Concurrent design for NiAl-based (β/γ') two-phase alloys by controlling microstructure and texture

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Abstract

Control of the crystallography of NiAl(γ') precipitates along grain boundaries of NiAl(β) was systematically studied using β bicrystals with controlled orientations. γ' phase preferentially precipitated along β grain boundaries showing a film-like shape. The variants of γ' precipitates were uniquely selected, which satisfies the Kurdjumov–Sachs (K–S) relation with a neighboring β grain and deviates from the relation with another adjacent β grain. In the course of tensile deformation, fracture occurred preferentially at the (β/γ'-film) interface deviating from the K–S relation and the fracture stress decreased with increasing deviation angle from the K–S relation. For improvement of the coherency at the irrational (β/γ'-film) boundaries, the control of microstructure and crystal orientation distribution in (β/γ') two-phase polycrystals was next attempted by thermomechanical processing. After hot-compression in β phase region and subsequently annealing in (β/γ') two-phase region, γ' phase transformed from β phase with ⟨111⟩₀ fiber texture satisfying the K–S relation, resulting in the formation of ⟨110⟩₀ fiber texture. In particular, a large number of (β/γ'-film) boundaries became partially coherent. This thermomechanical processing was effective in controlling the crystallography of γ'-film along β grain boundaries and leads to the harmonic design of strength and ductility for (β/γ') two-phase alloys. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

NiAl(β) with the B2 structure is a potential candidate for high temperature structural materials because of its high melting point, extreme strength and good oxidation resistance at high temperatures [1–3]. The major subjects which should be improved before the industrial application are its poor ductility and low fracture toughness at ambient temperature, particularly grain boundary embrittlement. Since {110}(100)- and {100}(100)-slips are primarily operative and only three independent slip systems can be provided in β-NiAl, the strain compatibility at boundaries cannot be maintained during deformation, resulting in formation of an intergranular crack [4,5]. Since Ni₃Al(γ') with the L1₂ structure deforms by {111}(110)-slip and satisfies the von Mises criterion for general deformation providing more than five independent slip systems, precipitation of the γ' phase at grain boundaries is effective in suppressing the intergranular brittle fracture in β-NiAl [6–8]. Therefore, strong β-NiAl including ductile γ'-Ni₃Al can be regarded as the so-called concurrent designed material with high strength and good ductility.

For Ni–Al alloys containing 34–38at.%Al, γ' phase precipitates from the β parent phase by a diffusion process on cooling. β and γ' phases satisfy the Kurdjumov–Sachs (K–S) orientation relationship: {110}₀/{111}₁γ' and {111}₀γ'/{⟨110⟩₀γ'}. The γ' phase preferentially precipitates along β grain boundaries showing a film-like shape, resulting in the improvement of ductility. Although there exist 24 possible variants for the K–S relationship, a specific variant is preferentially selected in γ' phase precipitated at a grain boundary in an attempt to maintain the coherence with the neighboring β grains. Even if γ'-film precipitates along a grain boundary maintaining the K–S relation with a neighboring β grain, the orientation relationship between γ'-film and another adjacent β grain often deviates from the K–S relation. Fracture occurs preferentially at the incoherent (β/γ') interface near grain boundaries in (β/γ') two-phase alloys [10]. The deviation angle from the K–S relation at the irrational interphase boundary is known to be affected by a combination of orientations between two neighboring
grains, grain boundary planes and so on [10–12]. Therefore, the deviation angle from the K–S relation at the (β/γ'–film) interfaces is believed to influence the mechanical properties of (β/γ') two-phase alloys. β bicrystals with controlled orientations would provide us valuable information on the crystallography of γ'-film and its effect on the mechanical properties.

