Analytical Electron Microscopy Study of γ/γ' Phase Equilibria in Ni-Al-Ta Alloys

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Phase equilibria between γ and γ' in Ni-base Ni-Al-Ta alloys have been investigated by analytical electron microscopy (AEM). The chemical compositions of the γ and γ' phases in the two-phase alloys were determined by an energy dispersive X-ray spectrometer (EDS) and then tie lines in the $\gamma + \gamma'$ two-phase region and the partitioning of Ta in the γ and γ' phases were evaluated. The ALCHEMI (atom location by channeling enhanced microanalysis) technique revealed that Ta occupies most favorably the Al sites in the γ' phases irrespective of stoichiometry. The lattice constants of the γ and γ' phases were determined by convergent beam electron diffraction (CBED). The lattice misfits between the γ and γ' phases were then calculated from the lattice constants and were compared with those determined from the analysis of the misfit dislocations.

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I. Introduction

Additions of refractory elements such as tantalum, hafnium, molybdenum, tungsten, etc. are known to be effective to improve the high temperature strength of the superalloys⁽¹⁾. Recent nickel-base superalloy usually contains large amounts of these refractory elements. Among these, tantalum confers both high levels of creep strength and oxidation resistance known as "Ta effects"⁽²⁾⁽³⁾. Tantalum also reduces significantly the casting defects referred to as freckling⁽⁴⁾.

The recent single crystal nickel-base superalloys, for example, alloy 454, contain no intentionally added grain boundary strengthening elements such as boron, carbon, hafnium and zirconium⁽²⁾ but a large amount of tantalum up to 12 mass%. Therefore the chemical composition of alloy 454 is very different from that of existing advanced superalloys. The compositions of single crystal superalloys are simple; single crystal alloy 454 contains only six alloying elements but directionally solidified Mar-M200 contains ten. Therefore the Ni-Al-X (X=refractory element) ternary alloy phase diagrams have attracted increasing attention. Although various studies have been performed on the effects of Ta addition to the existing advanced nickel-base superalloys⁽³⁾⁻⁽⁶⁾, systematic investigations of the behavior of tantalum in

simple Ni-Al-Ta ternary-alloys have not been reported.

The purpose of this paper is to measure the parameters determining the phase equilibria between the γ and γ' phases in a Ni-Al-Ta model superalloy system by analytical electron microscopy (AEM). Until the AEM was developed for the application to materials science, it was not possible to measure, with a single specimen and with a single equipment, various parameters such as the γ/γ' misfits, lattice constants, chemical compositions of various phases, partitioning coefficients, particle sizes and distribution, and site occupancy of alloying elements, which determine the strength of the superalloys through the phase stability, solid solution strengthening and precipitation strengthening.

II. Experimental

Table 1 lists the specimens used in this study, which includes chemical compositions, heat treatments, phase states and objects of the specimens used.

All the specimens were prepared from high purity Ni (99.9%), Al (99.99%) and Ta (99.99%). Except pure Ni (specimen A), totals of approximately 30 g were weighed and arc-melted in an argon atmosphere on a water-cooled copper hearth with a non-consumable tungsten electrode. To avoid segregation of alloying elements, the ingots were reversed and re-melted at least five times. Since the weight losses were less than 0.05%, the nominal compositions can represent the specimen compositions which are given in Table 1. Each ingot was encapsulated in a quartz tube under an atmosphere of argon and subjected to homogenization and equilibration under the

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Table 1 Compositions, heat treatments, phase states and objects of specimens used in this study.

