ISOTHERMAL PHASE TRANSFORMATIONS AND AGE HARDENING OF

RAPIDLY SOLIDIFIED Ti-24Al-11Nb

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Introduction

Isothermal phase evolution in a rapidly-solidified (RS) Ti-24at.%Al-11at.%Nb alloy was previously studied[1-5]. The supercooled β (or B2) phase in the RS Ti-24Al-11Nb underwent a sequential phase transition: β (B2) → T (ordered tetragonal) → O (ordered orthorhombic) → α_2 (ordered hcp) during isothermal aging at temperatures below 900°C. Both the T → O and O → α_2 transitions could be explained by a shape deformation mechanism which included a homogeneous lattice distortion and a rigid body rotation. In addition to the shape deformation, diffusion of excess Nb through the O/α_2 interface also was involved in the O → α_2 transition. Banerjee recently examined the T phase using a diffraction computation method and found that the crystal structure of the T phase should be orthorhombic[6]. At the same time, we have obtained evidence which revealed that a (110)<110> type shear strain (i.e. a shuffling of atoms on alternating (110) planes along <110>) was operated during the B2 → T transition, this atomic shuffling could change the stacking sequence of the T phase of 2H-type (ABAB stacking sequence). Accordingly, the symmetry of the T unit cell would be altered to an orthorhombic unit cell. While our work for a redefinition of the T phase will be published elsewhere, the T phase will still be used here to describe the phase evolution occurred in the aged sample. Recently, we also found that there exists an age hardening/softening behavior in the aged RS Ti-24Al-11Nb alloy. Age-hardening was found to occur during the stage of B2 → T → O transitions. The hardness increased to a maximum as the aged sample contained mostly the O phase. Subsequent aging resulted in a softening during the O → α_2 transition. The results are reported here.

Experimental detail

A rapidly solidified Ti_3Al+Nb alloy with a nominal composition of Ti-24at.%Al-11at.%Nb (Ti-14wt.%Al-21wt.%Nb) was used in this study. Two types of samples were used: powder and foil. The powder was produced from ingot via the plasma rotary electrode process (PREP) by Nuclear Metals Inc., Concord, MA. The chemical composition of the PREP powder was reported in [4]. The foil was produced from powder of identical composition via the inductive coupled plasma deposition (ICPD) process at GE Aircraft Engines, Lynn, Mass. Both the powder and foil samples were examined in their as-prepared and aged status using X-ray diffractometry, and electron microscopy. Prior to aging treatments, both the powder and foil samples were wrapped with tantalum foils and encapsulated in cleaned and evacuated quartz ampoules under vacuum (~10^-7 torr). Isothermal heat treatments of the PREP powders were performed at temperature range between 500°C and 700°C for different times (1 min to 168 h). Foil samples were aged at 650°C for different times (10 min to 24h). All the samples were water-quenched after heat treatment. Phase evolution in the powder samples was examined by X-ray diffraction (XRD) using Cu(Kα) radiation (λ = 1.5406 Å) in a Scintag XDS-2000 diffractometer operated at 40 kV and 35 Ma.
The employed scanning angles (2θ) for diffraction ranged from 25° to 75° with a scanning rate of 2° min⁻¹. Different phases were identified by matching the measured d_{hkI} with the calculated d_{hkI} of each phase based on the lattice parameters reported in [1,5]. Microhardness measurements were performed at room temperature from powder samples using a MICROMET Vickers-hardness indentor (applied load: 100 g, dwell time: 35 sec). Seven to eleven tests were conducted for each data point. Hardness value with both mean and standard deviations are reported.

