Microstructure and compressive flow stress of directionally solidified ternary Ni$_3$(Al, Nb) and quaternary Ni$_3$(Al, Nb, Ti) alloys with duplex phase

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Received 19 November 2001; revised 25 December 2001; accepted 25 December 2001

Abstract

The microstructure and compressive flow stress of directionally solidified ternary Ni$_3$(Al, Nb) and quaternary Ni$_3$(Al, Nb, Ti) alloys were examined. Three compositions of Ni–16.0 at.% Nb–9.0 at.% Al (Alloy 1), Ni–13.3 at.% Nb–7.5 at.% Al–4.2 at.% Ti (Alloy 2) and Ni–10.7 at.% Nb–6.0 at.% Al–8.3 at.% Ti (Alloy 3) were selected for investigation. Alloy 1 was composed of the L1$_2$ and the D0$_{19}$ phases while the constituent phases varied for the D0$_{19}$ and the D0$_0$ phases for Alloys 2 and 3 with Ti content. The definite crystallographic relationship was observed between the D0$_{19}$ and the D0$_0$ phases to be (0001)$_{D0_{19}}$///(001)$_{D0_0}$ and (1120)$_{D0_{19}}$///(100)$_{D0_0}$ in Alloy 3. Compression tests were conducted along the growth direction in the temperature ranging from room temperature to 1000 °C. Alloy 1 exhibited high yield stress at low temperatures, but it rapidly decreased above 700 °C. Similar temperature dependence of yield stress was observed in Alloy 2, although the onset temperature of a rapid decrease in yield stress was somewhat lower. Alloy 3 with the highest Ti content showed the lowest compressive strength among the three alloys, while relatively good low-temperature ductility was obtained in Alloy 3. Yield stress of Alloy 3 exhibited anomalous strengthening behaviour accompanied by the basal slip in both D0$_{19}$ and D0$_0$ phases. Transition in operative slip systems from the basal slip to the prism slip occurred at the peak temperature of yield stress anomaly (600 °C), resulting in a gradual decrease in yield stress. Slip transfer behaviour between the D0$_{19}$ and the D0$_0$ phases was briefly discussed. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Geometrically close-packed structure; Directional solidification; Ni–Nb–Al alloy; Compressive flow stress; Basal slip; Anomalous strengthening; Orientation relationship; Slip transfer

1. Introduction

Many Ni$_3$X type intermetallics (X = Al, Ti, Nb, Sn, V, etc.) are known to crystallise in various geometrically close-packed (GCP) crystal structures which are constructed by various stacking sequences with a combination of twofold hexagonal layer (h-layer) and threefold cubic layer (c-layer). In addition, some ordered atomic arrangements are produced on the close-packed plane: triangular (T-type), rectangular (R-type) and compound type (TR-type) in the GCP phases [1,2]. Among the Ni-based GCP phases, most attention has been paid to Ni$_3$Al with the L1$_2$ structure because of its good deformability and superior high-temperature strength including anomalous strengthening behaviour. The important role of Ni$_3$Al in Ni-base superalloys has been well recognised to be a dominant constituent for reinforcement at high temperatures. Recently, we reported that the yield stress anomaly appears not only in the Ni$_3$Al(L1$_2$) phase, but also in other GCP phases of Ni$_3$Nb(D0$_{19}$), Ni$_3$Ti(D0$_{21}$) and Ni$_3$Sn(D0$_{19}$) by the slip operating on the close-packed plane [3,4]. Among them, Ni$_3$Al(L1$_2$), Ni$_3$Nb(D0$_{19}$) and Ni$_3$Sn(D0$_{19}$) have strong phase stabilities up to the temperatures close to their melting points. Therefore, multi-phase alloys composed of the above Ni-based GCP phases are expected to be developed as a new high-temperature structural material under a harmonic material design. Very recently, the detailed study on the phase relation and microstructure in Ni$_3$Al–Ni$_3$Nb–Ni$_3$Ti pseudo-ternary alloy system was reported by Tomihisa et al. [5]. However, the information on the mechanical behaviour of the pseudo-ternary alloys is still limited [6].