Specimen	Composition (mol%)			Heat treatment		Phase state	Objects
	Al	Ta	Ni	Homogenization	Equilibration		
A	0	0	100	1073 K, 3 h	—	γ	CBED*
B	7.2	10.0	82.8	1563 K, 1 w	1273 K, 1 w	$\gamma + \gamma'$	EDS, CBED, MS
C	9.2	7.9	82.9	1563 K, 1 w	1273 K, 1 w	$\gamma + \gamma'$	EDS, CBED, MS
D	14.4	3.6	82.0	1563 K, 1 w	1273 K, 1 w	$\gamma + \gamma'$	EDS, CBED, MS
E	16.0	5.0	79.0	—	1273 K, 2 w	$\gamma + \gamma'$	ALCHEMI
F	21.0	4.0	75.0	1563 K, 1 w	1273 K, 1 w	γ'	ALCHEMI
G	23.0	4.0	73.0	—	1273 K, 2 w	$\gamma' + \beta$	ALCHEMI
H	23.0	2.0	75.0	1563 K, 1 w	1273 K, 1 w	γ'	EDS**
I	25.0	0	75.0	1523 K, 1 w	—	γ'	CBED*

CBED: convergent beam electron diffraction for the determination of lattice constant

EDS: energy dispersive X-ray spectrometry for quantitative microanalysis

MS: microstructure observation

ALCHEMI: atom location by channeling enhanced microanalysis for the determination of site occupancy

*for effective voltage determination

**for k -factor determination

conditions given in Table 1. Both heat treatments were always terminated after quenching in ice-water. The ingots were cut, sliced and ground mechanically to discs with a final size of 3 mm diameter and 0.15 mm thickness. For specimen A, a sheet with a thickness of 0.15 mm was obtained by cold-rolling. All discs were then perforated by a twin-jet electron-polishing technique in a solution of $\text{H}_2\text{SO}_4(10\%) + \text{CH}_3\text{OH}(20\%) + \text{C}_2\text{H}_5\text{OH}(70\%)$.

A JEM-2000FX AEM was used in this study at an accelerating voltage of 200 kV. Electron beams were focused to sizes of 30–100 nm for all cases expect microstructure observations. Characteristic X-rays used for the EDS analyses were the $\text{NiK}\alpha$, $\text{AlK}\alpha$, $\text{TaL}\alpha$ and $\text{TaM}\alpha$, lines. These lines were acquired at a take-off angle of 70 degrees by the detector with a Li-drifted Si crystal and a beryllium-type window. Integration of the characteristic line peak and background subtraction were made by a TN-2000 multichannel analyser to obtain the net intensities. Thin specimens were mounted on a double-tilt and low-background beryllium holder. Hole counts were negligible for any characteristic line.

III. Results

1. Misfit dislocations

The thermal stability of microstructure of superalloys depends strongly on the misfit between the γ and γ' phases. Figure 1 is a microstructure of specimen C. The micrograph was taken under a two-beam bright-field condition with $g = 11\bar{1}$. Dislocation networks were observed at the interface between γ and γ' phases, which indicates that the thermal stability of simple ternary alloys should not be high. Figure 2 shows a dislocation network taken at six different $\mathbf{g}$ vectors. Using the criterion of $\mathbf{g} \cdot \mathbf{b} = 0$, the Burgers vectors of dislocation segments in the network were determined. A schematic illustration of the network is shown in Fig. 3 with possible Burgers vectors

identified for the segments. All the dislocation segments were found to be pure edge in nature. Similar observations have been reported by other investigators in Ni-base superalloys^{(7)–(9)}.

It is possible to estimate a γ/γ' lattice misfit (δ) from the dislocation spacing (d) and the magnitude of Burgers vector (b) using the equation⁽¹⁰⁾,

$$\delta = b/d. \quad (1)$$

In this study, we measured d from the distance between the dislocation segments marked E or F in Fig. 3. The micrographs taken near the $\langle 001 \rangle$ zone axis as shown in Fig. 2(d) and (e) were used to avoid the inclination of network. An average of 117 nm was obtained for d . Substitution of this average and $b = a/\sqrt{2}$ led to $\delta = 0.22\%$, where we used $a = 0.3594$ nm obtained from the CBED analysis of this study. It should be noted that the sign of the misfit cannot be determined only from the analysis of the spacing of interface dislocations, since the absolute values of lattice parameter could not be determined from the above-mentioned measurements.

