Formation and morphology of A1/D022 two-phase microstructure have been investigated in Ni-V alloys in order to study the fundamentals for microstructure control of wrought alloys containing high volume fraction of tetragonal compound. In the hypo-stoichiometric composition of Ni3V, formation of "multi-variant structure (MVS)" consisting of D022 single phase takes place below $T_C$ temperature. By aging in the A1/D022 two-phase region, A1 phase is introduced into MVS via two different modes: precipitation with incoherent interfaces and continuous phase separation with coherent interfaces. The latter case is attractive because the interface between the two phases remains coherency after long-term aging at elevated temperatures, even though coarsening of D022 phase occurs. In the binary alloy, the morphology of coherent two-phase microstructure becomes "maze", where the D022 phase has plate in shape with irrational habit planes close to (304)$_{D022}$. The habit plane is parallel to one of the a-axes of D022 and an invariant line between the two phases, since the misfit strains along a- and c-axes of D022, $\delta_a$ and $\delta_c$, respectively, have the relationship of $\delta_a < 0 < \delta_c$. The calculation based on the lattice invariant theory as well as experimental results clearly demonstrate that the misfit strain ratio $\delta_a/\delta_c$ is a dominant factor to determine the habit plane and that the value of $\delta_a$ is the key factor for the shape of D022 phase. The reduction in $\delta_a$ can change the shape drastically from plate into prism elongated along an a-axis, leading to the formation of thermally stable "chessboard" microstructure in the two-phase alloys.

Introduction

The volume fraction of Ni$_3$(Al, Ti)-$\gamma'$ (L1$_2$) phase is one of the most important factors for high temperature strength in Ni base superalloys. The single-crystal superalloys usually contain more than 70 vol. % of $\gamma'$ phase by means of composition control and heat treatment. On the other hand, wrought Ni base alloys usually have the volume fraction less than 50 %, because wide window is required for the hot working process. In the case of Waspaloy, the volume fraction is known to be limited up to 50 % at the most. However, the limitation can be overcome by using Kurnakov type compounds as strengtheners. This type of compounds has a congruent disordering temperature $T_C$, above which the wide range of process window exists. In addition, control of microstructure as well as the volume fraction (up to 100 %) can be possible through cooling process. Therefore, obtaining the fundamental knowledge for microstructure control using Kurnakov type compounds opens new possibility for developing new wrought Ni base alloys, especially for steam turbines of USC (ultra-super critical) power plants operated at over 700°C. In this study, we select Ni$_3$V as a model case based on the following two reasons; one is the high $T_C$ (1318 K) at its stoichiometric composition [1, 2], and the other is crystal structure of tetragonal D022. This structure has two misfit strains against fcc (A1) matrix along a- and c-axes, while $\gamma'$ phase has only one. It is known that Ni$_3$V coherently precipitates in Ni-$\gamma$ matrix due to small lattice misfit strains [3], but no attempt has been made to control the morphology by misfit strains.

Recently, we examined the microstructure of Ni-25at%V quenched from high temperature A1 region [4, 5]. The high temperature A1 phase completely transforms to D022 phase during quench ($V_f$=100%), and exhibits a fine coherent brick-like "multi-variant structure (MVS)" consisting of three variants of D022 with c-axes perpendicular to each other. In the MVS, one of the three variants with quadrangular prism shape (major variant) of 90x90x150 nm has coherent interfaces of (101)$_{D022}$, (101)$_{D022}$ and (010)$_{D022}$, in fcc base index. The major variant particles are periodically arrayed with an interval of 30 nm, and the remaining two channels parallel to (101)$_{D022}$ and (010)$_{D022}$ are filled with two other variants (minor variants) with their c-axes parallel to the a-axis of the major variant. The formation of MVS would be attributed to the minimization of the anisotropic strain fields due to the tetragonality against A1 matrix with the following lattice parameter relationship: $a_{D022} < a_{A1} < c_{D022}/2$ [2, 3]. Since the morphology of MVS is just like $\gamma'/\gamma$ two-phase morphology in superalloys, if rotated 45° around [010] axis, it is attractive as high temperature material. However, this MVS is not stable at elevated temperatures, and drastically coarsens by coherency loss at interfaces [5, 6]. The collapse of MVS is caused by a release from elastic constraint, accompanied by the increase in the lattice parameter ratio c/a from nearly cubic value (2.006) to equilibrium value (2.04). Therefore, some modification to accommodate the elastic constraint is required to improve the thermal stability of MVS.