Directional solidification (DS) is one of the useful fabrication processes to control the microstructures and to
machining from the DS crystals. Loading axes were chosen to be parallel to the longitudinal (growth) direction. Compression tests were performed on an Instron-type testing machine at a nominal strain rate of $1.7 \times 10^{-4} \text{ s}^{-1}$ in the temperature range from room temperature (RT) to 1000 °C in a vacuum. Deformation markings on the specimen surface were observed using an optical microscope with Nomarski interference contrast. Deformation substructures and the crystallographic relationship between the constituent phases were examined in a transmission electron microscope (TEM) (JEOL JEM-3010) operated at 300 kV.

3. Results and discussion

3.1. Microstructure and constituent phase

Fig. 1 shows the XRD profiles of Alloys 1–3 after the DS treatment. As reported previously [7–14], Alloy 1 with the Ni–Al–Nb ternary eutectic composition was confirmed to be composed of the L1$_2$(Ni$_3$Al) and the D0$_5$(Ni$_3$Nb) phases. However, the L1$_2$(Ni$_3$Al) phase disappeared by Ti addition. The D0$_5$(Ni$_3$Ti) phase appeared in addition to the D0$_5$(Ni$_3$Nb) phase in Alloys 2 and 3. Lattice constants and chemical compositions of the constituent phases of these alloys are listed in Table 1, where the chemical compositions were determined by SEM–EDS method. Large amounts of Nb and Al elements were contained in the D0$_5$(Ni$_3$Ti) phase. These results showed in good accordance with the recent report by Tomihisa et al. [5].

The GCP crystal structure is characterized by its stacking sequence and ordered configuration on the close-packed plane. The structures of L1$_2$ and D0$_5$ phases observed in Alloy 1, therefore, can be represented to be cT- and hR-structure, respectively. The structure of D0$_5$ phase is represented to be chT-structure, that is, the minority atoms are arranged in triangular fashion on the close-packed plane, and the close-packed planes are stacked in the sequence of fourfold ABAC. Hence, this structure can be regarded as one having an intermediate stacking sequence between hexagonal close-packed (HCP) and face-centred-cubic (FCC) structures. This structure is generally called a double hexagonal close-packed (DHCP) structure.

Fig. 2 shows the microstructures of the three DS alloys observed on transverse and longitudinal sections in the direction of crystal growth. The growth directions in Figs. 2(b), (d) and (f) are in the vertical direction. A fine lamellar structure composed of the D0$_5$ and the L1$_2$ phases is observed in Alloy 1 as reported previously [7–14], accompanied by the primary D0$_5$ phases. The lamellar structure is developed along the longitudinal direction. In Alloy 2, precipitates are homogeneously distributed in the matrix phase. According to XRD and SEM–EDS analysis, the matrix phase and the precipitates are the D0$_5$ and the D0$_5$ phases, respectively. Some D0$_5$ precipitates elongate roughly along the longitudinal direction, but the fine
Table 1
Lattice constants and analysed chemical compositions of constituent phases in the three directionally solidified alloys

| Crystal structure | Lattice constant (nm) | Chemical composition (at.%) |
|-------------------|-----------------------|-----------------------------|
|                   | $a$       | $b$       | $c$       | Ni  | Nb  | Al   | Ti   |
| Alloy 1 (Ni–16.0 at.%Nb–9.0 at.%Al) | L1$_2$ (cubic) | 0.3600 |              |     |     |      |      |
|                   | D0$_0$     | 0.5118 | 0.4237 | 0.4534 | 72.9 | 9.5  | 17.6 |
| Alloy 2 (Ni–13.3 at.%Nb–7.5 at.%Al–4.2 at.%Ti) | D0$_{24}$ (hexagonal) | 0.5117 | 0.8333 |              | 73.9 | 11.2 | 10.6 | 4.3  |
|                   | D0$_0$     | 0.5117 | 0.4227 | 0.4533 | 73.0 | 21.7 | 1.8  | 3.5  |
| Alloy 3 (Ni–10.7 at.%Nb–6.0 at.%Al–8.3 at.%Ti) | D0$_{24}$ (hexagonal) | 0.5117 | 0.8334 |              | 73.4 | 9.3  | 8.1  | 9.2  |
|                   | D0$_0$     | 0.5116 | 0.4218 | 0.4529 | 71.7 | 20.4 | 2.1  | 5.8  |

