizontal γ channels. (c) A high density of dislocations was observed after creep rupture.

interaction of SFs on different slip planes. In fcc structure materials, Shockley partial dislocations from different non-parallel slip planes may react to form a sessile stair rod dislocation at the lines of intersection, e.g. $a/6[\bar{1}21] + a/6[2\bar{1}\bar{1}] \rightarrow a/6[110]$. The configuration of two SFs associated with a stair rod dislocation is known as Lomer-Cottrell lock [13], which obstruct slip dislocations. Second, the interaction between SFs and dislocations may also contribute to the enhanced creep strength. Recently, it is demonstrated that a high density of stacking faults as a new strategy enables an extraordinary combination of ultrahigh strength and good ductility in an hcp Mg alloy [14]. Third, the Shockley partial dislocation is difficult to shear γ' precipitates due to the creation of high energy complex SF, i.e SF plus APB.

Stacking Fault Energy of The γ Matrix

The dislocation dissociation in the γ matrix is related to the γ/γ' microstructure, applied stress, lattice misfit stress and stacking fault energy (SFE) of γ matrix. Generally, the two alloys have similar γ/γ' microstructures, such as precipitates size, volume fraction of precipitates and γ channel width. The lattice misfit (see Table I) in MX-4 and TMS-238 is almost equal. It implies that the stress level in γ matrix is comparable. Consequently, it is believed that the γ matrix of TMS-238 has lower SFE than that of MX-4.

Based on the chemical compositions (at.%) of γ matrix predicted using the ADP at 800 °C (see Table II), the difference of Gibbs energy was calculated using Thermo-Calc. Table III shows the results [11]. For the γ matrix of MX-4 and TMS-238, the energy barrier of phase transformation from fcc to hcp is $0.21 \times 10^4 \text{ J mol}^{-1}$ and $0.17 \times 10^4 \text{ J mol}^{-1}$, respectively. The former is 23.5% higher than the latter. Actually, SF in an fcc structure results in a local lattice transformation from fcc to hcp. Thus, the lower energy barrier means the easier formation of SF. This accounts for lower SFE of TMS-238 with respect to MX-4.

Alloying elements significantly affect the SFE. Al is a main γ' forming element, and the content of Al in the γ matrix in two alloys is the same (see Table II). So the effect of Al on the SFE of γ matrix is not the main cause of lower SFE in TMS-238. Based on the calculation of Gibbs energy difference (see Table III), the energy barrier ($\times 10^4 \text{ J mol}^{-1}$) of phase transformation from fcc to hcp for Co, Mo and Cr is +0.03, -0.30 and -0.44, respectively. Whereas, the energy barrier for Re and Ru is -0.93 and -0.99, respectively. Mo, Cr, Re and Ru decrease the energy barrier, and Co slightly increases the energy barrier. It means that Re and Ru are more effective elements than Co, Mo and Cr for decreasing the SFE of γ matrix per unit content. From the viewpoint of crystal structure, Mo and Cr have stable bcc structure, and Co has stable fcc structure above approximately 450 °C [15]. In contrast, Re and Ru have stable hcp crystal structure. The crystal structure is closely related to the phase transformation from fcc to hcp. A stable hcp structure favors SF formation, therefore Re and Ru are thought to be mainly responsible for the lower SFE in TMS-238 though their content variation is smaller than Co and Cr. For accurate interpretation, the complicated interaction between alloying elements should be considered.

Creep Deformation Processes

Based on the present investigations, the creep deformation processes occurred in MX-4 and TMS-238 at 800 °C and 735 MPa

Figure 6 Deformation microstructures in TMS-238. Interrupted testing [11]: (a)-(d); creep rupture: (e). (a) SFs formed only in the γ matrix. BD is along $[001]$. Bright field image (b) and corresponding dark field image (c) showing the morphology of SFs viewed along $[101]$. The inset in (b) is a selected area diffraction pattern in which the streaks of SFs are evidenced in two directions. (c) Dark field image was taken using the small objective aperture to include part of the streaks in one direction. (d) SFs lying on four variant $\{111\}$ planes. BD is along $[101]$. $(\bar{1}11)$ and $(11\bar{1})$, marked as 2 and 4, were edge-on, whereas (111) and $(1\bar{1}\bar{1})$, marked as 1 and 3, were inclined. (e) A number of planar defects presented after creep rupture.

can be elucidated as follows:

In MX-4, only one of slip systems $\{111\} \langle 112 \rangle$ is active at early creep stage, and $a\langle 112 \rangle$ dislocation ribbons shear both the γ matrix and γ' precipitates. As creep deformation proceeds, more and more $a\langle 112 \rangle$ dislocation ribbons belonging to the same slip system are activated. Thus, the creep rate accelerates with increasing creep strain, as observed in Figure 2(e). Further increasing creep strain, the slip of dislocation ribbons becomes difficult, and the secondary slip system may be activated and impede the dislocation slip on the primary slip system. So the creep rate gradually decreases to the minimum, which roughly corresponds to the creep strain from 8% to 25% at later primary creep stage, as shown in Figure 2(e). For tertiary creep stage, the Orowan dislocation bowing in the γ channels and paired dislocations cutting through the γ' precipitates may take place due to the high enough stress level. As seen from Figure 5(c), a high density of tangled dislocations is present.

