TENSION/COMPRESSION ASYMMETRY IN YIELD AND CREEP STRENGTHS OF NI-BASED SUPERALLOYS

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Abstract

The tension/compression asymmetries of yield and creep strengths of three kinds of single-crystal superalloys—PWA1480, CMSX-4, and TMS-75—and a DS superalloy, Mar-M247LC, were investigated at intermediate and high temperatures. In PWA1480, tensile yield strength was higher than the compressive strength from 20°C to 750 °C. From the TEM observation, it was found that the asymmetry of yield strengths is primarily due to the microtwin formation associated with a superlattice extrinsic stacking fault (SESF). In CMSX-4 and TMS-75, tensile/compressive yield strengths were comparable at every temperature, and shearing of γ′ precipitates by a/2<110> dislocations pairs was the dominant deformation mechanism in both tensile and compressive tests at 750 °C. The creep response of these materials were quite different than their yielding response. CMSX-4 and TMS-75 showed distinctive creep tension/compression asymmetry. These two alloys showed large creep strain caused by {111}<112> slip at 750 °C under tensile stress, and mechanical twins at 750 °C and 900 °C under compressive stresses. Tension/compression asymmetry of CMSX-4 and TMS-75 was larger at 900 °C than at 750 °C because <112> viscous slip was not observed under tensile stress at 900 °C. The asymmetric nature of PWA1480 was small at both 750 °C and 900 °C because the dominant deformation mode in both tension and compression is a combination of both a/2<110>-dislocation’s bowing on the {111} plane in the matrix and climbing along the γ/γ′ interfaces.

Introduction

Ni-based single-crystal superalloys exhibit tension/compression asymmetry in yield strength and creep behavior at low temperatures [1, 2]. The flow stress behavior of Ni-based superalloys has been explained by the thermally activated cross-slip model suggested for the single γ′ phase [3, 4, 5]. Takeuchi and Kuramoto predicted the crystallographic-orientation dependence of the flow stress of Ni3Ga single crystals assuming that dislocations on the {111} slip planes become locally pinned by thermally activated cross-slip onto {100} planes [6]. Tension/compression asymmetry in the yield strength of Ni-based superalloys was explained in terms of the “core width effect” by Lall, Chin and Pope [7]. They modified the “cross-slip effect” model proposed by Takeuchi and Kuramoto, and assumed that the width of the core of the a/2[101] superpartial dislocation must be changed by the effect of the components of the stress tensor when the superpartial dislocation in the γ′ phase undergoes cross-slips from {111} to {100}. Since these a/2[101] superpartial dislocations dissociate into Shockley partials on the {111} plane, any component of the stress tensor that constrains superpartial dislocations promotes cross-slip, and any component that extends them impedes cross-slip. According to the “core width effect” [7], the tensile flow stress is greater than the compressive flow stress for orientations near [001].

Under thermo-mechanical fatigue (TMF), the number of cycles to failure of a single-crystal superalloy was found to decrease drastically with an increase of the compressive strain hold time [8]. The compressive creep strength of single-crystal superalloys is lower than the tensile creep strength on [001] loading [2]; therefore, high-tensile residual stress is produced by the larger stress relaxation during the compressive hold. This would decrease the TMF strength of the single-crystal superalloy. These phenomena are due to mechanical twinning associated with the operation of a {111}<112> viscous slip. Hence, in order to improve TMF life under condition of a compressive strain hold, it is important to clarify the mechanism of tension/compression asymmetry in the creep behavior in order to inhibit the operation of <112> viscous slip. Little attention has been given to the tension/compression asymmetry at intermediate temperatures. In this paper, emphasis is placed on creep and yield strengths in the temperature range 750-900°C to explain the phenomena of asymmetry behavior in single-crystal superalloys.