Fig. 1 A microstructure of specimen C taken under a two-beam bright-field condition with $g = 11\bar{1}$.

Fig. 2 TEM micrographs of a misfit dislocation network taken at six different g vectors. Beam directions are (a) $[112]$, (b) $[011]$, (c) $[011]$, (d) $[001]$, (e) $[001]$ and (f) $[101]$.

Dislocation	Direction of Burgers vector	Line direction
A	$[101]$	$[010]$
B	$[011]$	$[100]$
C	$[101]$	$[010]$
D	$[011]$	$[100]$
E	$[110]$	$[110]$
F	$[110]$	$[110]$

Fig. 3 A schematic illustration of the dislocation network deduced from Fig. 2. Table shows the Burgers vectors and line directions of the segments labeled in the illustration.

2. Lattice constants of γ and γ'

The absolute values of lattice constants of the γ and γ' phases in specimens B, C and D were determined by

analyzing the high order Laue zone (HOLZ) line patterns in the transmitted beam of convergent beam electron diffraction (CBED)⁽¹¹⁾⁽¹²⁾ and comparing with those of simulated HOLZ line patterns for various lattice constants.

In this determination, however, it is necessary to pay careful attention to the dynamical effect since this effect causes an appreciable change in HOLZ line pattern⁽¹³⁾⁽¹⁴⁾. To minimize the effect, we determined the effective voltages from specimen A (pure Ni) and specimen I (Ni_3Al) as in other studies⁽¹⁵⁾⁽¹⁶⁾ by the simulation of HOLZ line patterns at various voltages. In this simulation, the lattice constant of pure Ni (0.3524 nm) and that of Ni_3Al (0.3567 nm) were taken from the literature⁽¹⁷⁾. The effective voltage from specimen A is then used for the lattice constant determination of the γ phase and that from specimen I is for the γ' phase.

Figure 4(a) and (b) show the HOLZ line patterns taken from the $\langle 114 \rangle$ zone axis orientation of specimens A and I, respectively. Figure 4(c) and (d) are the simulated HOLZ line patterns. To avoid arbitrariness involved in the comparison process, p/q values in the simulated patterns are plotted against the voltage as shown in Fig. 5. Other segments than p and q for the ratios were turned

Fig. 4 HOLZ line patterns taken from the $[114]$ zone axis of (a) Ni and (b) Ni_3Al , and the patterns simulated for (c) Ni and (d) Ni_3Al .

Fig. 5 Relationships between the ratio p/q and accelerating voltage for Ni and Ni_3Al .

out to be unsuitable because some ratios were almost invariant to voltage change and other ratios were too sensitive to voltage change. The effective voltages are then determined from the ratios measured in the observed HOLZ line patterns through these relationships. The best

matching was attained at the effective voltages of 199.1 ± 0.5 and 200.2 ± 0.5 kV for specimens A and I, respectively.

Figure 6(a), (b) and (c) are the HOLZ line patterns of the γ phases in specimens B, C and D, respectively, and Fig. 6(d), (e) and (f) are those of the γ' phases in the corresponding specimens. All patterns were taken from the $\langle 114 \rangle$ zone axis orientation. The lattice constants were determined by using the p/q -lattice constant relationships given in Fig. 7 obtained by the simulation in which the lattice constant was changed.

Figure 8 shows the variation of lattice constant of the γ and γ' phases against the concentrations of Ta, C_{Ta} , in each phase obtained from the microanalyses which will be described in Section III.3. The dotted lines connecting the solid lines indicate the two phases in equilibria. It is seen that the lattice constant almost linearly increases with an increase of C_{Ta} for both the γ and γ' phases within the indicated range of C_{Ta} . It is also seen that the lattice constants of the γ' phases are invariably greater than those of the γ phases.

