Development of Microstructure and Texture in Rapidly Solidified and Annealed Ni–Al Alloys

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Development of microstructure and crystal orientation distribution in melt-spun and subsequently annealed Ni–36 at%Al and Ni–38 at%Al alloy ribbons was investigated. In Ni–36 at%Al ribbon, the martensitic transformation from \( \beta \) to \( \beta' \) phase occurred during rapid cooling showing strong (110)\(_\beta\) fiber texture, while \( \beta' \) phase with strong (100)\(_\beta\) texture was frozen in Ni–38 at%Al ribbon. During annealing at 1073 K, a peculiar fiber texture of strong (100)\(_\beta\) and (110)\(_\beta\) texture was developed in Ni–36 at%Al ribbon. Formation of the texture was discussed focusing on the initial microstructure and the orientation relationship between phases during phase transformation.

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1. Introduction

Considerable attention has been paid in recent years to NiAl-based shape memory alloys because of their phase transformation at high temperatures.\(^1\)\(^,\)\(^2\) However, poor ductility and low fracture toughness of NiAl(\( \beta \)) with the B2 structure at ambient temperature should be overcome before practical application as a shape memory device.\(^3\) Precipitation of Ni\(_3\)Al(\( \gamma' \)) with the L\(_1\)\(_2\) structure along \( \beta \) grain boundaries was reported to be effective in suppressing the brittle fracture of \( \beta \)-NiAl.\(^4\)\(^,\)\(^5\) In Ni–Al alloys containing 34–38 at%Al, phase transformation from \( \beta \) to \( \gamma' \) phase occurs satisfying the Kurdjumov-Sachs (K-S) relationship by a diffusion process during slow cooling.\(^6\) To improve mechanical properties of NiAl-based alloys, the microstructure and texture must be controlled, with emphasis on the phase transformation process and the orientation relation between \( \beta \) and \( \gamma' \) phases. In our previous paper, the microstructure and the orientation distribution of two-phase (\( \beta' / \gamma' \)) alloys were controlled by thermomechanical processing. Formation of texture in \( \beta \) matrix by hot deformation was effective in controlling the crystallography of \( \gamma' \) phase along \( \beta \) grain boundaries.\(^7\) Rapid quenching techniques can achieve a non-equilibrium state and a unique texture which differs from that obtained by a conventional fabrication method.\(^8\)\(^,\)\(^9\) In addition, rapid cooling from the \( \beta \) single-phase region induces the thermoelastic martensitic transformation to NiAl(\( \beta' \)) with the L\(_1\)\(_0\) structure and the transition from \( \beta \) to \( \beta' \) phase occurs accompanied by Bain distortion, satisfying the crystallographic orientation relation between their phases.\(^10\) Therefore, microstructure control of NiAl-based alloys through the martensitic transformation may result in the formation of a peculiar microstructure in (\( \beta' / \gamma' \)) two-phase alloys.

In this paper, we report the development of microstructure and crystal orientation distribution in rapidly solidified and subsequently annealed Ni–Al alloys, focusing on the orientation relation between phases formed by phase transformation.

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2. Experimental Procedure

Two mother ingots of Ni–36 at%Al and Ni–38 at%Al alloys were prepared by arc melting in a purified argon atmosphere. Rapidly solidified ribbons with a cross section of 3 \( \times \) 0.04 mm\(^2\) were produced at 42 ms\(^{-1}\) by a single-roller melt-spinning method in an argon atmosphere. The rapidly solidified Ni–36 at%Al alloy ribbons were annealed in quartz capsule filled with argon gas for 36 s–24 h at 1073 K in the (\( \beta' / \gamma' \)) two-phase region. Ni–38 at%Al alloy ribbons were used to examine the texture and microstructure of \( \beta \) matrix since the microstructure of \( \beta \) single phase could be frozen accompanied by no martensitic transformation. Microstructural observation and grain orientation measurement were carried out on the surface plane of ribbons. The microstructure was observed by a Hitachi-H800 transmission electron microscope (TEM) operated at 200kV. Texture and crystallographic measurement were made by an electron backscatter diffraction pattern (EBSP) technique. The electron beam was automatically moved in 1 \( \mu \)m steps to develop orientation maps in an area of about 100 \( \times \) 60 \( \mu \)m\(^2\).