Experimental Procedure

The investigation was carried out for a first generation SX superalloy, a second generation SX superalloy, as well as a third generation SX superalloy; i.e., PWA1480, CMSX-4 and TMS-75. A directionally solidified (DS) superalloy, Mar-M247LC, was also tested to study the influence of grain boundaries on the intermediate temperature properties. The nominal chemical compositions of the alloys tested in this study are listed in Table I. The alloys were solution heat-treated and subsequently aged using the respective conditions shown in Table II. After analyzing of the crystallographic orientation by the back-reflection Laue method, specimens were cut from a single as-aged bar using a spark cutter. The

<table>
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<th>Table I. Chemical composition of alloys (mass %)</th>
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<tr>
<td>Co</td>
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<tr>
<td>PWA1480</td>
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<tr>
<td>CMSX-4</td>
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<td>TMS-75</td>
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<td>MAR-M247LC</td>
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Table II. Heat treatment condition of alloys

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<th>Aging treatment</th>
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<tr>
<td>PWA1480</td>
<td>1282°C/1 hr + 1287°C/2 hr + 1294°C/1 hr/GFC</td>
<td>1080°C/4 hr/AC + 870°C/32 hr/AC</td>
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<tr>
<td>CMSX-4</td>
<td>1277°C/2 hr + 1288°C/2 hr + 1296°C/3 hr + 1304°C/3 hr + 1313°C/2 hr + 1316°C/2 hr + 1318°C/2 hr + 1321°C/2 hr/GFC</td>
<td>1140°C/4 hr/GFC + 870°C/20 hr/GFC</td>
</tr>
<tr>
<td>TMS-75</td>
<td>1240°C/1 hr + 1280°C/2 hr + 1300°C/2 hr + 1320°C/8 hr/GFC</td>
<td>1150°C/4 hr/GFC + 870°C/20 hr/GFC</td>
</tr>
<tr>
<td>MAR-M247LC</td>
<td>1230°C/2 hr/GFC</td>
<td>980°C/5 hr/GFC + 870°C/20 hr/GFC</td>
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The dimensions of the gage length were 19.6×2.8×3.0 (mm) in tensile specimens and 10.0×3.0×3.0 (mm) in compressive specimens. Tensile tests were performed at 20 °C, 400 °C and 750 °C under strain rates of 4.25×10⁻⁶ s⁻¹ (tension) and 8.33×10⁻⁴ s⁻¹ (compression). Tensile and compressive creep tests were performed at 750 °C under 750 MPa and 900 °C under 392 MPa in the <001> orientation. The specimens for TEM observation were cut from interrupted specimens along the desired crystallographic plane and electro-polished using a twin jet with a solution of 15% perchloric acid in methanol at 25 V and 5 °C. Specimens were observed with a JEOL 2000FX microscope at 200 kV and a JEM-3010 microscope at 300 kV. The Burgers vectors and the natures of stacking faults were determined using the contrast visibility of dislocations and stacking fault fringes [9].

Fig. 1. Yield strengths of tensile and compressive tests under initial strain rates of 4.25×10⁻⁶ s⁻¹ (tension) and 8.33×10⁻⁴ s⁻¹ (compression): (a) PWA1480, (b) CMSX-4 and (c) TMS-75. The points are the results of single tests.

Fig. 2. TEM images of the deformed structure in CMSX4 specimen after yielding: (a) tension, ε=1.5% and (b) compression, ε=4.3%.
Fig. 3. TEM microstructure of the yielded PWA1480 specimens: (a) bright-field image at 1.9% tensile strain, the foil normal [111], Beam Direction (BD)~112; and (b) g-3g weak beam image at 2.7% compressive strain, the foil normal [111], BD~112.

Fig. 4. TEM microstructure of PWA1480 of compressive specimens after yielding at 2.7%. (a) Dislocation structure, Beam Direction (BD)~112. (b) Schematic diagram illustrating that the a/3[211] dislocation cuts the γ′ phase and leaves a superlattice stacking fault in the γ′ phase. (c) Microstructure showing micro twin, BD~110 and (d) lattice image of circled area shown in (c).