The lattice misfit (δ) was calculated from the following equation:

Fig. 6 HOLZ line patterns taken from the $[114]$ zone axis for (a)~(c) γ' phase and (d)~(f) γ phase, and for (a) and (d) specimen B, (b) and (e) specimen C, and (c) and (f) specimen D.

Fig. 7 Relationships between the ratio p/q and the lattice constant for the γ and γ' phases. The effective voltages are 199.1 kV for the γ phase and 200.2 kV for the γ' phase.

Fig. 8 A plot of the lattice constants of γ phase a_{γ} and γ' phase $a_{\gamma'}$ against Ta concentration C_{Ta} . The nickel contents are kept almost constant.

$$\delta = 2\{a_{\gamma'} - a_{\gamma}\} / \{a_{\gamma'} + a_{\gamma}\} \quad (2)$$

where a_{γ} and $a_{\gamma'}$ are the lattice constants of the γ and γ' phases, respectively. The values of δ are listed in Table 2. It is found that δ increases with an increase of C_{Ta} . For comparison, Table 2 includes also the values of δ determined from the spacing of interface dislocations in Section III.1 and from X-ray diffraction analysis⁽¹⁸⁾. The

difference in δ will be discussed in Section IV.

3. Chemical compositions of γ and γ'

A quantitative microanalysis of chemical compositions of the γ and γ' phases were performed by using the ratio technique⁽¹⁹⁾. The k -factors were determined by measuring the characteristic X-ray intensities from the standard specimen H.

Table 2 Lattice misfits (%) determined by CBED, interface dislocations and X-ray diffraction analysis.

Specimen	CBED	Interface dislocations	X-ray diffraction
B	+0.42	—	+0.39
C	+0.25	0.22	+0.64
D	+0.17	—	+0.84

Fig. 9 Tie lines between γ and γ' phases determined by the EDS analysis. The results of Willemin *et al.*⁽²⁶⁾, Hong *et al.*⁽²⁵⁾, Nash and West⁽²³⁾ and Jia *et al.*⁽²⁴⁾ are also included.

Since the absorption correction is essential in the nickel-base Ni–Al–Ta system, the extrapolation method⁽²⁰⁾⁽²¹⁾ was used in both k -factor determination and compositional determination of γ and γ' phases. In this study, at least 7 measurements were made at different thicknesses to achieve extrapolation for each series of k -factor and compositional determinations. The effect of X-ray fluorescence is negligible in this system⁽²²⁾.

The measured chemical compositions of the γ and γ' phases in specimens B, C and D and the resultant γ/γ' tie lines are plotted in the nickel corner of the Ni–Al–Ta phase diagram reported by Nash and West⁽²³⁾ as shown in Fig. 9 which includes also the compositions of specimens B, C and D. The results of Jia *et al.*⁽²⁴⁾ obtained from a diffusion-couple study using EPMA and γ' solvus reported by Hong *et al.*⁽²⁵⁾ using the DTA and that estimated by Willemin *et al.*⁽²⁶⁾ basing upon the EPMA measurements are also included for comparison. It is seen that the $\gamma + \gamma'$ two-phase region apparently extends to the nickel-rich side in the Ni–Al–Ta ternary phase diagram as compared to that of Nash and West⁽²³⁾. The γ' solvus determined by the present study for 1273 K agrees fairly well with those of Hong *et al.*⁽²⁵⁾ and Willemin *et al.*⁽²⁶⁾. The $\gamma + \gamma'$ region extends to the nickel-rich side but a little lesser extent even at 1523 K⁽²⁶⁾.

