Plastic deformation behaviour and deformation substructure in Al-rich TiAl single crystals deformed at high temperatures

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Abstract

Plastic deformation behaviour in Ti–54.7 at.%Al and Ti–58.0 at.%Al single crystals was examined around and above the anomalous strengthening peak temperature ($T_s$) focusing on the effect of Al$_3$Ti$_3$ superstructure. The Al$_3$Ti$_3$ superstructure developed in the L1$_0$ matrix of Ti–58.0 at.%Al, and the size of the Al$_3$Ti$_3$ phase once increased during annealing at 800°C and then decreased with increasing temperature, while no significant evidence of the Al$_3$Ti$_3$ particles was obtained in Ti–54.7 at.%Al from TEM observation although diffuse scattering corresponding to the spots for the Al$_3$Ti$_3$ superstructure was observed. The transition of slip plane for $\frac{1}{2}(110)$ ordinary dislocations from $\{111\}$ to $\{110\}$ and/or $\langle 001 \rangle$ occurred at and above $T_s$ due to anisotropy of anti-phase boundary energies on $\{111\}$, $\{110\}$ and $\langle 001 \rangle$ in the Al$_3$Ti$_3$ superstructure. Anomalous strengthening is related to the development of this superstructure which may assist the cross-slip of some parts of $\frac{1}{2}(110)$ ordinary dislocations onto $\{110\}$ and/or $\langle 001 \rangle$ resulting in the formation of dragging points to the motion of the dislocations. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Titanium aluminate; High temperature deformation; Phase transformation; Slip system; Dislocation structure

1. Introduction

$\gamma$-TiAl with L1$_0$ structure has a wide solubility range from near stoichiometry in equilibrium with the $\alpha_2$ phase to the Al-rich side [1]. In the off-stoichiometric Al-rich TiAl containing about 58 at.%Al, the Al$_3$Ti$_3$-type superstructure developed [2] and its stability depended strongly on the Al concentration and annealing condition [3–5]. Recent residual electrical resistivity measurement suggests that ordering based on the Al$_3$Ti$_3$ superstructure occurs even in the L1$_0$ matrix with the low Al concentration of Ti–52.6 at.%Al [6]. Effects of the Al$_3$Ti$_3$ superstructure on the plastic deformation behaviour in Al-rich TiAl crystals have been reported [7–10]. Since the motion of $\frac{1}{2}(110)$ ordinary dislocations and the activation of $\frac{1}{2}(112)$ deformation twinning are suppressed by development of the superstructure, the dominant deformation mode in TiAl alloys changes from $\{111\}\frac{1}{2}(110)$ slip and $\{111\}\frac{1}{2}(112)$ twinning to $\{111\}<101>$ slip with increasing Al content [7,8].

In Al-rich TiAl alloys, anomalous strengthening is known to occur depending on the orientation [9,11–14]. Several mechanisms for the anomalous strengthening were proposed for each operative slip system [9,11–18]. One of them was focused on the effect of Al$_3$Ti$_3$-type ordering on the motion of $\frac{1}{2}(110)$ ordinary dislocations [9]. The anisotropy of APB energies on $\{111\}$, $\{110\}$ and $\langle 001 \rangle$ in the Al$_3$Ti$_3$ superstructure may assist the cross slip of some parts of $\frac{1}{2}(110)$ ordinary dislocations resulting in the formation of dragging points.

Few reports have focused on the plastic deformation behaviour above the anomalous peak temperature [12,13]. $\frac{1}{2}(110)$ ordinary dislocations were reported to glide not only on $\{111\}$ but also on $\langle 001 \rangle$ and $\{110\}$ planes at high temperatures [12]. In addition, $\frac{1}{2}(112)$ superlattice dislocations and deformation twinning were observed; these dislocations were found to be created by the interaction between $\frac{1}{2}(110)$ and $\langle 001 \rangle$ dislocations [13]. The critical resolved shear stress (CRSS) for each slip system was also examined [12,13]. However, the effect of the Al$_3$Ti$_3$ superstructure on the plastic deformation behaviour of Al-rich TiAl alloys is not yet clearly understood.