Results

Yield strength

The yield stress as defined by the 0.2% proof stress is shown in Fig. 1. Tensile/compressive yield strengths were comparable in CMSX-4 and TMS-75 at every temperature, whereas PWA1480 showed tension/compression asymmetry of the yield strengths. The tensile yield strength of PWA1480 was higher than the compressive strength from 20 °C to 750 °C. The tensile yield strength increased from 400 °C to 750 °C. In CMSX-4, the shearing γ′ by
a/2<110> dislocation pairs was the same (Fig. 2(a), (b)) for both tension and compression. Tensile and compressive yield microstructures in PWA1480 are shown in Fig. 3. The tensile microstructure showed matrix dislocations and many stacking faults in γ′ (Fig. 3(a)). The percentages of the numbers of superlattice intrinsic stacking faults (SISF) and superlattice extrinsic stacking faults (SESF) in the tensile and compressive specimens of PWA1480 at 750 °C are shown in Table III. The relative percent occurs in reverse for tension and compression: The applied stresses favored SISF formation in tension and SESF formation in compression. The compressive microstructure revealed matrix dislocations, many stacking faults and linear dislocations in the γ′ phase, which are shown by arrows (Fig. 3(b)). Using the invisibility criterion [9], the analysis of the Burgers vectors and line directions [10,11] revealed that linear dislocations in the γ′ phase were of the a/2<110> type on {111} planes. Cutting of the γ′ phase by the superlattice dislocation and the stacking fault, colored black, was also observed (Fig. 4(a)). Through the analysis of the Burgers vectors and line directions [9,10], it was determined that the Burgers vector of the dislocation in the γ′ phase was a/3<112>. The γ′ phase was cut by a a/3<112> dislocation, as illustrated in Fig. 4(b). The compressive TEM microstructure of the (110) foil is shown in Fig. 4(c). This image is an edge-on view of the fault using the <110> zone axis. From the enlarged High Resolution Transmission Electron Microscopy (HRTEM) image (Fig. 4(d)) of the linear structure circled in Fig. 4(c), it was found to be a mechanical twin having 9-12 atomic planes in its width. This twin was called a “microtwin” [11]. These results indicate that a stacking fault is indeed associated with twin formation in the γ′ phase.

Creep strength

Fig. 5 shows the tensile and compressive creep curves at 750 °C. In CMSX-4 and TMS-75, the compressive creep curve showed inferior strength compared to the tensile creep curve. In contrast, the creep curves of PWA1480 were comparable. The microstructures of tensile and compressive interrupted creep tests in TMS-75 and PWA1480 are presented in Fig. 6. In TMS-75 the <112> ribbons which were composed of extended stacking faults and a/3<112> dislocations were observed both in tension and compression (Fig. 6(a), (b)). The dislocations in tension and compression, which are indicated by arrowheads, were type a/3<112> on {111}. The tangled matrix dislocations were observed both in tension and compression in PWA1480 (Fig. 6 (c), (d)). The tangled dislocations in tensile specimens were of the type a/2<110> on {111}. The dominant mechanism in creep deformation was the a/2<110> dislocation motion, which occurred mainly in the γ matrix channels. In TMS-75 and CMSX4, mechanical twin lamellae in the compressive creep microstructure were confirmed by {011} plane observation, as shown in Fig. 7. They were not observed in the bright-field image of PWA1480 (Fig. 7(c)).

Tensile and compressive creep curves at 900 °C are presented in Fig. 8. Anisotropy between tension and compression was observed in CMSX-4 and TMS-75. As shown in Fig. 9, in all three alloys, it was observed that dislocations were concentrated into

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<th>SISF (%)</th>
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<tr>
<td>Tension</td>
<td>75.0</td>
<td>25.0</td>
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<tr>
<td>Compression</td>
<td>37.5</td>
<td>62.5</td>
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Table III. The proportion of SISF and SESF observed in tensile and compressive PWA1480 specimens at 750°C in (%).
the γ phase, and very few dislocations occurred in the γ′ phase. It is likely that the tensile creep deformation occurs by a combination of <110> slip in the matrix and dislocation's climbing along the γ/γ′ interface. The creep curves under tensile stress showed the transition from secondary creep to tertiary creep (Fig. 8). Fig. 10 shows the TEM microstructures of compressive creep specimens. The PWA1480 microstructure showed a concentration of dislocations in the γ phase and a few stacking faults in γ′. On the other hand, in CMSX-4 and TMS-75 specimens, distinctive stacking fault pairs were observed. The Burgers vectors of partial dislocations that formed stacking fault pairs were determined. The Burgers vectors of dislocation A were identified as a/3<112>, and those of dislocation B were identified as a/6<112>. Primary creep strains in CMSX-4 and TMS-75 (Fig. 8) would be brought about by the motion of the stacking fault pairs. These creep curves of these alloys showed the transition from primary creep to secondary creep (Fig. 8(c), (d)). Moreover, deformation twins were observed in the compressive specimens in these two alloys (Fig. 10(d)).
Deformation mechanisms of creep