From Fig. 9, it is possible to examine a partitioning behavior of Ta into the γ and γ' phases at 1273 K. The partitioning coefficient κ is defined as $\kappa = C_{Ta}^{\gamma} / C_{Ta}^{\gamma'}$, where C_{Ta}^{γ} and $C_{Ta}^{\gamma'}$ are the concentrations of Ta in the γ' and γ phases, respectively. Then, κ is plotted in Fig. 10 as a function of C_{Ta}^{γ} . The values of κ obtained by Jia *et al.*⁽²⁴⁾ for 1273 K using a diffusion couple/EPMA method

Fig. 10 A plot of partitioning coefficient (κ) against the concentration of Ta in γ phase (C_{Ta}^{γ}). The results of Jia *et al.*⁽²⁴⁾ and Willemin *et al.*⁽²⁶⁾ are also plotted.

and those obtained by Willemin *et al.*⁽²⁴⁾ for 1523 K in terms of ordinal EPMA are also plotted in Fig. 10. It is found that all values lie on a single line for each temperature and κ decreases to ~ 1 as C_{Ta}^{γ} increases. This result indicates that Ta is more likely to be dissolved in the γ' phase than the γ phase when it is less contained in the alloy but, as the Ta content increases, it tends to be dissolved equally in both phases. It must be also noted that the AEM-EDS method can determine the partitioning coefficient in a wide range of temperature as compared to the EPMA method because of its high spatial resolution.

4. Determination of site occupancy

Since the electronic structures of Ni and Al are significantly different, it must be important to know the lattice sites into which the alloying elements enter. The ALCHEMI technique⁽²⁷⁾⁽²⁸⁾ was used to determine the site occupancy of Ta in the γ' phases. The γ' phases with three different nickel stoichiometry, specimen F (stoichiometric), specimen E (nickel-rich) and specimen G (aluminum-rich), were prepared for this purpose. Specimen F exhibits a γ' single phase, whereas specimen E consists of two phases, γ and γ' and specimen G of β and γ' . Here, the β phase has a B2 structure based on NiAl. For the two-phase specimens E and G, only the compositions of γ' phase were analysed. Table 3 lists the compositions of the γ' phase in specimens E, F and G, where the Ta contents of the γ' phases are approximately 4 mol%.

The γ' phase of each alloy was oriented such that the beam incidence was parallel to the $\langle 111 \rangle$ zone axis. The characteristic X-ray intensities were then measured at the exact $\langle 111 \rangle$ zone axis and a few degrees away from the zone. The fractional occupancy of Ta on the Al site, F_{Ta} , was determined from the following equation⁽²⁹⁾⁽³⁰⁾:

$$F_{Ta} = \left\{ (I_{Ta}/I_{Ni})_C / (I_{Ta}/I_{Ni})_N - 1 \right\} / \left\{ (I_{Al}/I_{Ni})_C / (I_{Al}/I_{Ni})_N - 1 \right\} \quad (3)$$

where the subscripts C and N represent the characteristic

Table 3 Compositions and site occupancy in the γ' phase.

Specimen	Compositions of γ' phase (mol%)			Occupancy F_{Ta} (%)
	Al	Ta	Ni	
E	17.2	4.4	78.4	98.4 ± 2.1
F	21.0	4.0	75.0	103.2 ± 2.3
G	21.8	4.2	74.0	101.3 ± 2.4

X-ray intensities measured at the channeling and non-channeling conditions, respectively, which correspond to the beam illumination at the exact $\langle 111 \rangle$ zone axis and a few degrees away from the zone.

We conducted seven series of F_{Ta} determination in specimen E, five series in specimen F and two series in specimen G. The average values of F_{Ta} for the three specimens are listed in Table 3. There is no significant difference in F_{Ta} between specimen E and specimen G. The all values of F_{Ta} result in nearly 100%. The present result signifies that Ta is located most favorably on the Al site irrespective of stoichiometry of Ni_3Al .

According to equilibrium phase diagrams of the Ni–Al–Ta system^{(23)–(26)} including the result in Fig. 9, the F_{Ta} is expected to be closer to 100% because the single-phase region based on the Ni_3Al ordered structure is extended into the Ta-rich side with the Ni fraction kept almost constant⁽³¹⁾. The present result is consistent with these phase diagrams and also is consistent with the theoretical prediction of Enomoto and Harada⁽³²⁾ based on a cluster variation method. The replacement of Al by Ta in the γ' lattice is considered to play an important role in the favourable “Ta effect” in nickel-base superalloys possibly through the variation of the nature of atomic bonding in the γ' phase.