To improve the mechanical properties of NiAl-based alloys, control of microstructure and crystal orientation distribution is thought to be effective taking into account the coherence of the interface between β and γ' phases. Since orientation distribution of γ' precipitates and (β/γ') boundary character distribution are closely related to the orientation distribution of β matrix grains, control of the crystallographic texture of β matrix, which depends on the thermomechanical processing is necessary. Controlled rolling is known to be a representative means for controlling microstructure and texture in ferrite transformed from austenite during rolling of steels [13–15]. Similar thermomechanical processing is expected to be effective in controlling the texture of γ' phase precipitated from parent β phase in NiAl-based alloys, resulting in the ductility improvement.

In this paper, control of the crystallography of Ni3Al(γ') precipitates along grain boundaries was investigated using β bicrystals with controlled orientations. The effect of γ' precipitates on the mechanical properties of β bicrystals was also studied focusing on the deviation angle from the K–S relation at (β/γ') interphase boundaries. Moreover, control of microstructure and texture in two-phase (β/γ') polycrystals was attempted for the improvement of the coherency at (β/γ') interphase boundaries by thermomechanical processing.

2. Effect of (β/γ') interphase boundary on mechanical properties of β bicrystals

2.1. Experimental procedure for bicrystal study

Master ingots of Ni–Al alloys containing 38 at.%Al were prepared by melting high purity Ni and Al in a plasma arc furnace. Single crystals were grown from the ingots by the Bridgman method. After homogenizing at 1523 K for 48 h, oriented β bicrystals were made by a pressure bonding at an uniaxial pressure of about 5 MPa and at 1523 K in the temperature region of β single phase together two β single crystals (composed of β(1) and β(2) crystals). The orientation of β(1) crystal with {110} boundary plane was fixed, while β(2) crystals with various plane orientations were further rotated every 10° about the normal axis to the grain boundary plane as shown in Fig. 1. The β bicrystals were annealed at 1073 K for 24 h in (β/γ') two-phase region, in order to precipitate γ' phase along β grain boundaries. The tensile specimens were cut from the β bicrystals by spark-discharge machining such that the (β/γ') interphase boundary is perpendicular to the tensile axis, as shown in Fig. 2. Tensile tests were conducted in air at a constant cross-head speed of 0.03 mm min⁻¹ corresponding to an initial strain rate of 1.6 × 10⁻⁴ s⁻¹ and at ambient temperature. The specimen surfaces were electrolytically polished in a methanol-based solution containing 20% perchloric acid after mechanical polishing and then observed by an optical microscope. Fracture surfaces were examined by a scanning electron microscope. Crystallographic observations were carried out using an electron back-scatter diffraction pattern (EBSP) technique. The electron beam was automatically moved in 1 µm steps to develop an orientation map over an area 15 × 20 µm².

2.2. Crystallography of γ' precipitates along grain boundaries in β-NiAl bicrystals

Fig. 3 shows an example of EBSP analysis of the crystallography of γ'-film precipitated along a β grain boundary in a β bicrystal. In the orientation imaging micrograph (OIM), as shown in Fig. 3(a), γ' phase can be clearly distinguished from β phase; γ'-film is bounded by β(1) and β(2) grains. From the pole figures of β and γ' phase, as shown in Fig. 3(b) and (c), the film-like γ' phase and β(1) grain perfectly holds the K–S relationship of (110)β(1)/[(111)γ' and [111]β(1)/[(110)γ', while β(2) grain is irrational with the γ'-film. The deviation angle from the K–S relation at (β(2)/γ') interface is about 20°. Ameyama et al., reported that the variant selection of precipitates at the grain boundaries depends strongly on the orientation relationship between neighboring grains, grain boundary planes and so on [11,12]. The selected variant of γ' precipitates along β grain boundaries depends strongly on grain boundary plane: one of twelve [110]β planes in the neighboring β grains, which is most parallel to the grain boundary plane, is chosen as the close packed plane for the K–S relation (criterion 1). It is also noted that the deviation angle from the K–S relation at the irrational (β(2)/γ') interface should be as small as

![Fig. 1. A schematic illustration showing crystallography of β-NiAl bicrystal.](image)