The tension/compression asymmetry was distinctive in the creep strengths of CMSX-4 and TMS-75. In CMSX-4 and TMS-75, the compressive creep curve showed inferior strength compared to the tensile creep curve. Under compressive stresses, as shown in Fig. 7, the TEM micrograph of the [001] specimen showed microtwins extending over both the γ and γ′ phases. The results suggest that the asymmetry is also caused by mechanical twinning under compressive stress. Tension/compression asymmetry of creep strength in the [001] orientation is attributed to the directional-formation characteristic of the mechanical twins; i.e., twin formation occurs only under [001] compressive stress conditions, and its formation stress is lower than the critical resolved shear stresses for ⟨110⟩⟨111⟩ and ⟨112⟩⟨111⟩ slip systems.

Twinning is not a dominant deformation mechanism in metals which possess many possible slip systems; it generally occurs when the slip systems are restricted or when something increases the critical resolved shear stress so that the twinning stress is less than the slip for slip. The yield stress of γ′ precipitates increases with increasing temperature up to a peak temperature (650 °C – 800 °C). Since the critical stress for twinning is lower than that for the slip near the peak temperature, novel mechanical twins can be expected to occur in the Ni-based superalloys. The twinning shear is always directional in the sense that the shear in one direction is not equivalent to the shear in the opposite direction. Twinning in FCC metals occurs on ⟨111⟩ planes in the ⟨112⟩ direction. In [001]-compressive deformations, the resolved shear stress on the ⟨111⟩ plane acts in the twinning direction, and the formation of the mechanical twin results in weakness.

However, if long-range ordering produces a superlattice structure, the ordinary twinning mode will become a pseudo mode which results in incorrect ordering in the sheared lattice. In the superalloys, an a/6 ⟨112⟩ Shockley partial is unable to pass through the γ′ precipitate easily because it creates a high-energy fault. If a/6⟨112⟩ Shockley dislocations shear the γ′ precipitates, the L1₂ ordered structure will be destroyed. Kolbe [12] suggested a microtwin-formation model for the superalloys. After an a/6 ⟨112⟩ dislocation glides on the ⟨111⟩ plane, a CSF (Complex Stacking Fault) is produced behind the dislocation. The second a/6⟨112⟩ dislocation cannot follow the first on the same ⟨111⟩ plane because it produces an energetically unfavorable structure. Therefore, it is assumed that it glides on a neighboring ⟨111⟩ glide plane.

The third dislocation follows on the next ⟨111⟩ plane. However, in the L1₂ ordered structure, the movement of dislocation produces an energetically unfavorable pseudotwin. A pair of double steps of diffusion reestablishing order in the pseudotwin, or "interchange shuffles", will follow the dislocation movement.

The final result of the process is the creation of thin twin lamellae that extend through both the γ and γ′ phases as shown in Fig. 7. The twin dislocation rotates around its pole on every successive ⟨111⟩ plane [13]. By such a mechanism, a twin can propagate very quickly with the interchange shuffles, and is not subjected to the need for a separate partial dislocation source on every ⟨111⟩ plane [14]. Therefore, the nucleation of the twin must be the rate-determining step rather than the propagation process. Massive microtwins (Fig. 7) were observed at the same time as SISF/SESF stacking fault pairs (Figs. 6, 10). The nucleation of the microtwin was then associated with the nature of superlattice stacking faults.