In this article, the effect of the Al$_3$Ti$_3$ superstructure on the plastic deformation behaviour and deformation substructure of Ti–54.7 at.%Al and Ti–58.0 at.%Al single
crystals was examined around and above the anomalous peak temperature.

2. Experimental procedure

Master ingots of Ti–54.7 at.% Al and Ti–58.0 at.% Al alloys were prepared by melting high purity raw materials in a plasma arc furnace. Single crystals were grown from the ingots by a floating zone method using the NEC SC-35HD single crystal growing apparatus in a high purity Ar gas flow. Thin foils for observation of the microstructure were cut parallel to (001) and were sealed in quartz capsules under high purity Ar gas. They were quenched into ice water after annealing in the temperature range between 800 and 1200°C for 3.6 ks. The microstructure was examined by a Hitachi H-800 transmission electron microscope (TEM) operated at 200 kV.

Oriented compression specimens with dimensions of approximately 2 × 2 × 5 mm³ were cut from the crystals by spark machining. The stress axes of A, B and C specimens were chosen to be [201], [210] and [100] in a [001]–[100]–[110] unit triangle, respectively. Miller indexes of the Al₂Ti₃ superstructure in this article are represented on the basis of the L₁₀ structure, although the volume of the unit cell and the length of a-axis are different between the Al₂Ti₃ superstructure and the L₁₀ structure. Schmid factors for the possible slip systems are given in Table 1. Compression tests were performed in a vacuum on an Instron type testing machine in the temperature range between 700 and 1300°C at a nominal strain rate of 1.7 × 10⁻⁴ s⁻¹. The strain rate sensitivity parameter, m, was calculated by an abrupt change in the strain rate between 1.7 × 10⁻⁴ and 1.7 × 10⁻³ s⁻¹ at each selected temperature. Slip markings were analysed by an optical microscope equipped with Nomarski interference contrast. Deformation substructure was observed by TEM.

3. Results

3.1. Microstructure and phase stability of Al₂Ti₃ superstructure

Fig. 1 shows [001] electron diffraction patterns in Ti–54.7 at.% Al and Ti–58.0 at.% Al single crystals annealed at 800 and 1000°C for 3.6 ks. Diffuse scattering and/or extra spots are seen in addition to the strong spots from the matrix phase with the L₁₀ structure. Diffuse scattering with very weak intensity indicated by arrows is observed at the position corresponding to the Al₂Ti₃ superstructure in Ti–54.7 at.% Al crystal annealed at 800°C as shown in Fig. 1(a). The intensity became weaker with increasing annealing temperature but very faint scattering was still observed even after annealing at 1200°C. No evidence of a strict long-range ordered phase with the Al₂Ti₃ superstructure was obtained from a dark field image using the diffuse scattering in Ti–54.7 at.% Al annealed between 800 and 1000°C.

Extra spots were clearly observed at the positions between the spots based on the L₁₀ structure in Ti–58.0 at.% Al crystal. Since all the extra spots in Fig. 1(b) appear at a quarter of the distance between the L₁₀ spots, they are appropriate for the Al₂Ti₃-type superstructure. Similar spots were previously confirmed in some Al-rich TiAl alloys containing more than 56 at.% Al [2–5,7,9,12]. The intensity of the extra spots decreased with increasing temperature and {1100} spots disappeared prior to {3140} spots at 1000°C as shown in Fig. 1(d).

Fig. 2 shows the dark field images of Ti–58.0 at.% Al alloy annealed at various temperatures using a (3140) spot corresponding to the Al₂Ti₃ superstructure. The Al₂Ti₃ particles with the long-range order are homogeneously distributed in the L₁₀ matrix. The average diameter of the particles was about 10 nm in the as-grown Ti–58.0 at.% Al single
crystal [7]. The size once increased during annealing at 800°C as shown in Fig. 2(a) and then decreased with increasing temperature. Bright and dark regions in the figure correspond to the Al₃Ti₁ and L1₀ matrix, respectively. The Al₃Ti₁ precipitates as large particles. Since the periodicity of Al layers in the Al₃Ti₁ superstructure and L1₀ matrix are common, the interface exhibits obscure coherency [5]. The interface between Al₃Ti₁ and matrix was confirmed to be coherent by high resolution electron microscope observation. The volume fraction rapidly decreased with increasing temperature, particularly above 900°C where the extra spots were weakened. However, the particles distributed homogeneously as fine precipitates still maintained a long-period superstructure even after annealing at 1200°C.