In this study, the beam illumination was made along the $\langle 111 \rangle$ zone axis for the determination of site occupancy. The $\langle 100 \rangle$ zone axis orientation was not used because, as reported by Bentley⁽²⁹⁾, it led to a large scattering of data points including non-physical values which are greater than 100% or negative. The determination of site occupancy is possible with the beam direction parallel to the lattice planes such as (100) and (110). The occupancy determination with such beam illumination is called the planar-ALCHEMI, whereas that with zone-axis illumination is called the axial-ALCHEMI. In $L1_2$ -type Ni_3Al ordered alloys, there are no lattice planes consisting only of Al atoms. Thus, the channeling effect is weak in the planar-ALCHEMI, and a large error is likely to be introduced in occupancy determination although some results were available with use of the planar-ALCHEMI⁽³³⁾. By contrast, the channeling effect is large in the axial-ALCHEMI but instead this condition is susceptible to the delocalization effect due to the difference in impact parameter⁽³⁴⁾. In this study, we were able to minimize the delocalization effect by using the $TaM\alpha$ line which exhibits almost the same impact parameter as the $AlK\alpha$ line.

IV. Discussion

There are significant differences in the values of lattice misfit determined by CBED, misfit dislocation spacings and X-ray diffraction. In order to explain these differences, it may be necessary to consider firstly the effect of thermal expansion. CBED analysis yields the lattice constant at room temperature, whereas the dislocation structure at the γ/γ' interface represents the one at the annealing temperature (1273 K) because the annealing was terminated after quenching. Since the misfit dislocations are not lying on their slip planes, they are considered to be sessile and cannot change their spacings without long-range climb-process during rapid cooling from the annealing temperature. Thus, the lattice misfit obtained by CBED analysis may involve the difference in temperature when compared with the one derived from the spacing of interface dislocations. The thermal expansion coefficients are available for the γ and γ' phases such that $\alpha_\gamma = 1.48 \times 10^{-5}/K$ and $\alpha_{\gamma'} = 1.32 \times 10^{-5}/K$, respectively⁽³⁵⁾. Thus, the lattice constants at 1273 K can be estimated as $a_\gamma = 0.3608$ nm and $a_{\gamma'} = 0.3616$ nm for the γ and γ' phases, which then leads to the lattice misfit of +0.22% at 1273 K. This misfit is now in good agreement with the one obtained from the spacing of interface dislocations.

It is considered that the lattice misfit obtained from X-ray diffraction analysis also corresponds to the one at room temperature. However, the values of misfit as well as the temperature variation of the misfit are different. Since the difference is so large that it cannot be accounted for only by the difference in temperature.

Because the tie lines connecting γ and γ' phases are not given in the equi-lattice parameter diagram reported by Hong *et al.*⁽¹⁸⁾, the tie lines obtained in Fig. 9 are superimposed on the equi-lattice parameter diagram as shown in Fig. 11. Then the values of misfit shown in Table 2 for X-ray diffraction are obtained by reading the lattice parameters of the γ and γ' phases at the both ends of the tie lines and then using eq. (2).

In Fig. 11, the lattice parameters obtained by CBED are also included. The misfit values for CBED in Table 2 are calculated from these values. It should be noted that the lattice parameters of γ' phases obtained by CBED agree quite well with the values on the equi-lattice parameter lines and those reported by Balden⁽³⁶⁾. However, there is a considerable difference in the lattice parameters obtained for the γ phases by CBED and X-ray diffraction.