In this study, introduction of A1 phase into MVS has been attempted. First, we have focused on the hypo-stoichiometric composition of Ni$_3$V in the binary system, and identified the region to form MVS with A1 phase. Then, morphology control of the two-phase microstructure has been examined in terms of misfit strains through alloying.

Experimental Procedures

Four binary alloys with compositions of Ni-23V, Ni-20V, Ni-18V and Ni-17V (all compositions are given in atomic percent unless otherwise stated) were prepared in order to investigate the A1/D022 two-phase microstructure. Ternary alloys containing Co, Nb or Fe were also prepared so as to change the misfit strains between A1 and D022 phases against Ni-20V. Note that Co and Fe preferentially substitute Ni-sublattice while Nb substitutes V-sublattice in Ni$_3$V [7]. The five alloys of Ni-20V-10Co, Ni-15V-
5Nb, Ni-19.5V-2Fe, Ni-19V-4Fe and Ni-19V-6Fe, having 50-60 vol.% of D022 phase at 1073 K, were selected based on the reported binary and ternary diagrams [1, 2, 8-13]. All of these alloys were prepared by arc melting in argon atmosphere with a non-consumable tungsten electrode as 35 g button ingots, using 3N7 nickel pellets, 3N vanadium tablets, 3N cobalt lumps, 3N niobium grains and electrolytic iron. Each button ingot was melted five times by turning over each time in order to avoid segregation. These ingots were cut to pieces with a size of 6.0x6.0x10.0 mm^3 by an electro-discharged machine.

All samples were homogenized at 1573 or 1523 K (A1 single phase region) for 3.6~10.8 ks, followed by water quench. These treatments were conducted using a specially designed vertical furnace under argon atmosphere [14]. Aging treatments were employed in the A1+D022 two-phase region: 1273 K for Ni-23V and 1073 K for binary Ni-(17-20)V and ternary alloys. All samples for aging were sealed off in silica capsules under argon back-filled after evacuating to 8x10^-4 Pa. After holding the capsules for 0.9~8640 ks in the furnace, the samples were quenched into water quickly by breaking the capsules.

Microstructures were examined by transmission electron microscope (TEM). TEM discs with 0.15 mm in thickness and 3 mm in diameter were machined, then mechanically polished, followed by twin-jet electropolishing in a solution of ethanol with 12 vol. % perchloric acid at 253 K. The TEM observation was performed with an incident beam direction B=<100> so as to distinguish the three variants of D022. The planes and directions of D022 phase are indexed in the same way as A1 structure, although the unit cell of D022 structure is twice of that of A1 along its c-axis. The misfit strains between A1 and D022 phases were calculated using selected area diffraction patterns obtained by means of microbeam method. The detail procedures will be described later.

Results and Discussion

Formation of A1/D022 Two-phase Microstructure

Before investigating the morphology of two-phase microstructure, it is important to identify the region to form MVS in hypo-stoichiometric alloys. Figure 1 shows TEM dark field images (DFIs) of the binary alloys with D022 phase. The typical MVS in quenched Ni-25V from 1573 K exhibits the major variant particles in the hypo-stoichiometric compositions can further investigate. Figure 2 shows TEM images of Ni-23V and Ni-20V aged in A1+D022 two-phase region. After aging at 1273 K for 86.4 ks, Ni-23V exhibits coarse A1 phase with plate and particle shapes with the size of 100 ~ 500 nm in D022 matrix [Fig. 2(a)], and many dislocations are observed at the interfaces. On the other hand, Ni-20V aged at 1073 K /864 ks exhibits aligned D022 microstructure [Fig. 2(b)], where the bright and dark regions correspond to D022 and A1 phases, respectively. The shape of D022 plate is face-centered cubic with about 130 nm in thickness, and A1 phase also has plate shape with about 70 nm in thickness. No dislocation is observed at the interfaces. In both cases, the selected area diffraction pattern (SADP) reveals the existence of only one variant of D022 phase, indicating that the major variant particles in MVS grow at the expense of minor variants and disordered A1 phase forms during aging. However, the formation mode of A1 phase is completely different; in Ni-23V, A1 phase precipitates incoherently at variant interfaces, whereas it forms coherently in Ni-20V. Figure 3 shows the change in volume fraction of D022 phase during the aging for the two alloys, together with those of other binary alloys. In Ni-23V, the volume fraction gradually decreases from 100 to 65% with aging. In contrast, the quenched Ni-20V with a certain amount of A1 phase change to near single phase of D022 after short time aging (0.9 ks), and then the fraction starts decreasing to 65% with aging. The interfaces in the two-phase microstructure keep coherency even after prolonged aging, indicating that the A1 phase should be formed spinodally through continuous composition change. Unlike these two alloys, the volume fraction increases with aging in the alloys with V content less than 18 at%.