lamellae formed in Alloy 1 are rarely observed. Alloy 3 is also composed of the D0$_{24}$ and the D0$_0$ phases, but the morphology is different from that in Alloy 2. The grains of D0$_{24}$ matrix phase grow largely in Alloy 3. The plate-like precipitates of the D0$_0$ phase form colonies in the D0$_{24}$ matrix phase. The colonies are roughly aligned along the longitudinal direction (the deviation angle is within 20°). From an analysis of the selected area electron diffraction (SAED) pattern in the TEM and X-ray back Laue diffraction method, it was found that the colonies were aligned nearly parallel to the (0001) basal plane in the D0$_{24}$ matrix phase. Details of the crystal orientation relationship between the constituent phases are described in Section 3.2.

3.2. Orientation relationship between the constituent phases

Microstructure and deformation substructure in Alloy 3 were examined in the TEM. A thin foil for observation was cut from the Alloy 3 sample compressed to 1% at RT, parallel to the growth direction. Fig. 3(a) shows the bright field image of the microstructure. A large number of dislocations with straight morphology exist in both the phases. Focusing on the geometry of interfaces between the D0$_{24}$ and D0$_0$ phases, some interfaces are on the basal or prism plane with flat shapes. But most interfaces have the waved morphology as shown in Fig. 3(a), and those interfaces do not show a tendency to lie on a specific crystallographic plane.

It is known that the L1$_2$(Ni$_3$Al) and the D0$_2$(Ni$_3$Nb) phases which compose lamellar structure in the ternary DS alloy show the definite orientation relationship as follows [8]:

lamellar interface : \{111\}_{L1_2}/(010)_{D0_0} \quad (1)
growth direction : \langle 110\rangle_{L1_2}/(100)_{D0_0} \quad (2)

Constituent phases in Alloy 1 are considered to satisfy the above relationship. The orientation relationship between the D0$_{24}$ and the D0$_0$ phases in Alloy 3 was examined from an SAED pattern analysis. Fig. 3(b) and (c) show the SAED patterns taken from the areas marked A and B in Fig. 3(a),

Fig. 2. Optical micrographs of microstructures in the three alloys in (a,b) Alloy 1; (c,d) Alloy 2; (e,f) Alloy 3. Figures (a), (c) and (e), and (b), (d) and (f) are observed on transverse and longitudinal sections, respectively.
Fig. 3. (a) Bright field image of microstructure in Alloy 3 sample deformed to 1% at RT. (b,c) Corresponding SAED patterns taken from the areas marked A and B in (a), identified as the (2110) and (102) zone diffraction patterns in the D0₄ and the D0₅ phases, respectively. (d,e) SAED patterns taken from the same areas after tilting the foil by 30° along [0001] axis in the D0₄ matrix phase, identified to be (1010) and (001) zone diffraction patterns, respectively.

respectively. These were identified as the (2110) and (102) zone diffraction patterns in the D0₂₄ and the D0₄ phases, respectively. Fig. 3(d) and (e) show the SAED patterns taken from the same areas after tilting the foil by 30° along [0001] axis in the D0₂₄ matrix phase. They correspond to (1010) and (001) zone diffraction patterns, respectively. Some extra spots which are unexpected to appear in the D0₂₄ structure are clearly observed in the (1010) SAED pattern at the positions indicated by arrows, suggesting the solute Nb and Al atoms are arranged in the D0₂₄(Ni₃Ti) phase with higher-ordered symmetry. From the above analysis, it was confirmed that there exists the definite crystallographic relationship between the D0₂₄ and the D0₄ phases as follows:

\[
(0001)_{\text{D0}_4} / (010)_{\text{D0}_4} \quad (3)
\]

\[
<11\bar{2}0>_{\text{D0}_4} / (100)_{\text{D0}_4} \quad (4)
\]