There must be at least two reasons for the difference in the obtained lattice parameters. First, the correction for the dynamical effect might be sufficient for the γ' phase but insufficient for the γ phase. Secondly, the linear assumption made by Hong *et al.*⁽¹⁸⁾ in the diagram might not be strictly valid for the Ni–Al–Ta ternary system. Hong *et al.*⁽¹⁸⁾ described by themselves that the equi-lattice parameter lines shown in Fig. 11 are “hypothetical” ones since they measured only the lattice parameters of

Fig. 11 Lattice constants of γ and γ' phases determined by CBED compared with the equi-lattice parameter diagram of Hong *et al.*⁽¹⁸⁾ obtained by X-ray diffraction analysis.

binary Ni-Al and Ni-Ta alloys in addition to that of γ' phase. It should not be certain that the misfit derived from the equi-lattice parameter lines is reliable or not. Further work is required.

V. Summary and Conclusions

(1) The parameters important to the mechanical properties of Ni-based Ni-Al-Ta alloys were examined by means of analytical electron microscope (AEM).

(2) Quantitative X-ray microanalysis showed that the $\gamma + \gamma'$ two-phase region extended to the Ni-rich side in the Ni-Al-Ta phase diagram. This was consistent with the results of Hong *et al.*⁽²⁵⁾ and Willemin *et al.*⁽²⁶⁾ but different from those of Nash and West⁽²³⁾.

(3) It was found that Ta was more partitioned into the γ' phase than the γ phase when it was less contained in the alloy, but as the Ta content increased, it tended to be partitioned equally into the γ and γ' phases.

(4) The lattice constants were determined from the analysis of high order Laue zone (HOLZ) line patterns in the transmitted beam of convergent beam electron diffraction (CBED). It was found that the lattice constants of both γ and γ' increased with an increase of C_{Ta} . It was also found that the lattice constants of γ' phase were invariably greater than those of the γ phase.

(5) Calculation of the lattice misfit, δ , between the γ and γ' phases showed that δ increased with an increase of C_{Ta} . Correction of the thermal expansion led to the lattice misfit consistent with the one derived from the spacing of interface dislocations.

(6) A good agreement was obtained for the lattice parameters of γ' phases obtained by CBED and X-ray diffraction. However, a significant difference was recognized for the lattice parameters of the γ phases, obtained by these two methods.

(7) The determination of site occupancy by the ALCHEMI technique showed that Ta was located most favorably on the Al site irrespective of the stoichiometry of the γ' phase.