From above results, there are three formation modes of A1/D022 two-phase microstructure, depending on the alloy composition and heat treatment temperature:

1) D022→D022+A1 (precipitation of A1, Ni-23V)
2) D022→D022+A1 (phase separation, Ni-20V)
3) A1→D022+A1 (precipitation of D022, Ni-(17-18)V)

The difference in the mode can be interpreted by the thermodynamic approach proposed by Soffa and Laughlin [15]. Figure 4 (a) shows a part of Ni-V binary diagram, showing the region where each mode occurs, together with the schematic Gibbs energy – composition curves of A1 (G_A1) and D022 (G_D022) phases at 1073 K [Fig. 4(b)]. Note that the phase boundaries of A1/A1+D022 and A1+D022/D022 are coherent boundaries drawn based on the measured volume fraction, with an assumption that molar volume of A1 and D022 phases are nearly equal, and they are consistent with other reports [8, 9].

The region to form MVS in hypo-stoichiometric compositions can be specified below T_0 temperature, as shown by hatching in Fig. 4(a). In this region, A1 phase becomes energetically unstable against D022 phase since T_0 is a trace of composition where the Gibbs energy of A1 and D022 at given temperatures are equal. In quenching, the higher the T_0, the easier the formation of MVS consisting of D022 phase, because the driving force for the A1→D022 phase transformation becomes large enough. This is the case for the quenched Ni-23V. With decreasing V content, however, the formation of MVS becomes more difficult because of lower supersaturation of V as well as slow diffusion rate even though the driving force is still enough to form MVS. This is the case for the quenched Ni-20V. Thus, once the sample is heated again at temperatures just below T_0, the ceased transformation
could proceed again, resulting in MVS.

The different formation modes of the two-phase mixture (1) and (2) by aging can be explained by introducing the miscibility gap in D0\textsubscript{22} phase. Then, the region showing MVS in the two-phase region in Fig. 4(a) can be divided further into two regions A and B sectioned by T\textsubscript{S} line, where T\textsubscript{S} is an inflection point (x) on the G\textsubscript{D022} curve (d\textsuperscript{2}G/dx\textsuperscript{2}=0) in Fig. 4(b). The region A is inside the T\textsubscript{S} point as well as the T\textsubscript{0} point, where the G\textsubscript{D022} curve has a positive curvature (d\textsuperscript{2}G/dx\textsuperscript{2}>0), so that the D0\textsubscript{22} phase is energetically metastable against two-phase mixture as shown by the common tangent. This leads to a nucleation and growth of disordered A1 phase with a fixed composition marked by open circle on G\textsubscript{A1} curve to reduce the chemical energy in the initial MVS state. The region B, on the other hand, is inside the T\textsubscript{0} point but outside of the T\textsubscript{S} point, where the G\textsubscript{D022} has a negative curvature (d\textsuperscript{2}G/dx\textsuperscript{2}<0), so that the thermodynamically unstable D0\textsubscript{22} phase continuously decomposes to V-lean and V-rich ordered phases first, followed by phase separation to the stable A1.