![Fig. 2. Geometry of the tensile specimen of β-NiAl bicrystal with γ'-film.](image)
possible, resulting in the selection of the close packed direction (criterion 2). Thus, the variant of $\gamma'$ precipitates is uniquely selected due to these two criteria. Low energy ($\beta/\gamma'$) interfaces on both sides of $\gamma'$ precipitate may reduce the activation energy of $\gamma'$ nucleation and assist the precipitation at the $\beta$ grain boundary. Considering the variant selection of $\gamma'$ precipitates at the grain boundaries, it may be possible to control the variant of $\gamma'$-film by controlling the crystal orientation of neighboring $\beta$ grains, especially in $\beta$ bicrystals with controlled orientations. According to the criteria 1 and 2, $\gamma'$-film in $\beta$ bicrystals, used in this study, always satisfies the K–S relation with a neighboring $\beta(1)$ grain, while $\beta(2)$ and $\gamma'$ phases do not hold the relation. The deviation angle at ($\beta(2)/\gamma'$) interface can be controlled by rotating $\beta(2)$ grains with selected orientations as shown in Fig. 1.

2.3. Effect of deviation from the K–S relation at interphase boundary on the fracture

The calculated deviation angles from the K–S relation at the irrational ($\beta(2)/\gamma'$) interfaces are plotted against the rotation angles of $\beta(2)$ grain in Fig. 4. The orientation of $\beta(1)$ crystal was fixed such that grain boundary plane is $\{110\}_{\beta(1)}$. On the other hand, the $\beta(2)$ crystal was rotated every $10^\circ$ along the rotation axis perpendicular to grain

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Fig. 3. (a) OIM microstructure, (b and c) orientation relationship between $\beta(1)$ and film-like $\gamma'$ phase, and that between $\beta(2)$ and film-like $\gamma'$ phase, in $\beta$-NiAl bicrystal.

Fig. 4. Change in calculated deviation angle from the K–S relation at irrational ($\beta(2)/\gamma'$-film) interface with rotation angle of $\beta(2)$ crystal about the normal axis to various grain boundary planes. (a) $\{110\}_{\beta(2)}$, (b) $\{111\}_{\beta(2)}$, (c) $\{100\}_{\beta(2)}$. 
taking into account the criterion 2, periodically change at a period of 60° with the rotation angle of β(2) crystal. As the β(2) crystal is rotated about the normal of a grain boundary plane of \{111\}_β, the ideal deviation angle from the K–S relation at the irrational (β(2)/γ') interface reaches a maximum of about 35°. Moreover, the β(2) crystals with different orientations were selected to control the various deviation angles from the K–S relation. The deviation angle measured by the SEM–EBSP method is consistent with the theoretical calculation shown in Fig. 4, although the measured data is not shown here.

Fig. 5 shows microstructure of β bicrystal fractured at ambient temperature in β bicrystal with film-like γ'. The tensile axis of both β(1) and β(2) crystals were selected to \{110\}_β, and the deviation angle from the K–S relation against the irrational (β(2)/γ') interface was controlled to be about 20°. The fracture occurred preferentially at the (β(2)/γ') interface within the elastic limit, as shown in this figure. In this case, slip traces are observed in neither β matrix nor γ' precipitate.

The fracture stresses at the irrational (β/γ') interphase boundary are plotted against the deviation angle from the K–S relation in Fig. 5. In β bicrystals deformed in tension at ambient temperature, fracture occurred preferentially at the irrational (β/γ') interface, and the fracture stress decreases with increasing the deviation angle from the K–S relation. The fracture stress shows a maximum, when the deviation angle is almost zero, that is, γ' precipitate is also coherent with the neighboring β(2) crystal. In this case, fracture occurred not at (β/γ') interphase boundary but in β matrix grain. Although the fracture stress at the coherent (β/γ') interphase maintaining the K–S relation could not be obtained, the stress should be much higher than that of incoherent one.