3.2. Deformation behaviour and yield stress

Fig. 3 shows typical stress–strain curves in the oriented specimens of Ti–58.0 at.%Al single crystals deformed at various temperatures. In an early stage of deformation, the high-temperature yield phenomenon is observed for A and C orientations in the temperature range between 800 and 1000°C. The yield phenomenon was reported in many intermetallics including Ni₃(Al,Ti) single crystals [19,20]. The yield drop in Ti–58.0 at.%Al single crystal disappears at 1200°C and the steady-state deformation continues after yielding, while the yield drop in Ni₃(Al,Ti) occurred more remarkably at high temperatures. An oscillated flow occurs in the stress–strain curve for B orientation at around 1000°C. The oscillated flow corresponds to inhomogeneous deformation under constricted stress condition between the specimen and the jig in compression. The peculiar shape change in the specimen was observed. A similar behaviour was observed in Ti–54.7 at.%Al crystals but the temperature for the oscillated flow was lower.

Fig. 4 shows temperature dependence of the yield stress (0.2% offset stress) in Ti–54.7 at.%Al and Ti–58.0 at.%Al single crystals for A, B and C orientations. Anomalous strengthening is seen for three orientations in the two crystals. The anomalous strengthening behaviour depends

![Graph of stress-strain curves](image-url)

Fig. 3. Typical stress–strain curves in Ti–58.0 at.%Al single crystals with various orientations (A: [201]-axis; B: [210]-axis; C: [100]-axis) deformed at 800, 1000 and 1200°C.
strongly on orientation and aluminium content of the crystals. The anomalous strengthening is more accelerated and the peak shifts to the lower temperature in the order of C, A and B orientations for the two crystals. Increase in aluminium content lowers the peak temperature, in particular, for C orientation. Above the anomalous strengthening peak temperature, the yield stress monotonously decreases. The yield stress of Ti–58.0 at.%Al is higher than that of Ti–54.7 at.%Al between 800 and 1000°C, while no significant difference in the stress is observed between the two crystals at and above 1100°C.

3.3. Operative slip systems

Fig. 5 shows slip traces on the specimen surface of Ti–58.0 at.%Al single crystals deformed to 2% plastic strain in the temperature range between 700 and 900°C. At 700°C where anomalous strengthening occurred, only the traces on {111} are seen for three orientations. At 800°C, the slip traces on {111} are still dominant and no significant change in slip system occurs for B orientation, while for C orientation wavy slip traces on {110} become dominant. For A orientation, dominant slip occurs on {111} but slight traces deviated from {111} to (001) can be seen at 800°C. At and above 900°C, the traces on {111} were no longer observed for three orientations. Very wavy slip traces appeared on (110) for B orientation. For C orientation, the traces deviated from {110} to non-crystallographic planes. For A orientation very wavy slip occurred along non-crystallographic planes near {110} planes. The transition in the slip plane and slip morphology may correspond to an abrupt change in yield stress at temperatures above and below the peak in Fig. 4.

According to the contrast analysis of dislocations in Ti–58.0 at.%Al crystal, \( \frac{1}{2} \{110 \} \) ordinary dislocations were dominantly activated for A and C orientations above 800°C, while for B orientation {110} superlattice and \( \frac{1}{2} \{110 \} \) ordinary dislocations were operative at and above 800°C, respectively. Therefore, this monotonous decrease in yield stress above the peak temperature corresponds to the activation of the \( \{110\} \frac{1}{2} \{110 \} \) or \( \{001\} \frac{1}{2} \{110 \} \) slip system. These slip systems were reported by the groups of Inui et al. [12] and Jiao et al. [13].