Figure 1. TEM DFIs of Ni-V binary alloys with D0\textsubscript{22} phase taken with g=002\textsubscript{D022} (B=010\textsubscript{A1}): (a, b) typical MVS in the quenched Ni-25V from 1573 K showing (a) major and (b) minor variants, (c) MVS in the quenched Ni-23V from 1573 K showing major variant, (d) MVS in Ni-20V obtained after aging at 1073 K/0.9 ks showing major variant, (e) D0\textsubscript{22} particles precipitated in Ni-18V aged at 1073 K/3.6 ks. (f) Schematic illustration of MVS, together with D0\textsubscript{22} structure.
and D022 phases. The former is the case for Ni-23V aged at 1273 K whereas the latter is for Ni-20V aged at 1073 K. The existence or absence of interfacial dislocations (Fig. 2) is one of the strong evidence to support these formation modes of A1 phase in D022 matrix. The other mode (3) in Ni-18V and Ni-17V is, just like opposite case of mode (1), where the alloy compositions are initially outside of the T0 point on the positive curvature of GA1, and the disordered single phase state is energetically unstable with respect to the stable two-phase mixture, resulting in precipitation of D022 phase in Al matrix.

**Morphology of Coherent A1/D022 Two-phase Microstructure**

The two-phase mixture in the region B is attractive because of the high volume fraction of D022 phase as well as the coherency of the interface even after long-term aging at elevated temperatures. However, the initial MVS is somehow coarsened, so that further improvement of thermal stability as well as morphology control of the two-phase microstructure are key issues for alloy development. In this section, we focus on the microstructure evolution of Ni-20V formed in the region B and identify the fundamental factors governing the microstructure morphology in terms of the misfit
In order to identify the relationship between the misfit strains of the D022 phase and aging. The A1/D022 habit plane is quantified as an angle of 45° in MVS, and it gradually decreases to 35°. (1) and (2) crystallographically equivalent {304} D022 in the unit cell of D022, another plates along <100> D022 were also confirmed to be parallel to {304} D022, which are not edge-on [5]. Since there are four crystallographically equivalent {304} D022 in the unit cell of D022, the morphology of this microstructure can be hereafter called as “maze”. 

The change in morphology from the initial MVS to maze microstructure is characterized by the following two points; (1) the change in shape of D022 from a quadrangular-prism into a plate by merging into each other, (2) the inclination of the habit plane from (101) D022 to (304) D022. Figure 5 shows change in the A1/D022 habit plane with aging. The habit plane is quantified as an angle θ between the edge-on habit plane and (001) D022. The line connecting two intersection points is the invariant line. Based on the fact that all of the observed habit planes are parallel to one of the 𝑎-axes, the plane consisting of an invariant line and an 𝑎-axis would be the habit. Therefore, two-dimensional treatment can be applied. The habit plane angle θ corresponds to the angle between the invariant line and [100] D022 (a-axis). Thus, the θ = tan⁻¹(y/x) can be geometrically calculated using the following circle and ellipse equations;

\[
\begin{align*}
\frac{x^2}{(1+\delta_a)^2} + \frac{y^2}{(1+\delta_c)^2} &= 1 \quad \text{(D022 lattice)} \\
\frac{x^2}{(1+\delta_a)^2} + \frac{y^2}{(1+\delta_c)^2} &= 1 \quad \text{(A1 lattice)}
\end{align*}
\]

where x and y are the coordinate of the intersection point. Then, the θ can be simply expressed as functions of the misfit strains;

\[
\theta = \tan^{-1} \left( \frac{-(1 + 2\delta_a^{-1})(\delta_c^{-1} + \delta_a^{-1})^2}{\delta_c^{-1} (\delta_a^{-1} + 2\delta_a^{-1})(1 + \delta_a^{-1})^2} \right)
\]

This equation indicates that θ depends on both the misfit strain of δa and the ratio of misfit strains δc/δa. Figure 6(b) shows a plot of calculated θ as a function of misfit strain ratio using the above equation at two fixed values of δc: one (thick line) is the case of δc = -0.003, and the other (thin line) is δc = -0.030. In both cases, the angle θ apparently approaches to 45° as the misfit ratio approaches to -1, and the deviation of θ between the two calculated lines is as much as 2°, even though the δc is changed by an order of magnitude. This result indicates that the habit plane strongly depends on the misfit strain ratio. In order to verify the prediction, the misfit strains are measured in the sample of Ni-20V aged at 1073 K /864 ks. Since all fundamental reflections of D022 structure coincide with those of A1 structure, it is impossible to measure misfit strains between these phases using conventional diffraction patterns. Then, microbeam method was applied in this study. The diameter of microbeam is small enough (10~40 nm) to obtain a diffraction pattern from each phase region. Figure 7 shows TEM DFIs [Fig. 7(a)] and the microbeam diffraction patterns of A1 [Fig. 7(b)] and D022 [Fig. 7(c)] phases taken from the neighboring regions indicated by white circles in Fig. 7(a). Since the lattice parameters are inversely proportional to the distance of two reflections, δa and δc can be expressed as follows, using 𝑟A1, 𝑟a, and 𝑟c, which are indicated in Fig. 7(b, c):