Fig. 7 shows the microstructure of β bicrystal fractured at ambient temperature, which contain film-like γ' precipitate satisfying the K–S relation with both neighboring β crystals. Fig. 7(b) is a magnified photograph of Fig. 7(a). Slip traces are observed in film-like γ' phase and in β(1) crystal in the vicinity of (β(1)/γ') interface, and the operative slip planes were examined by a two-surfaces

Fig. 5. A scanning electron micrograph of β-NiAl bicrystal fractured at room temperature.

Fig. 6. Relationship between the fracture stress and the deviation angle from the K–S relation at irrational (β/γ') interphase boundary.

Fig. 7. Optical micrographs of β-NiAl bicrystal with γ'-film maintaining the K–S relation with both neighboring β(1) and β(2) crystals; deformed to fracture at room temperature. (b) is a high magnification image of (a).
trace analysis method. From the slip-trace analysis, the activated slip system in film-like γ’ phase is considered to be (11̅1)[101] which has the maximum Schmid factor among possible slip systems while the slip plane (01̅1) observed in β(1) phase did not follow Schmid law. Moreover, the ductile γ’ phase can yield even at low temperatures while the β phase is still deformed elastically at room temperature. This suggests slip transfer across the (β(1)/γ’) interface from ductile film-like γ’ to hard β(1) phase [16,17]. Therefore, the development of coherent (β/γ’) interphase boundaries could result in improving the embrittlement in NiAl-based (β/γ’) two-phase alloys.

3. Control of microstructure and crystal orientation distribution in (β/γ’) two-phase alloys by thermomechanical processing

3.1. Method for control of microstructure and texture in (β/γ’) two-phase alloys by thermomechanical processing

Master ingots of Ni–Al alloys containing 36 and 38 at.% Al were prepared by melting high purity Ni and Al in a plasma arc furnace. After homogenizing at 1523 K for 48 h, cylindrical specimens of 8 mm in diameter and 12 mm in height were cut from the ingots. The thermomechanical processing as schematically shown in Fig. 8 was applied for the specimens to control microstructure using a thermomechanical processing simulator (Fuji Electronic Industrial Thermecmaster-Z); the cylindrical specimens which were first annealed in an Ar gas atmosphere at 1523 K for 1 h to obtain β single phase containing no γ’ phase were compressed at a constant strain rate (ε) of 1 × 10^{-3} s^{-1} to true strain (ε) of 0.4 or 0.9 at 1523 K in the β single phase region; they were subsequently annealed at 1123 K for 1 h in (β/γ’) two-phase region under no applied stress, and then finally quenched by Ar gas flow. The test was sometimes stopped at the stage of thermomechanical processing and the sample was quenched by Ar gas flow to examine the evolution process of texture and microstructure. Ni–38 at.% Al alloy was used for this test because the microstructure of β phase could be frozen without martensitic transformation [18].

The treated sample was cut parallel to the compressive axis in the normal direction at the center of the cross-section by spark-discharge machining. The specimen surfaces were observed by an optical microscope. Thin foils for transmission electron microscopic observation, cut parallel to the compressive axis was perforated by a twin-jet method. Texture analysis and crystallographic observation were carried out using the EBSP technique.

3.2. Development of microstructure and texture in β phase during hot deformation

Fig. 9 shows EBSP analysis of β single phase in Ni–38 at.% Al polycrystals compressed at 1523 K and ε = 1 × 10^{-3} s^{-1}.