3. Results and Discussion

3.1 Rapidly solidified Ni–Al alloy ribbons

Figures 1(a) and (b) are TEM micrographs of rapidly solidified Ni–36 at%Al and Ni–38 at%Al ribbons, respectively. The microstructure of Ni–36 at%Al ribbon in Fig. 1(a) consists of \( \beta' \) single phase with thin martensite plates formed in the prior \( \beta \) grains with an average grain size of about 3 \( \mu \)m. A large number of twins exists within the martensite plates although they are not clearly seen in Fig. 1(a). In Ni–36 at%Al ribbon, \( \beta \) phase completely transforms to \( \beta' \) phase during rapid quenching since the martensite-start temperature is about 500 K.\(^13\) In contrast, the saturated \( \beta \) single phase is obtained in Ni–38 at%Al ribbon in Fig. 1(b) showing no martensitic transformation. A typical diffraction pattern taken from a martensite plate with [101]\(_{\beta}\) zone axis is shown in Fig. 2(a). The diffraction pattern can be regarded as a twin pattern whose plane of twinning is (1 1 1)\(_{\beta} \).\(^11\)\(^,\)\(^12\) It should be also
Fig. 1  TEM micrographs of rapidly solidified (a) Ni–36 at%Al and (b) Ni–38 at%Al alloy ribbons.

Fig. 2  (a) A (101)β electron diffraction pattern from a localized region in Ni–36 at%Al ribbon. (b) The corresponding bright field image.

noted that the twin pattern of β' phase with the L1₀ structure can be distinguished from that of γ' phase with the L1₂ structure by comparing their superlattice reflections. Moreover, streaks are observed to be aligned normal to (111)β' plane due to the thin twin plate. The bright field image shown in Fig. 2(b) clearly indicates the presence of high density twins in a variant.

Figures 3(a) and (b) show the inverse pole figures for the surface plane of Ni–36 at%Al and Ni–38 at%Al ribbons, respectively. In Ni–36 at%Al ribbon, β' single phase exhibits strong (110)β' and weak (100)β' fiber texture as shown in Fig. 3(a). The intensity of (110)β' poles for β' phase is about two times higher than that of (100)β' poles. In contrast, (100)β' fiber texture strongly develops in β matrix in Ni–38 at%Al ribbon (Fig. 3(b)). This texture may result from the preferential crystal growth along cubic axis during rapid solidification. The transition from β to β' phase is known to occur in Ni–36 at%Al ribbon accompanied by Bain distortion; dilation along one axis and contraction along other (100)β axes resulting in the (100)β formation of three groups in martensite variants. 14 In a rapid solidification process, (100)β fiber texture develops. If the three variants are equivalently selected, the fraction of β' variants oriented near (110)β' direction along the normal direction of the ribbon is double that near (100)β'.

Fig. 3  Inverse pole figures of EBSP analysis on the surface plane of rapidly solidified (a) Ni–36 at%Al and (b) Ni–38 at%Al alloy ribbons.

orientation following Bain distortion. This hypothesis is in good agreement with the experimental results shown in Fig. 3(a).

3.2 Development of microstructure in Ni–36 at%Al alloy ribbon during annealing in (β/γ') two-phase region

Figure 4 shows TEM micrographs in Ni–36 at%Al ribbon annealed at 1073 K in (β/γ') two-phase region. After annealing for 30 s (Fig. 4(a)), γ' phase preferentially precipitates along the prior β grain boundaries, since the nucleation at the boundaries is energetically favourable. In contrast, β phase cannot be seen in the figure, although the specimen was an-
nealed in (β'/γ') two-phase region. Even if the reverse transformation from β' martensite to β phase partly occurs interior grains during annealing, β phase transforms to β' martensite again upon cooling to room temperature. In further annealing for 100 s, β' phase containing martensite plates transforms to β and γ' alternately, resulting in formation of a lamellar structure composed of β and γ' phases (Fig. 4(b)). After annealing for 24 h (Fig. 4(c)), fine equiaxed grains composed of β and γ' phases with an average diameter of about 3 μm are obtained, satisfying the orientation relations with β' phase. Generally, anomalous grain growth occurs in cast β alloy and grains of a few millimeters are obtained. However, γ' precipitates in rapidly solidified Ni-Al alloys effectively restrict the migration of β grain boundary, resulting in the fine grain structure.