The above orientation relationship is schematically shown with the atomic configurations of both the phases in Fig. 4. Here the structure of the D0₂₄ and the D0₄ phases are based on DHCP and HCP unit lattice, respectively. The above relationship indicates the close-packed basal planes and the direction of the shortest unit vector in both the phases overlap each other, similar to the relation reported between the L1₂ and the D0₄ phases [8]. The orientation relationship observed in Alloy 3 sample in this study is in good agreement with the previous report on that observed in the Ni–13.5 at.%Nb–7.5 at.%Al ternary DS alloy which is composed of Ni solid solution, D0₄ and D0₂₄ phases [16]. Whether or not the constituent phases in Alloy 2 show the same orientation relationship has not been examined yet.

The atomic arrangements on the basal plane differ between the D0₂₄ and the D0₄ phases. Minority atoms are arranged as rectangular and triangular fashion in the D0₄ and the D0₂₄ phases respectively, as shown in Fig. 4. Since the grains of D0₂₄ matrix phase grew largely in the DS process in Alloy 3, the D0₂₄ matrix phase could be regarded to have a single-crystalline form in the compressive specimen size level (dimension of 2 × 2 mm² × 5 mm). Taking the difference of atomic configuration on the close-packed plane into consideration, therefore, three kinds of D0₄ precipitates which satisfy the below orientation relationships were thought to equivalently exist in the D0₂₄ single-crystalline matrix phase.

\[
[11\bar{2}0]_{\text{D0}_4} / [100]_{\text{D0}_4} \quad (5)
\]

\[
[11\bar{2}0]_{\text{D0}_4} / [102]_{\text{D0}_4} \quad (6)
\]

\[
[11\bar{2}0]_{\text{D0}_4} / [10\bar{2}]_{\text{D0}_4} \quad (7)
\]

In the TEM observation, however, it was confirmed that all the D0₄ precipitates in the D0₂₄ single-crystalline matrix phase have the same orientation for each other, hence one of the above three relationships was only observed in a D0₂₄ single-crystalline matrix phase. This implies that all the D0₄ phases preferentially precipitate along a specific growth

![Fig. 4. Crystallographic relationship between the D0₂₄ matrix phase and the D0₄ precipitates observed in Alloy 3 sample.](image-url)
direction in Alloy 3. As described above, the preferential growth direction of Ni$_3$Nb in ternary DS composites was reported to be [100] [8]. In Alloy 3 sample, [100] direction in the D0$_x$ precipitates existed within 25° of the growth direction.

3.3. Compressive deformation behaviour in the duplex-phase alloys

Fig. 5 shows the temperature dependence of yield stress of the three DS alloys deformed along a growth direction in compression. Alloy 1 exhibits high yield stress at low temperatures. The stress keeps approximately a constant value at and below 700 °C, but it rapidly decreases above 700 °C. Similar temperature dependence of yield stress is observed in Alloy 2, although the value is somewhat lower than that of Alloy 1 at all tested temperatures, and the onset temperature of a rapid decrease in yield stress is also lower. Sheffler et al. [11] pointed out the rapid decrease in yield stress at high temperatures in the Ni$_3$Al–Ni$_3$Nb DS alloy is closely related to the softening of Ni$_3$Nb(D0$_x$) phase. The softening was confirmed using Ni$_3$Nb single crystals by the present authors [3]. When Ni$_3$Nb crystal was compressed along [100] direction, which is the growth direction in the Ni$_3$Al–Ni$_3$Nb DS alloy, [201] slip was operative under extremely high stress and showed anomalous strengthening behaviour with a maximum yield stress around 300 °C. At high temperatures above 500 °C, the yield stress rapidly decreased with increasing temperature accompanied by the operation of {011} twin. Although the Schmid factor for {011} twin was negligible at [100] orientation, this twin was operative owing to its low critical resolved shear stress (CRSS). Also, the strength of D0$_{24}$(Ni$_3$Ti) phase contained in Alloys 2 and 3 is considered to decrease rapidly above 600 °C, which was confirmed using binary Ni$_3$Ti single crystals [17].