\[
\begin{align*}
\delta_a &= \frac{a_{D022}}{a_{A1}} - 1 = \left( \frac{r_{A1}}{r_a} \right) - 1 \\
\delta_c &= \frac{c_{D022}}{a_{A1}} - 1 = \left( \frac{r_{A1}}{r_c} \right) - 1
\end{align*}
\]

The obtained misfit strains are shown in Table I, together with the observed and calculated angles of habit plane. The values of δc and δa are about -0.003 and +0.006, respectively, and the misfit strain ratio is about -2. Then, θ is calculated as 34.5°. This value agrees well with the observed value of 36.5°. Thus, the inclination of habit plane with aging is associated with the continuous change in misfit strains due to the phase separation.

The Shape of D022 Phase. The change in the shape of D022 phase during aging is related to the growth directions of initial particle. The growth along two directions, an 𝑎-axis and the invariant line,
is necessary to form plate shape observed in Ni-20V. In other words, the plate shape is the case that the growth rate along \(a\)-axis \((v_a)\) and the invariant line \((v_i)\) are on the same level. Based on the fact that the direction with smaller misfit becomes preferred direction to grow, as is clear in case of Ni-Ti phase \([17]\), \(v_a\) depends on the value of \(|\delta_c/a|\). Thus, the value of \(|\delta_c/a|\) should be the key factor for the shape of \(D_{022}\). Based on this prediction, reduction in \(|\delta_c/a|\) would be effective in improving the coarsened morphology of \(D_{022}\) by suppressing the growth along the invariant line, since \(v_c\) becomes larger than \(v_i\) \((v_c>v_i)\).

**Microstructure Control of A1/D022 Two-phase Alloys**

In the previous section, \(\delta_c/\delta_a\) and \(|\delta_c/a|\) are found to be the key factors for the habit plane and the shape of \(D_{022}\), respectively. Based on the findings, an attempt has been made to control the two-phase microstructure by changing the misfit strains through alloying.

Table II shows effect of third element addition to Ni-25V on the lattice parameters of A1 and \(D_{022}\) phases in alloys which become single phase of A1 and \(D_{022}\) at 1473 K and 1273 K, respectively \([7]\). From these lattice parameters, misfit strains between the two phases can be calculated. In the stoichiometric Ni-25V, \(\delta_c\) and \(\delta_a\) are \(-0.0054\) and \(+0.0130\), respectively. Additions of Co (Ni site) and Nb (V site) decrease \(|\delta_c/a|\) and the effect of Nb is more obvious than that of Co. However, \(\delta_c\) decreases by Co addition, whereas it increases by Nb addition. On the other hand, addition of Fe to Ni site increases \(|\delta_c/a|\), while \(\delta_c\) remains almost unchanged. Although these misfit strains cannot be directly applied to the microstructure consisting of two phases with different

<table>
<thead>
<tr>
<th>Lattice misfit strain</th>
<th>(\theta / \text{degree (observed)})</th>
<th>(\theta / \text{degree (calculated)})</th>
</tr>
</thead>
<tbody>
<tr>
<td>(-0.0027)</td>
<td>0.0058</td>
<td>36.5</td>
</tr>
</tbody>
</table>
compositions, these trends can be referred for predicting the effects of the third element on the A1/D0_{22} two-phase microstructure. In the early stage of continuous phase separation (D0_{22}+D0_{22}+A1), these trends would be helpful, since the compositions of the two phases are very close to each other. However, after prolonged aging, partition coefficients of Ni and M between the two phases have to be taken into consideration, since the lattice parameters also depend on Ni content [2, 3]. According to Table II, it is expected that addition of Co and Nb are effective in suppressing the coarsening of D0_{22} along the invariant line. On the other hand, Fe addition is expected to maintain the shape unchanged.