In TMS-238, the $a/2 \langle 110 \rangle$ dislocation dissociates into two Shockley partial dislocations in the γ matrix at early creep stage. The dislocation dissociation occurs on four $\{111\}$ atom planes and partial dislocations shear only the γ matrix. As creep deformation proceeds, the slip of partial dislocations becomes more difficult due to dislocation interaction on various $\{111\}$ planes and the attracting force from SFs, which reduce the creep rate, as observed in Figure 2(f). The γ matrix is difficult for further deformation due to numbers of SFs. Thus the stress level increases rapidly in the γ matrix. The accumulated high enough stress may activate Orowan dislocation bowing and paired dislocations cutting through γ' precipitates. These processes gradually accelerate the creep rate corresponding to tertiary creep stage, as seen in Figure 2(f). Note that the extent of paired dislocations cutting through γ' precipitates in TMS-238 is less significant than that in MX-4. In Figure 6(e), even at late creep stage many areas inside γ' precipitates are free of dislocation, which is different from that observed in Figure 5(c). It is probably because the creep rupture strain of TMS-238 is much smaller than that of MX-4.

In the present study, the crystallography misorientations of all MX-4 and TMS-238 specimens are within 6° of $[001]$. The small

orientation deviation of specimens cannot account for the largely enhanced creep strength of TMS-238. It has been reported that the Co addition may increase the orientation sensitivity of SC superalloys during tensile creep deformation at 800°C and 735 MPa [16]. The Co content in MX-4 and TMS-238 is 16.5% and 6.5%, respectively. MX-4 has higher Co content than TMS-238, which may be partly responsible for the large primary creep strain occurred in MX-4.

Summary

A 6th generation Ni-Base single crystal superalloy TMS-238 has been developed. TMS-238 has much stronger creep strength than MX-4 at 800°C and 735 MPa.

At primary creep stage, the $a\langle 112 \rangle$ dislocation ribbons in MX-4 sheared both the γ matrix and γ' precipitates, whereas SFs were formed in only the γ matrix of TMS-238 and did not shear the γ' precipitates.

For MX-4, the creep deformation is heterogeneous. The dislocation ribbons occurred mainly on one of the $\{111\}$ plane. For TMS-238, the creep deformation is homogeneous. Stacking faults were formed on four $\{111\}$ planes.

After creep rupture, a high density of dislocations was observed in MX-4. In contrast, numbers of interconnected planar defects presented in TMS-238 apart from some tangled dislocations.

The calculation of difference of Gibbs energy indicates that higher Re and Ru additions are mainly responsible for the lower SFE in TMS-238.

MX-4 and TMS-238 have different creep deformation processes. For MX-4, the $a\langle 112 \rangle$ dislocation ribbons are the main mechanism for primary creep stage. For TMS-238, Shockley partial dislocations shearing the γ matrix are the dominant mechanism. At tertiary creep stage, dislocation bowing in the γ channels and paired dislocations cutting through the γ' precipitates may take place in MX-4 and TMS-238. Due to much smaller creep rupture strain, the extent of paired dislocations cutting through the γ' precipitates in TMS-238 is less significant than that in MX-4.

Table II Chemical compositions (at.%) of γ matrix and γ' precipitates predicted using the ADP at 800°C .

	Ni	Co	Cr	Mo	W	Al	Ta	Re	Ru	Hf
γ in MX-4	41.03	32.32	6.62	2.72	2.28	3.56	0.61	6.80	4.07	0.01
γ' in MX-4	60.85	13.09	1.03	0.87	2.05	16.71	3.77	0.40	1.16	0.07
γ in TMS-238	47.43	14.42	16.75	1.49	1.66	3.56	0.54	7.40	6.75	0.01
γ' in TMS-238	68.79	4.51	1.89	0.47	1.28	17.28	3.37	0.44	1.93	0.05

Table III The Difference of Gibbs Energy at 800°C Calculated Using Thermo-Calc [11].

	Gibbs energy ($\times 10^4 \text{ J mol}^{-1}$)		Energy barrier of phase transformation from fcc to hcp ($\times 10^4 \text{ J mol}^{-1}$)
	hcp	fcc	
γ matrix of MX-4	-6.26	-6.47	0.21
γ matrix of TMS-238	-6.20	-6.37	0.17
Co	-4.91	-4.94	0.03
Re	-5.48	-4.55	-0.93
Ru	-4.63	-3.64	-0.99
Cr	-3.64	-3.34	-0.30
Mo	-3.45	-3.01	-0.44
W	-3.54	-3.02	-0.52
Ta	-4.56	-4.23	-0.33

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