Yield stress in Alloy 3 shows a different temperature dependence. The yield stress is considerably lower than those of other two alloys at RT. But it increases with rising temperature at intermediate temperatures (i.e. yield stress anomaly), reaching a peak at around 600 °C. The stress gradually decreases above 600 °C, and shows similar temperature dependence to those of other two alloys. At RT, more than 5% fracture strain was obtained in Alloy 3, while the sample was fractured at less than 1.5% plastic strain in Alloy 2. The low-temperature ductility was fairly improved in Alloy 3.

Since the D0$_{24}$ matrix phase in Alloy 3 had single-crystalline form in the compressive specimens (the loading axis was parallel to [1 3 7 2 7 1 3]), the operative slip systems could be determined by two-face slip trace analysis. Fig. 6 shows the slip markings in Alloy 3 samples deformed at various temperatures. At RT, slip traces are observed parallel to the colonies of D0$_x$ precipitates (Fig. 6(a)). This indicates the (0001) basal slip is operative in the D0$_{24}$ matrix phase. The slip traces in the D0$_{24}$ matrix phase propagate through the D0$_x$ precipitates, suggesting the slip transfer between the two phases easily occurs. Slip traces nearly perpendicular to the colonies are observed in addition to
the basal slip traces at 600 °C (Fig. 6(b)). Those slip traces were identified to be a slip on (11̅0) prism plane. Above 600 °C, the prism slip is dominantly observed as shown in Fig. 6(c) accompanied by a gradual decrease in yield stress. The propagation of slip traces on the prism slip plane is frequently suppressed at the phase boundary between precipitates, in contrast to the basal slip. The temperature for transition of operative slip system is in agreement with the peak temperature of anomalous strengthening. The results clearly indicate the yield stress anomaly and the gradual decrease in stress above the peak temperature were caused by the operation of basal slip and prism slip, respectively, in Alloy 3. Similar temperature dependence of yield stress was reported in binary Ni$_3$Ti(D0$_{22}$) [4,17] and in binary Ni$_3$Nb(D0$_0$) single crystals [3].