Figure 8 shows TEM images of two-phase microstructure in ternary alloys aged at 1073 K for 864 ks. In the alloy with 10% Co [Fig. 8(a)], the microstructure observed from B=010_{A1} consists of two variants of D0_{22} (dark) with parallelogram shape and A1 phase (bright) with rhombic shape aligned alternately, exhibiting so-called "chessboard" microstructure. Since all of the observed habit planes in this figure are edge-on, the morphology of both phases are prism shape elongated along a-axis. The habit plane is close to \{304\}_D0_{22}. In case of 5% Nb addition, however, "chessboard-like" microstructure is obtained as shown in Fig. 8(b). The morphology of D0_{22} phase is recognized to be prism elongated along a-axis, but unlike the Co added alloy, the cross sectional shape is somehow irregular parallelogram and the habit plane is much closer to \{001\}_D0_{22}. In contrast, Fe added alloys remain the "maze" microstructure [Fig. 8(c)], regardless of Fe content [5].

**Table II. Effect of third element M (M: Co, Nb, Fe) addition to Ni-25V on the lattice parameters of A1 and D0_{22} phases and misfit strains between the two phases with the same composition**

| Alloy composition / at% | Lattice Parameter /Å | Misfit strain |  |
|------------------------|-----------------------|---------------|
|                         | A1^{1)                        | D0_{22}^{2)    | \(\delta_a\) | \(\delta_c\) |
| Ni-25V                  | 3.562 3.543 3.608          | -0.0054 0.0130 |
| Ni-25V-7Co              | 3.564 3.549 3.604          | -0.0042 0.0111 |
| Ni-23V-2Nb              | 3.567 3.554 3.618          | -0.0038 0.0141 |
| Ni-25V-3Fe              | 3.564 3.544 3.611          | -0.0058 0.0129 |

^{1) Water quenched from 1473 K, ^{2) Water quenched from 1273 K

![Figure 8. TEM images of A1/D0_{22} two-phase microstructure in Ni-V-M (M: Co, Nb, Fe) ternary alloys aged at 1073 K for 864 ks taken with B=100_{A1}; (a) Ni-20V-10Co (BFI), (b) Ni-15V-5Nb (BFI) and (c) Ni-19V-4Fe (DFI).](image-url)
Table III. Misfit strains measured by microbeam method in the binary and the ternary alloys with Co, Fe and Nb aged at 1073 K/864 ks, together with the morphology (habit plane angle and shape) of D0$_{22}$ phase

<table>
<thead>
<tr>
<th>Alloy composition / at%</th>
<th>Lattice misfit strain</th>
<th>θ / degree (observed)</th>
<th>Shape of D0$_{22}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-20V</td>
<td>-0.0027</td>
<td>0.0058</td>
<td>-2.14</td>
</tr>
<tr>
<td>Ni-20V-7Co</td>
<td>-0.0020</td>
<td>0.0039</td>
<td>-1.97</td>
</tr>
<tr>
<td>Ni-15V-5Nb</td>
<td>-0.0015</td>
<td>0.0110</td>
<td>-7.36</td>
</tr>
<tr>
<td>Ni-19.5V-2Fe</td>
<td>-0.0031</td>
<td>0.0069</td>
<td>-2.20</td>
</tr>
<tr>
<td>Ni-19V-4Fe</td>
<td>-0.0060</td>
<td>0.0077</td>
<td>-1.29</td>
</tr>
<tr>
<td>Ni-19V-6Fe</td>
<td>-0.0061</td>
<td>0.0067</td>
<td>-1.10</td>
</tr>
</tbody>
</table>

Table III shows measured misfit strains, together with observed habit plane angle and the shape of D0$_{22}$. In all alloys, δ$_a$ and δ$_c$ are negative and positive, respectively, and |δ$_a$| is larger than |δ$_c$|. By comparing the value of δ$_c$, it is clear that the shape of D0$_{22}$ phase is plate in alloys with |δ$_a$|>0.0027 (Ni-20V and Fe added alloys) and prism in alloys with |δ$_a$|<0.0020 (Co and Nb added alloys). This result agrees well with the prediction, and there is no dependency on δ$_a$, although δ$_c$ is small in Co added alloy (+0.0039) while it is much larger in Nb added alloy (+0.0111). Thus, the value of δ$_a$ is a key factor for control of the morphology, and the threshold exists at around |δ$_a$|=0.002.