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REFERENCES

- (1) A. F. Giamei, D. D. Pearson and D. L. Anton: *Mat. Res. Soc. Symp. Proc.*, Vol. 39, MRS, Pittsburgh, (1985), p. 293.
- (2) M. Gell, D. N. Duhl and A. F. Giamei: *Proc. 4th Intern. Symp. on Superalloys*, Ed. by J. K. Tien, S. T. Wlodek, H. Morrow, M. Gell and G. E. Mauer, ASM, Ohio, (1980), p. 205.
- (3) G. M. Janowski, R. W. Heckel and B. J. Pletka: *Met. Trans.*, 17A (1986), 1891.
- (4) Z. Meng, G. Sun, M. Li and X. Xie: *Proc. 5th Intern. Symp. on Superalloys*, Ed. by M. Gell, C. S. Kortovich, R. H. Bricknell, W. B. Kent and J. F. Radavich, AIME, Warrendale, (1984), p. 563.
- (5) S. M. Copley, A. F. Giamei, S. M. Johnson and M. F. Hornbecker: *Met. Trans.*, 1 (1970), 2193.
- (6) J. R. Brinegar, J. R. Mihalisin and J. VanderSluis: *Proc. 5th Intern. Symp. on Superalloys*, Ed. by M. Gell, C. S. Kortovich, R. H. Bricknell, W. B. Kent and J. F. Radavich, AIME, Warrendale, (1984), p. 53.
- (7) V. K. Lindoors: *Phil. Mag.*, 24 (1971), 709.
- (8) A. Lasalmonie and J. L. Strudel: *Phil. Mag.*, 32 (1975), 937.
- (9) D. F. Lahrman, R. D. Field, R. Darolia and H. L. Fraser: *Acta Metall.*, 36 (1988), 1309.
- (10) H. Brooks: *Metal Interface*, American Society for Metals, (1952), p. 20.
- (11) P. M. Jones, G. M. Rackham and J. W. Steeds: *Proc. R. Soc. London*, A354 (1977), 197.
- (12) R. C. Ecob, M. P. Shaw, A. J. Porter and B. Ralph: *Phil. Mag.*, A44 (1981), 1117, 1135.
- (13) Y. Tomokiyo, S. Matsumura, N. Kuwano, M. Kominami, T. Okuyama and K. Oki: *J. Electron Microsc.*, 35 (1986), 359.
- (14) T. Okuyama, S. Matsumura, N. Kuwano, K. Oki and Y. Tomokiyo: *Ultramicroscopy*, 31 (1989), 309.
- (15) T. Okuyama, S. Matsumura, Y. Tomokiyo, N. Kuwano and K. Oki: *ISIJ International*, 29 (1989), 191.
- (16) Y. Tomokiyo, T. Okuyama, S. Matsumura, N. Kuwano and K. Oki: *Mater. Trans.*, JIM, 31 (1990), 641.
- (17) *Crystal Data Determinative Tables*, 3rd Edition, Vol. 2 Inorganic Compound, Ed. by J. D. H. Donnary and H. M. Ondik, U.S. Department of Commerce, National Bureau of Standards and the Joint Committee on Powder Diffraction Standards, (1973), p. C-11, C-12.
- (18) Y. M. Hong, A. Mitsuhashi, Y. Mishima and T. Suzuki: *J. Japan Soc. Heat Treatment*, 28 (1988), 286.
- (19) G. Cliff and G. W. Lorimer: *J. Microsc.*, 103 (1975), 203.
- (20) Z. Horita, T. Sano and M. Nemoto: *Ultramicroscopy*, 21 (1987), 271.
- (21) Z. Horita, T. Sano and M. Nemoto: *J. Electron Microsc.*, 35 (1986), 324.
- (22) Z. Horita, T. Sano and M. Nemoto: *ISIJ International*, 29 (1989), 179.
- (23) P. Nash and D. R. F. West: *Metal Sci.*, 13 (1979), 670.
- (24) C. C. Jia, K. Ishida and T. Nishizawa: Tohoku University, unpublished work.

- (25) Y. M. Hong, Y. Mishima and T. Suzuki: *Mat. Res. Soc. Symp. Proc.*, Vol. 133, MRS, Pittsburg, (1989), p. 429.
- (26) P. Willemin, O. Dugue, M. Durand-Charre and J. H. Davidson: *Mater. Sci. Technol.*, **2** (1986), 344.
- (27) J. Taftø: *J. Appl. Cryst.*, **15** (1982), 378.
- (28) J. C. H. Spence and J. Taftø: *J. Microsc.*, **130** (1983), 147.
- (29) J. Bentley: *Proc. 11th Congr. on Electron Microscopy, Vol. 1*, Ed. by T. Imura, S. Maruse and T. Suzuki, Japanese Soc. Electron Microscopy, Tokyo, (1986), p. 551.
- (30) E. Goo: *Appl. Phys. Lett.*, **48** (1986), 1779.
- (31) R. W. Guard and J. H. Westbrook: *Trans. Met. Soc. AIME*, **215** (1959), 807.
- (32) M. Enomoto and H. Harada: *Metall. Trans.*, **20A** (1989), 649.
- (33) D. Shindo, M. Kikuchi, M. Hirabayashi, S. Hanada and O. Izumi: *Trans. Japan Inst. Metals*, **29** (1988), 956.
- (34) Z. Horita, H. Kuninaka, T. Sano, M. Nemoto and J. C. H. Spence: *Phil. Mag. A*, **66** (1992), in press.
- (35) D. A. Grose and G. S. Ansell: *Metall. Trans.*, **12A** (1981), 1631.
- (36) A. Balden: *Z. Metallk.*, **76** (1985), 24.