3.4. Slip transfer behaviour between the D0$_{24}$ and the D0$_{6}$ phases

It is well known that in composite materials, the slip transfer process at interfaces plays a significant role in the mechanical behaviour (for example, see Ref. [18]). Slip transfer behaviour in Ni$_3$Al–Ni$_3$Nb DS alloys have been discussed by several researchers [10–13]. Since a distinct orientation relationship exists between the D0$_{24}$ and the D0$_6$ phases in Alloy 3 on the basis of a similar atomic arrangement, the analogous slip transfer behaviour is expected to occur. It has been pointed out that one of the principal factors for the slip transfer behaviour between the two phases is the line of intersection between the incoming slip plane and the two-phase interface [18–20]. The incoming slip system at the interface may be accompanied by the activation of other outgoing slip systems in both the phases. In this process, the slip planes of outgoing systems are selected to have a common intersect line of phase boundary with incoming slip plane [18–20]. Basal slip was observed in both the phases at low temperatures. Since most of two-phase interfaces in Alloy 3 did not lie on a specific crystallographic plane as shown in Fig. 3(a), the directions of intersection lines were different depending on each interface geometry. However, the slip transfer between phases easily occurred at low temperatures from the basal slip in the D0$_{24}$ phase to basal slip in the D0$_6$ phase and vice versa. Since the basal planes in both the phases are aligned parallel in Alloy 3, the intersect line always overlaps independent of the interface geometry. But the difference in atomic configuration on the basal plane may influence the slip transfer behaviour. The slip direction on the (010) basal plane in the D0$_6$ phase is limited to one direction along [100] at low temperatures owing to the R-type configuration of minority atoms, while three (1120) slip directions exist in the D0$_{24}$ phase. Thus, the dominantly operative slip direction in the D0$_{24}$ phase may be limited to that parallel to [100] in the D0$_6$ phase in order to maintain the strain compatibility between the two phases, which may result in increase of flow stress. The R-type configuration on the basal plane in the D0$_6$ phase may also strongly affect the slip transfer behaviour for the prism slip. Both (001) and (201) planes in the D0$_6$ structure correspond to {11̅00} prism planes in the simple HCP structure. But the plastic behaviour and the temperature dependence of CRSS for (001) prism slip and (201) prism slip exhibited significant difference in Ni$_3$Nb(D0$_0$) crystals [3]. The CRSS for the (201) slip exhibited a much higher value than that for (001) slip. Therefore, the slip transfer may occur easily on the {11̅00} prism plane in the D0$_{24}$ phase which is parallel to (001) plane in the D0$_6$ phase, but it may be difficult on the other two {11̅00} planes. In fact, since the (11̅00) prism plane with the highest Schmid factor in the D0$_{24}$ matrix phase was not parallel to the (001) plane in the D0$_6$ precipitates in Alloy 3, the prism slip was suppressed at the interface between the precipitates and the matrix as shown in Fig. 6(c).

4. Conclusions

The microstructure and compressive flow stress of directionally solidified ternary Ni$_3$(Al, Nb) alloy (i.e. Ni–16.0 at.%Nb–9.0 at.%Al (Alloy 1)) and quaternary Ni$_3$(Al, Nb, Ti) alloys (i.e. Ni–13.3 at.%Nb–7.5 at.%Al–4.2 at.%Ti (Alloy 2) and Ni–10.7 at.%Nb–6.0 at.%Al–8.3 at.%Ti (Alloy 3)) were investigated, and the following conclusions were obtained.

1. Alloy 1 was composed of L1$_2$(Ni$_3$Al) and D0$_{24}$(Ni$_3$Nb) phases while Alloys 2 and 3 were composed of D0$_{24}$(Ni$_3$Ti) and D0$_6$(Ni$_3$Nb) phases.
2. The definite crystallographic relationship between the D0$_{24}$ and the D0$_6$ phases was observed to be (0001)$_{D0_{24}}$// (010)$_{D0_{6}}$, and (1120)$_{D0_{24}}$//(100)$_{D0_{6}}$ in Alloy 3.
3. Alloy 1 exhibited high yield stress at low temperatures, but it rapidly decreases above 700 °C. Similar temperature dependence of yield stress was observed in Alloy 2, although the onset temperature of a rapid decrease in yield stress was somewhat lower. Alloy 3 with the highest Ti content showed the lowest compressive strength along the growth direction among the three alloys, while relatively good low-temperature ductility was obtained in Alloy 3.
4. Yield stress of Alloy 3 exhibited anomalous strengthening behaviour accompanied by the operation of basal slip in both D0$_{24}$ and D0$_6$ phases. Transition in operative slip systems from the basal slip to the prism slip occurred at the peak temperature of yield stress anomaly (600 °C), resulting in a gradual decrease of yield stress.

Acknowledgements

This work was supported by a Grant-in-Aid from the Japanese Ministry of Education, Culture, Sports, Science and Technology. This work has partly been carried out at the Strategic Research Base ‘Handai Frontier Research
Center’ supported by the Japanese Government’s Special Coordination Fund for Promoting Science and Technology. One of the authors (K.H.) would like to thank the Japan Society for the Promotion of Science (JSPS) for a research fellowship.

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