The observed slip systems for three orientations in Ti–54.7 at.%Al and Ti–58.0 at.%Al single crystals are summarised in Table 2. The transition temperature of the systems shifts to lower temperature with higher Al concentration. For C orientation, for example, change in the operative slip plane from {111} to {110} occurs in Ti–54.7 at.%Al single crystals between 800 and 900°C, while it can be achieved at 800°C in Ti–58.0 at.%Al single crystals.

3.4. CRSS for the slip systems by \( \frac{1}{2} \{110 \} \) ordinary dislocations

Fig. 6 shows variations in CRSS for dominant operative slip in Ti–58.0 at.%Al single crystals with deformation temperature. The anomalous strengthening was closely related to \( \{111\} \frac{1}{2} \{110 \} \) ordinary slip and \( \{111\} \{110 \} \) superlattice slip, and their slips were dominant below each peak temperature [9,11,12]. At 700°C, the CRSS for \{111\} \{101\} slip at C orientation is higher than that at B orientation and Schmid law is violated as reported previously [12]. Above the peak temperature, \( \{110\} \frac{1}{2} \{110 \} \) slip becomes dominant and the CRSS for this slip rapidly decreases with increasing temperature. The slip transition induces anomalous peak in yield stress. The CRSS for B and C orientations in the high temperature range is higher than that for A.
orientation, and Schmid law is violated for \{110\} \frac{1}{2} \{110\} slip as reported by Jiao et al. [13] although the reason for this is not yet clear.

3.5. Deformation substructure

Fig. 7 shows the deformation substructure for A orientation.

Table 2
Operative slip systems in Al-rich TiAl single crystals deformed with [201], [210] and [100] axes between 700 and 1000°C (● \{111\}(110) slip system, ▶ \{110\}(110) slip system, ○ \{111\}(110) slip system)

| Load axis | Composition | 700°C | 800°C | 900°C | 1000°C |
|-----------|-------------|-------|-------|-------|--------|
| A         | Ti–54.7at.%Al ● | ●  | ●  | ●  | ●  |
|           | Ti–58.0at.%Al ● | ●  | ●  | ●  | ●  |
| B         | Ti–54.7at.%Al ○  | ○  | ○  | ○  | ●  |
|           | Ti–58.0at.%Al ○  | ○  | ○  | ◆  | ◆  |
| C         | Ti–54.7at.%Al ○  | ○  | ○  | ◆  | ◆  |
|           | Ti–58.0at.%Al ○  | ○  | ○  | ◆  | ◆  |

Fig. 5. Slip markings on the surface of Ti–58.0 at.%Al single crystals deformed with [201], [210] and [100] axes in the temperature range between 700 and 900°C: (a) [201], 700°C; (b) [210], 700°C; (c) [100], 700°C; (d) [201], 800°C; (e) [210], 800°C; (f) [100], 800°C; (g) [201], 900°C; (h) [210], 900°C; (i) [100], 900°C.

In Ti–58.0 at.%Al single crystal deformed at 800°C. It consists of \frac{1}{2} \{110\} ordinary dislocations on (111); the dislocations are pinned at many points and are locked at the end of the bowed segments. The curved dislocations form many cusps. These cusps have also been reported in some Al-rich TiAl alloys and are thought to be formed by the cross-slip of the dislocation segments [12,14,16–18,21]. The cross-slip plane can be verified by tilting the foil and obtaining the edge-on image of dislocation segments on the cross-slip plane. Fig. 7(b)–(d) shows dislocation images in the same area with different incident beams. The dislocation segment marked by an arrow in Fig. 7(b) is fairly linear with the [110] beam direction while it curves with the [001] beam direction in Fig. 7(d). This suggests that the top segment lies on the (001) plane due to the cross-slip from (111) to (001) plane. However, since the bowed dislocation is projected as the longest segment with the [111] beam direction, the ordinary dislocation may form a cusp on (111).