In contrast with CMSX-4 and TMS-75, in PWA1480 and DS Mar-M247LC, the extent of creep asymmetry was small. The
crept PWA1480 specimen in this study did not show the mechanical twin structure that was observed in CMSX-4 and TMS-75 (Fig. 7). The dominant deformation mode of PWA1480 both in tension and compression is a combination of a/2<110> dislocation bowing on the {111} plane in the matrix and climbing along the γ/γ′ interface (Fig. 6). In an Ni-based superalloy containing a high level of tantalum, PWA1480, the {111}<112> slip was prohibited and the critical stress of mechanical twin formation would be increased because of the strengthened γ′ phase. A high level of tantalum is expected to induce the following effects: the solid-solution strengthening of the γ′ phase resulting from lattice parameter changes [15, 16], the increase of APB energy on (111) [15] (The APBE of Ni₃Al is increased from 180 mJm⁻² to 240 mJm⁻² when substituting 1 at% Ta for 1at% Al) [17, 18], and the promotion of the Suzuki locking effect of the {111}<112> slip as a result of the segregation of tantalum into the SSF [19]. It is reasonable to expect that the dislocation motion in the γ′ phase would be restrained by the effects of tantalum. As a result, the dominant deformation mode of PWA1480 both in tension and compression should be a combination of both a/2<110> dislocation bowing on the {111} plane in the matrix and the climbing process along the γ/γ′ interface; that is to say, the creep rates would be controlled by the dislocation climb process.

Single-crystal Mar-M247LC shows a distinct {111}<112> slip [20]; however, in case of the DS alloy, tension/compression asymmetry associated with the {111}<112> slip was not observed. It seems reasonable to suppose that the grain boundaries inhibit {111}<112> slip system operation.

Effect of temperature on creep deformation mechanisms

The ratio of compressive to tensile primary-creep strain as a function of the test temperature is illustrated in Fig. 11. Tension/compression asymmetry was more pronounced at 900 °C than at 750 °C in CMSX-4 and TMS-75. In general, the dominant deformation mechanism of creep in the temperature range above 850 °C is viscous slipping of a/2<110> dislocations and the climbing process along the γ/γ′ interface, and the <110> slip does not show a clear primary creep strain [21, 22]. In the present study, tensile creep specimens showed no primary creep strain (Fig. 8) and the Burgers vector was determined as <110> by the contrast visibility of the dislocation (Fig. 9) at 900 °C. The resolved critical shear stress for the {111}<112> slip falls above the peak temperature of yield stress. As a result, at 900°C, tensile creep deformation occurred from a combination of an a/2<110> slip and climbing along the γ/γ′ interface. We can speculate on the cause of tension/compression asymmetry of creep at 900 °C based on our observation of tension/compression asymmetry at both 750°C and 900 °C in CMSX-4 and TMS-75. In general, as mentioned above, the dominant deformation mechanism in the temperature range above 850 °C is the viscous slip of a/2<110> dislocations and the climbing process along the γ/γ′ interface; the stacking fault mode of deformation plays a minor role at high temperatures. However, in this study, the γ and γ′ phases were sheared by <112> dislocation pairs, and microtwins were observed under compressive stress at 900 °C. The critical shear stress of twinning formation associated with <112> slipping appears to be lower than that for <110> slipping even at 900°C. One explanation for this may be that twinning nucleation is associated with the critical radius of the extrinsic and intrinsic stacking fault loop, as described below.

Knowles [23] described the correlation between the formation of deformation twins during creep and the nature of superlattice stacking faults in CMSX-4. Tension/compression asymmetry is brought about by the following mechanism associated with <112> slip. SISF (Superlattice Intrinsic Stacking Fault) tends to form under tensile stress and SESF (Superlattice Extrinsic Stacking Fault) under compressive stress. Superlattice stacking faults are formed by two Shockley partial dislocations. Under compressive stress, the leading partial is subjected to a high Schmid factor; therefore, the partial enters the γ′ phase. After it cuts the γ′ phase, the trailing partial enters the γ′ phase to form SESF. On the other hand, under tensile stress, the Schmid factor for the trailing partial is higher than that of the leading partial. As a result, it is possible for the trailing dislocation to cross the leading dislocation and