Figure 9 shows change in the habit plane angle [Fig. 9(a)] and misfit strain ratio [Fig. 9(b)] as a function of M content (M: Co, Fe). In Ni-20V, the value of θ is 36.5°. Addition of Co has little effect on the change in the habit plane and the angle θ is 37.7° even by 10% addition. On the other hand, the angle θ is sensitive to Fe content. It increases linearly toward 45°, which is {110}$_{D0_{22}}$ habit plane, with increase in Fe content. In the mean time, misfit strain ratio increases from -2 to -1 by Fe addition, although it remains almost unchanged by Co addition. Thus, it is clear that the habit plane can be controlled by misfit ratio and becomes {101}$_{D0_{22}}$ when the ratio becomes -1, as predicted in Fig. 6. This is also consistent with the result of Nb added alloy, which exhibits the habit planes much closer to {001}$_{D0_{22}}$ and has large misfit strain ratio of -7.

All of these above results are the cases that the invariant line exists between the two phases due to δ$_a$<0<δ$_c$. Figure 10 shows whole picture of the morphology map of D0$_{22}$ phase as functions of δ$_a$ and δ$_c$, together with the plots obtained in this study. In the region of δ$_c$<0<δ$_a$ and |δ$_a$|>|δ$_c$|, it is clear that the value of δ$_a$ is a factor determining the microstructure morphology, either maze or chessboard microstructure, regardless of δ$_c$. On the other hand, in the region without invariant line, the relative relationship between the misfit strains, δ$_c$<δ$_a$ or δ$_c$>δ$_a$, is important, because the morphology of D0$_{22}$ phase becomes elongated along directions with minimum misfit strain. For example, the morphology of Ni$_3$Nb-$\gamma'$ phase, which is a metastable D0$_{22}$ phase in wrought Ni-base superalloys such as Inconel 718, is disc shape with habit plane of {001}$_{D0_{22}}$, since δ$_a$ and δ$_c$ are +0.009 and +0.037, respectively [18]. In the case of δ$_c$>δ$_a$, the morphology should become a rod shape elongated along c-axis. In addition, in the case of metastable (Cu, Ni)$_3$Sn phase, the misfit strains δ$_a$ and δ$_c$ were reported to be +0.038 and -0.003, respectively [19]. These values imply the existence of invariant line, but observed morphology is rod shape elongated along c-axis. This is reasonable because of |δ$_a$|>|δ$_c$|, so that the θ becomes close to 90°, leading to the preferred growth along the c-axis.

Finally, from the viewpoint of thermal stability of MVS, chessboard microstructure is the most attractive, since the coarsening of D0$_{22}$ phase takes place only along an a-axis. Thus, control of misfit strains to be |δ$_a$|<0.002 and δ$_a$/δ$_c$=-1 is effective in preventing drastic morphology change of D0$_{22}$ phase at elevated temperatures.

**Conclusion**

1) In the hypo-stoichiometric composition, MVS of D0$_{22}$ phase is formed below T$_g$ temperature. By aging in the A1/D0$_{22}$ two-phase
region, A1 phase is introduced into MVS by two different modes; precipitation with incoherent interfaces (above $T_S$) and continuous phase separation (below $T_S$). The latter case is attractive because interface between the two phases remains coherency after long-term aging.

2) In the coherent A1/D0$_{22}$ two-phase microstructure, the plane parallel to an $a$-axis of D0$_{22}$ and the invariant line between the two phases becomes the habit. The shape of D0$_{22}$ phase becomes either plate or prism, depending on the relative growth rate along an $a$-axis and the invariant line.

3) Control of the morphology of D0$_{22}$ phase is possible by changing misfit strains ($\delta_c<0<\delta_a$). The misfit strain ratio $\delta_a/\delta_c$ is the dominant factor to determine the habit plane, and the habit plane becomes {101}$_{D0_{22}}$ when the ratio becomes close to -1. On the other hand, the value of $|\delta_a|$ can change the shape of D0$_{22}$ phase from plate to prism when $|\delta_a|$ becomes smaller than 0.002, leading to the change in the microstructure morphology from maze to chessboard.

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