![Fig. 9. Orientation imaging of EBSP analysis for β single phase structure in Ni–38 at.% Al alloy compressed to various strains at 1523 K and ε = 1 × 10^{-3} s^{-1}. (a) Compressed to ε = 0.4. (b) 0.9. The thin and bold lines represent β grain boundaries whose misorientation angles (Δθ) are Δθ = 1–15° and Δθ > 15°, respectively. (c) and (d) are inverse pole figures of (a) and (b), respectively.](image-url)
to $\epsilon = 0.4$ and 0.9. The thin and bold lines represent $\beta$ grain boundaries whose misorientation angles ($\Delta \theta$) are $1-15^\circ$ and more than $15^\circ$, respectively. In the present study, a grain surrounded by boundaries with $\Delta \theta = 1-15^\circ$ is regarded as a subgrain. At $\epsilon = 0.4$, initial $\beta$ grains are reduced in thickness by hot compression, showing a pancake-like morphology and serrated grain boundaries with the development of numerous subgrains in the initial grains (Fig. 9(a)). In contrast, the equiaxed $\beta$ grains of 300 $\mu$m in size surrounded by high angle boundaries ($\Delta \theta > 15^\circ$) are homogeneously developed at $\epsilon = 0.9$ and also contains numerous subgrains, as shown in Fig. 9(b). The refinement of $\beta$ grains and increase in the frequency of high angle boundaries strongly suggest the occurrence of dynamic recrystallization in $\beta$ phase during hot deformation. Although the photograph is not shown here, numerous dislocations and subgrains were confirmed in grains by TEM. The local migration of serrated grain boundaries leads to the formation of dynamically recrystallized grains [19,20]. Since the serration of $\beta$ grain boundaries becomes more irregular and frequent as compressive reduction increases, the dynamic recrystallization occurs more easily at higher strains. In addition, so-called ‘geometric dynamic recrystallization’ may occur in $\beta$ matrix during hot compression [21]; if grains become thinner and their size approaches to the height of serration of grain boundaries, the serrated boundary impinges on next grain and a new grain surrounded by high angle boundaries is geometrically formed. In the present study, the strongly serrated $\beta$ grain boundaries imply the occurrence of the geometric dynamic recrystallization. Moreover, the inverse pole figures for the compressed plane are also analyzed in Fig. 9(c) and (d). The grains examined are randomly oriented at $\epsilon = 0.4$, while strong (111)$_{\beta}$ fiber texture along the compressive axis is developed and more than 90% of $\beta$ grains are aligned along (111) at $\epsilon = 0.9$. In general, dynamic recrystallization accompanied by grain boundary serration does not result in texture formation, since the recrystallized grains inherit the orientation of the initial grains [22]. However, some researchers reported that lattice rotation by activated slips during hot deformation induces the development of texture during dynamic recrystallization [23–27]. The crystal rotation can be predicted by numerical calculation.

In the present paper, stable orientation by slip deformation in $\beta$ phase was calculated by using van Houtte’s algorithm [28–31]. Three different slip systems of {110}(100), {100}(100) and {110}(110) which were operative at higher temperatures in this alloy were taken into account in the calculation. Moreover, two types of calculation were done based on the full and relaxed constraint Taylor models [32]. In the full constraint Taylor model, five independent slip systems were selected among all the possible combinations to minimize the internal work of slip deformation, while only three slip systems were assumed to be operative to achieve the macroscopic plastic strain in the relaxed constraint condition because

Fig. 10. Simulated compression texture evolution. (a) FC-model with assumption of {110}(100) and {110}(110)-slip; (b) RC-model with assumption of {110}(100), {100}(100)-, and {110}(110)-slip.

the two strain components of $\epsilon_{xy}$ and $\epsilon_{xz}$ were relaxed with $x$ being the compressive axis. The lattice rotations calculated by these two models are displayed in the inverse pole figures in Fig. 10. Both the full (Fig. 10(a)) and relaxed (Fig. 10(b)) constraint Taylor models predict that the (111)$_{\beta}$ direction of $\beta$ grains rotates to the compressive axis. Therefore, the lattice rotation by slip deformation results in the development of (111)$_{\beta}$ fiber texture in $\beta$ phase along compressive axis.

3.3. Crystallography of $\gamma'$ phase precipitated from hot-compressed $\beta$ phase

There was no significant difference in deformation behavior or microstructural change during hot deformation in supersaturated $\beta$ phase region between Ni–38 at.%Al and Ni–36 at.%Al alloys. Since a larger quantity of $\gamma'$ phase can precipitate in Ni–36 at.%Al alloy from supersaturated $\beta$ phase during annealing at 1123 K than in Ni–38 at.%Al alloy, Ni–36 at.%Al polycrystals were used to examine microstructure and crystallography of $\gamma'$ precipitates during annealing.