Fig. 9. Dislocation structures of interrupted tensile crept specimens at 900 °C.(a) PWA1480 (ε=0.5%), (b) CMSX-4 (ε=0.5%) and (c) TMS-75 (ε= 0.4%). BD-001.
enter the γ' precipitate where the nucleation of a third Shockley partial allows a super Shockley partial formation to trail an SISF, crossing between the leading and trailing partial forms an y; i.e SI A in [0 s an SESF twinning mechanism is smaller than that r an SISF mechanis d high temperatures, suggesting that twinn  through an SESF echanism at both temperatures. The critical size generally in-
creases at higher temperatures, which indicates that twinning becomes less likely to occur at higher temperatures. However, at 950 °C and 350 MPa, the critical size of a twin nucleus in compression reaches a minimum at n (number of atomic layers in a nucleus) =4, and is smaller than the critical size of an SESF (n=2). Therefore, a twin nucleus of four atomic layers is more likely to form than an SESF. It should be clear from the above consideration that, in this study, the compressive stress favored twin nuclea-
tion and SESF formation even at 900 °C.

To sum up, at 900 °C, the deformation mechanisms in tension and compression are completely different; i.e., the dominant deformation mode in tension occurs by an a/2<110> dislocation motion; however, the creep mechanism in compression was associated with <112> slip and twin formation. At 750 °C, however, tension and compression mechanisms were both associated with <112> slip. The tension/compression asymmetry of CMSX-4 and TMS-75 was larger at 900 °C than at 750 °C because the <112> viscous slip does not occur under tensile stress at 900 °C.

In PWA1480, at temperatures of 750 °C and 900 °C, the dominant deformation mode in both tension and compression are a combination of a/2<110> dislocation bowing on the {111} plane in the matrix and climbing along the γ'/γ interface; that is to say, the creep rates are controlled by the dislocation climbing process. This is thought to be the reason why there is no difference in the creep deformation mechanism for tensile and compressive stresses, and why the asymmetry was smaller in PWA1480 than in the other single-crystal superalloys.
Yield deformation mechanisms

For yield strength, tension/compression asymmetry has generally been explained in terms of the "core width effect", which was accounted for by the following reaction.

\[ \frac{a}{6}[211]+S.F.\rightarrow \frac{a}{6}[112] \rightarrow \frac{a}{2}[101] \]

Since constriction of the superpartial dislocation aids the cross-slip process, tension is stronger than compression in the [001] orientation in which the extended superpartial dislocation retreats cross-slip activity. In CMSX-4 and TMS-75, while the γ' shearing by a/2<110> dislocations was the dominant deformation mechanism in both tensile and compressive tests at 750 °C, tensile/compressive yield strengths were comparable at every temperature in both alloys. From these results, the "core width effect" would not be a primary factor of tension/compression asymmetry in yield strength. However, since the lattice image of a microtwin (Fig. 4(d)) and an a/3<112> dislocation on the {111} plane (Fig. 4(b)) were clearly observed in PWA1480, the asymmetry of tensile/compressive yield strengths is considered to be caused by mechanical twin formation. Therefore, the asymmetry of yield strengths at 750 °C would be primarily due to the microtwin.

Conclusions

The tension/compression asymmetries of yield and creep strengths of three-generations single-crystal superalloys and a DS superalloy have been investigated; the following results were obtained.

1. Tension/compression asymmetry of both yield and creep strengths in the [001] orientation are attributed to the directional-formation characteristic of a mechanical twin; i.e., twin formation will occur only under the [001] compressive stress condition, and the formation of mechanical twins results in weakness.

2. Massive microtwins were observed at the same time as SIFS/SESF stacking fault pairs. The nucleation of the microtwin is associated with the nature of superlattice stacking faults.

3. In PWA1480 and DS Mar-M247LC, the extent of creep asymmetry was small. A high level of tantalum will prohibit the formation of an SIFS/SESF stacking fault pair and increase the critical stress of mechanical twin formation. Grain boundaries also inhibit {111}<112> slip operation and the formation of mechanical twins.

4. The tension/compression asymmetry of the strengths was not observed when the dominant deformation mechanism was the motion of the a/2<110> dislocations.

5. The extent of creep asymmetry of CMSX-4 and TMS-75 was found to be larger at 900 °C than at 750 °C. The reason is that the <112> viscous slip does occur under compressive stress even at 900 °C; whereas, a/2<110> dislocation's motion is the dominant deformation mechanism under tensile stress at the temperature.

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References