In (β'/γ') lamellar structure in Fig. 4(b), highly dense twins appear in γ' phase as indicated by an arrow in Fig. 5(a). A diffraction pattern taken from γ' plate with [101]γ' zone (Fig. 5(b)) can be interpreted as a twin pattern of the L12 structure with (111)γ' twinning plane. Formation of numerous twins in γ' phase may be due to the characteristics of phase transformation from β' to γ' phase; since both β' and γ' phases have fcc-based superlattice structure, the twins in γ' phase are inherited from β' martensites resulting in the orientation relationship between phases formed by phase transformation. On the other hand, the reverse transformation from β' to β phase during annealing can be detected from [100]β diffraction pattern in β phase as shown in Fig. 5(c). Streaks along the (110)β directions are also observed in the pattern, which is typical in β matrix phase accompanied by a peculiar tweed contrast in the bright field image.

Figure 6 shows the inverse pole figures of the surface plane in Ni-36 at%Al ribbon annealed for 24 h at 1073 K in (β'/γ') two-phase region. A peculiar fiber texture along the normal direction of ribbons develops in both β and γ' phases; β phase shows strong [100]β fiber texture (Fig. 6(a)), while γ' phase exhibits strong (110)γ' and weak (100)γ' fiber texture (Fig. 6(b)). The fiber texture in γ' phase along the surface plane direction is believed to be formed during annealing to satisfy the [100]γ' || [100]γ' and (100)γ' || (100)γ' orientation relationship with β' phase. In contrast, a reverse transformation from β' to β phase occurs without precipitation of γ' phase and a peculiar (100)β fiber texture, which is identical with that of as solidified state in Ni-38 at%Al ribbon, is formed in β phase.

Figure 7 shows schematic illustrations of the formation process of (β'/γ') two-phase fiber texture along the surface plane direction in Ni-36 at%Al ribbon annealed in the (β'/γ') two-phase region. In the ribbon, the fiber texture in β' phase is formed at stage I accompanied by grain boundaries and martensite plates which may act as preferential nucleation
sites of $\gamma'$ phase. At stage II, the phase transformation from $\beta'$ to $\gamma'$ occurs preferentially along grain boundaries and martensite plates during annealing at 1073 K, while residual $\beta'$ phase is reversibly transformed to $\beta$ phase accompanied by no precipitation of $\gamma'$ phase. The $\beta$ and $\gamma'$ phases are then transformed from $\beta'$ phase with fiber texture which satisfies the orientation relationship between their phases, resulting in the peculiar fiber texture in $(\beta/\gamma')$ two-phase structure at stage III. The formation of the $(\beta/\gamma')$ two-phase fiber texture in annealed Ni–36 at%Al ribbon is closely related to not only the initial microstructure and crystal orientation distribution in $\beta'$ phase before annealing but also the orientation relationship between phases during phase transformation. The $(\beta/\gamma')$ two-phase fiber texture may exhibit certain specific features with respect to the mechanical properties of Ni–Al alloys.

4. Conclusions

Development of the microstructure and the crystal orientation distribution in rapidly solidified and subsequently annealed Ni–Al alloys was examined focusing on the crystallographic orientation relation between the two phases during phase transformation, and following conclusions were reached.

(1) The rapidly solidified Ni–36 at%Al ribbon consists of $\beta'$ single-phase structure with strong $(110)_\beta$ and weak $(100)_{\beta'}$ fiber texture along surface plane normal, while $\beta$ phase with strong $(100)_\beta$ texture is frozen in Ni–38 at%Al ribbon. Since the martensitic transformation from $\beta$ to $\beta'$ phase occurs during rapid cooling accompanied by Bain distortion, development of $(110)_{\beta'}$ fiber texture is due to the formation of $(100)_\beta$ fiber texture in $\beta$ phase matrix.

(2) The $(\beta/\gamma')$ two-phase fiber texture in the normal direction of the Ni–36 at%Al ribbon surface is formed even during annealing in $(\beta/\gamma')$ two phase region. Development of $(\beta/\gamma')$ two-phase fiber texture is closely related to not only the initial microstructure and crystal orientation distribution in $\beta'$ phase before annealing but also the crystallographic orientation relation between two phases during phase transformation.

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