Fig. 11 shows ($\beta/\gamma'$) two-phase microstructure in Ni–36 at.%Al polycrystal annealed at 1123 K after hot compression at 1523 K and $\dot{\epsilon} = 1 \times 10^{-3}$ s$^{-1}$ to $\epsilon = 0.9$. Lath-$\gamma'$ precipitates are densely and homogeneously distributed within $\beta$ grains. Moreover, the film-like $\gamma'$ phase is

Fig. 11. An optical micrograph of Ni–36 at.%Al alloy annealed in ($\beta/\gamma'$) two-phase region after hot compression to $\epsilon = 0.9$ at 1523 K and at $\dot{\epsilon} = 1 \times 10^{-3}$ s$^{-1}$.
developed at the circumference of β grains. The β grain size decreases to about one-tenth of the initial grains. Although the data is not shown here, the formation of γ′-film-like precipitates suppresses the grain boundary embrittlement, resulting in improvement of ductility of NiAl-based alloys [10].

Fig. 12 shows the inverse pole figure of γ′ precipitates for the compressive plane in Ni–36 at.% Al polycrystal annealed at 1123 K after hot deformation. Orientation of γ′ precipitates along the compressive axis was concentrated along the (110)γ′ direction. Since γ′ phase precipitates from β phase on the basis of the K–S relationship, the formation of (110)γ′ fiber texture for γ′ phase is closely related to the development of (111)β fiber texture in β matrix phase during hot deformation.

The crystallography of γ′-film precipitated along β grain boundaries was investigated in Ni–36 at.% Al polycrystal annealed at 1123 K after hot deformation. An example is given in Fig. 13. The γ′-film is bounded by β(1) and β(2) grains as shown in Fig. 13(a). From the pole figures of β and γ′ phase, the film-like γ′ phase and β(1) grain perfectly holds the K–S relationship of (110)β(1)//[111]γ′ and [111]β(1)//[110]γ′ (Fig. 13(b)), while β(2) grain is irrational with the γ′-film (Fig. 13(c)). But, the deviation angle from the K–S relation is small as about 5°. This suggests that the deviation angle from the K–S relation at the irrational (β/γ′) boundaries is reduced by thermomechanical processing.

4. Conclusions

Control of microstructure and texture in Ni–Al(β/γ′) two-phase alloys was attempted by thermomechanical processing focusing on the effect of crystallography of γ′ precipitates along β grain boundaries on mechanical properties in (β/γ′) two-phase alloys, and the following conclusions were reached:

1. The ductile γ′ phase preferentially precipitates along brittle β grain boundaries forming a film-like shape and the variant selection of the γ′ precipitates at β grain boundary depends strongly on the orientation relationship between neighboring grains, grain boundary planes and so on. Considering the variant selection of γ′-film along β grain boundaries, the variant of γ′-film in β
bicrystals is controlled, which maintains the K–S relation with a neighboring β grain and deviates from the K–S relation with another adjacent β grain. Fracture at ambient temperature occurs preferentially at the (β/γ) interface deviated from the K–S relation and the fracture stress decreases with increasing deviation angle from the K–S relation.

2. After hot deformation in β phase region, equiaxed β grains surrounded by high angle boundaries are homogeneously developed due to dynamic recrystallization under adequate condition. Moreover, strong (111) fiber texture evolves parallel to the compressive axis in β phase because of the lattice rotation during hot deformation. After annealing in (β/γ) two-phase region, lath γ precipitates and film-like γ precipitates are formed in β grains and along β grain boundaries, respectively. The crystallography of γ' phase depends strongly on the orientation distribution of β phase controlled by thermomechanical processing. (110)γ' fiber texture of γ' precipitates inherits the (111)β fiber texture in β matrix to hold the K–S orientation relationship during annealing. For γ'-film-like precipitates among β grain boundaries, the distribution of the deviation angle from the ideal K–S relationship is shifted to the lower angle one by the thermomechanical processing.

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