Fig. 8(a)–(c) shows the deformation substructure for A orientation in Ti–58.0 at.%Al single crystal deformed at 900°C. According to the g-b analysis and the tilting experiment, the dislocations lay on the (110) plane and their
Burgers vector was \( \frac{1}{2} [\overline{1}0] \). The ordinary dislocations no longer show preferential alignment and many dislocation loops exist elongating along their edge orientation. Similar dislocation loops were also observed in Ti–54.7 at.%Al single crystals deformed at 900°C; these loops were previously reported in Al-rich TiAl single crystals [12,14]. The tilting experiments show that the elongated loops open wider with the [110] beam direction while they become narrower with the [001] beam direction. This indicates that the loops lie on the (110) slip plane. The density of dislocation loops slightly increased and they elongated with increasing Al concentration. The average sizes in Ti–54.7 at.%Al and Ti–58.0 at.%Al single crystals were about 110 and 70 nm, respectively. The sizes may be related to the frequency of the cross-slip events.

Fig. 9 shows an electron micrograph of the deformation substructure in Ti–58.0 at.%Al single crystal deformed at 1000°C for C orientation. Analysis showed the dislocations to be \( \frac{1}{2} [\overline{1}10] \) ordinary dislocations on (110) and \( \frac{1}{2} [110] \) ordinary dislocations on (1\overline{1}0). The [001] dislocation which was reported by Jiao et al. [13] was never observed at this orientation. The \( \frac{1}{2} [110] \) dislocations on (110) mostly

![Fig. 6. Temperature dependence of CRSS in Ti–58.0 at.%Al single crystals.](image)

Fig. 7. Deformation substructure in Ti–58.0 at.%Al single crystals deformed with [20\( 1 \)] axis at 800°C: (a) Beam direction (B)/[1\( 1 \)1], \( \vec{g} = [\overline{1}0] \); (b) B/[1\( 1 \)0], \( \vec{g} = 1\overline{1}0 \); (c) B/[1\( 1 \)1], \( \vec{g} = [\overline{1}0] \); (d) B/[00\( 1 \)], \( \vec{g} = [\overline{1}0] \); (b)–(d) high magnification images of dislocations in the same area.
stretching along [001], have an edge character and often form subboundaries. This suggests that the climb motion of the ordinary dislocations is easy even at 1000°C.

4. Discussions

Slip planes for $\frac{1}{2}(110)$ ordinary dislocations changed from {111} to {110} and partly {001} at high temperatures. The transition temperature depended on the crystal orientation and Al concentration. Development of the Al$_3$Ti$_3$ superstructure depending on the Al concentration affects the transition in the slip system as shown in Table 2. Effect of the Al$_3$Ti$_3$ superstructure on the slip transition and the anomalous strengthening will be discussed in this section. In Al-rich TiAl crystals, dislocations easily enter the Al$_3$Ti$_3$ superstructure due to the coherent phase boundary. A $\frac{1}{2}(110)$ ordinary dislocation can not create an APB on any slip plane in the L1$_0$ matrix but several types of APB with different energies are formed behind the motion of the dislocation on each plane in the Al$_3$Ti$_3$ superstructure [7]. The calculated APB energies on {111} produced by the ordinary dislocations were reported to be higher than those on {001} and {110} in the Al$_3$Ti$_3$ superstructure [9]. When the ordinary dislocations enter the Al$_3$Ti$_3$ superstructure in Ti–58.0 at.%Al crystal, they show a strong tendency to cross slip from {111} to (001) planes in order to decrease their APB energy at high temperatures. Some parts of

Fig. 8. Deformation substructure in Ti–58.0 at.%Al single crystals deformed with [201] axis at 900°C: (a) B/[110], $\overline{g} = \overline{1}0$; (b) B/[111], $\overline{g} = \overline{1}0$; (c) B/[001], $\overline{g} = \overline{1}1$.

Fig. 9. Weak beam image of ordinary dislocations in Ti–58.0 at.%Al single crystal deformed with [100] axis at 1000°C: beam direction $\parallel[110]$, $\overline{g} = \overline{1}1$. 

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ordinary dislocations gliding on \{111\} cross slip on \{110\} and/or \{001\} planes and may act as dragging points for the motion of the entire dislocations. The Al\textsubscript{2}Ti\textsubscript{3} ordering may provide a driving force for the cross slip onto \{110\} and/or \{001\} resulting in anomalous strengthening. Although there is no direct evidence of the Al\textsubscript{2}Ti\textsubscript{3} precipitates in Ti–54.7 at.%Al crystal, short range order of the Al\textsubscript{2}Ti\textsubscript{3} superstructure may assist the cross slip of segments of moving dislocations on \{111\} slip plane. A large number of dislocation loops in Ti–54.7 at.%Al deformed around and above the peak temperature suggests the strong tendency of cross slip of ordinary dislocations. In the Al\textsubscript{2}Ti\textsubscript{3} superstructure, \(\frac{1}{2}\{110\}\) dislocations must glide to create the APB which requires an additional stress for the motion of the dislocations. At high temperatures, the APB created behind dislocations may play an important role in their motion. The macroscopic slip plane changes from \{111\} to \{110\} to decrease the APB energy.

The climb motion of dislocations, which is controlled by diffusion of solute atoms and vacancies is known to have an important function in the deformation at high temperatures. The apparent activation energy for deformation (\(Q\)) is defined by the following equation:

\[
Q = \frac{R\delta \langle \log \sigma \rangle}{m\delta(1/T)}
\]

where \(m\) is the stress exponent factor, \(R\) the gas constant and \(\delta\) is the flow stress at the steady state. In this work, \(\sigma\) is the flow stress at 5\% plastic strain where the steady-state deformation was achieved. The activation energy showed no dependence on the operative slip system but depended strongly on the Al concentration. The activation energies of Ti–54.7 at.%Al and Ti–58.0 at.%Al single crystals were 360 and 450 kJ mol\(^{-1}\), respectively. In the ordered structure, the diffusion process is known to be more complex than that in disordered alloys and pure metals, and high activation energy is required for diffusion of solute atoms in the ordered state [22,23]. The diffusion in the Al\textsubscript{2}Ti\textsubscript{3} superstructure may be more difficult than that in the L\ell_0 structure since the Al\textsubscript{2}Ti\textsubscript{3} superstructure is formed by excess Al atoms replacing Ti atoms on the \{002\} layers and has four-times the periodicity with respect to the L\ell_0 structure along the \(a\)-axis. It was also reported that the activation energy for interdiffusion of Ti atoms in \(\gamma\)-TiAl increased with increasing Al concentration [24] and the activation energy for diffusion in Ti–54 at.%Al alloy was 295 kJ mol\(^{-1}\) [25,26]. Since the activation energy for deformation in Ti–54.7 at.%Al single crystals is much higher than that for diffusion, the thermal activation process for the motion of ordinary dislocations may be influenced by the Al\textsubscript{2}Ti\textsubscript{3} ordering. Development of the Al\textsubscript{2}Ti\textsubscript{3} superstructure in Ti–58.0 at.%Al alloy greatly increases the activation energy for diffusion. Deformation in Ti–58.0 at.%Al alloy may be influenced not only by diffusion but also by the glide and/or climb motion of ordinary dislocations which pass through the Al\textsubscript{2}Ti\textsubscript{3} superstructure.

5. Conclusions

1. The transition of slip plane from \{111\} to \{110\} and/or \{001\} occurs around the anomalous peak temperature. This may be due to the anisotropy of the energy of APB created by the motion of \(\frac{1}{2}\{110\}\) ordinary dislocation in the Al\textsubscript{2}Ti\textsubscript{3} superstructure.

2. Anomalous strengthening is related to development of the Al\textsubscript{2}Ti\textsubscript{3} superstructure. Some parts of the ordinary dislocation gliding on \{111\} plane cross slip on \{110\} and/or \{001\} plane at elevated temperatures and act as dragging points for the motion of the dislocation, resulting in anomalous strengthening.

3. The activation energy for the deformation in Ti–54.7 at.%Al and Ti–58.0 at.%Al single crystals is 360 and 450 kJ mol\(^{-1}\), respectively. The big difference in the activation energy is due to the Al\textsubscript{2}Ti\textsubscript{3} superstructure.

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