Results and discussion

Phase evolution

Different phases contained in powder samples were mostly identified by matching the experimental d_{hkI} values from the XRD patterns and the calculated d_{hkI} of each phase (since the lattice constants of each phase are known). The XRD pattern generated from the as-prepared PREP powder is shown in Fig. 1(a). It shows that the β (a = 0.324 nm) is the only phase existed in the as-prepared powder. However, it is worth noting that an electron diffraction pattern [Fig. 1(b)] generated from the powder sample indicated that the β → B2 ordering reaction had already occurred within the powder during preparation. The absence of superlattice peaks in Fig. 1(a) indicates that the superlattice peaks generated by X-ray diffraction using Cu(Kα) radiation were too weak to be detected. XRD patterns generated from powder samples after aging at 500°C, 600°C and 700°C for different times are shown in Figs. 2-4. Figures 2(a) and (b) shows that the T phase (a = 0.64 nm, c = 0.65 nm, c/a = 1.02; or a = 0.65 nm, b = 0.90 nm, c = 0.45 nm on the basis of an orthorhombic unit cell) was formed in the powder sample after aging for 10 min 500°C, the T → O transition occurred when the powder samples were subsequently aged. The sample contained both the T and O (a = 0.60 nm, b = 0.98 nm, and c = 0.47 nm) phases after aging for 1 h at 500°C [Fig. 2(b)]. The phase retained in the sample after prolonged aging for 60 h at 500°C was mostly the O phase[Fig. 2(c)]. A similar result obtained from a sample after prolonged aging at 450°C was previously reported in [5]: the O phase was retained even after aging for 168 h. When samples were aged at temperatures above 500°C, the O phase became unstable and an onset of the O → α₂ transition was observed. The O phase was found to coexist with the α₂ phase (a = 0.58 nm and c = 0.465 nm) after aging at 600°C for 10 min [Fig. 3(a)]. The samples contained mostly the α₂ phase after aging for 48 h at 600°C [Fig. 3(b)] and for 5 h at 700°C [Fig. 4(b)]. Similar results for samples aged at 650°C and 850°C were previously reported in [5]. The X-ray results clearly suggest that there exists a critical temperature (~500°C), below which the O → α₂ reaction becomes very sluggish.

Age hardening/softening

Since hardness was found to be very sensitive to the phase content of Ti₃Al+Nb alloys[7,8], Vickers-hardness tests were employed to study the change of mechanical properties associated with the phase evolution. The measured hardness values ranged between 206 Hv and 572 Hv, where the unit of Hv is kg mm⁻² (1 kg mm⁻² = 9.8 Mpa). The size of an indentation is ~20 μm. Since the size of phase domains within a grain was in a nanometer range, the data sampled by the microindentation from a multi-phase region were considered to be statistically adequate. The hardness of the as-prepared powder (contained mostly B2 phase) was measured to be (~395 Hv or 3.9 Gpa). This corresponds to an estimated yield strength (σ_y) of ~1.3 GPa according to σ_y (MPa) = (9.8 Hv)/3. The hardness data measured from samples aged at 500°C, 600°C and 700°C are shown in Fig. 5. For all temperatures studied, the hardness initially increased with increasing aging time, reached a peak value ~550 HV and then decreased. However, as can be seen in Fig. 5(a), the hardness variation obtained from samples aged at 500°C show only a slight decrease (decreased from ~550 Hv to ~520 Hv) even after prolonged aging. On the contrary, for samples aged at 600°C and 700°C [Figs. 5(b) and 5(c)], a significant decrease of hardness occurred after prolonged aging. A comparison made between hardness data and XRD results indicates that the age hardening took place during the β(B2) → T → O transitions and the age softening occurred during the O
Fig. 1. (a) XRD pattern (Cu K\textsubscript{α}) showing the existence of the β phase in the as-prepared powder; (b) electron diffraction pattern (zone axis = [021]) showing the existence of the B2 phase in the as-prepared powder (superlattice spots are indicated by arrows).

Fig. 2. XRD patterns (Cu K\textsubscript{α}) generated from powder samples after aging at 500°C for different times: (a) 10 min, (b) 1 h and (c) 60 h.
Fig. 3. XRD patterns (Cu $K_{α}$) generated from powder samples after aging at 600°C for different times: (a) 10 min and (b) 48 h.

Fig. 4. XRD patterns (Cu $K_{α}$) generated from powder samples after aging at 700°C for different times: (a) 5 min and (b) 5 h.
Fig. 5. Hardness variations of powder samples after aging at (a) 500°C, (b) 600°C and (c) 700°C for various times. The mean deviation is indicated by the vertical bars.
\(-\alpha_2\) transition. The result shown in Fig. 5(a) simply indicates that the O phase was stable when samples were aged at 500°C. While careful work is needed to elucidate the mechanisms of the age hardening and softening behavior, here we directly correlate the age hardening and softening with the microstructural evolution. This is visualized in Fig. 6, where hardness variation obtained from powder samples aged at 650°C is displayed in accompany with the microstructural evolution that was observed from foil samples aged at 650°C. The hardness increases to a maximum \((-572 \text{ Hv or 5.4 GPa})\) when the aged sample contained mostly lath-like O phase. The O phase appeared to be the hardest phase among all \((\sigma_y = 1.8 \text{ GPa})\). This was mainly because it contained a high density of dislocations, stacking faults and microtwin as a result of a large extent of shape deformation during the \(T \rightarrow O\) transition\[5\]. Subsequent aging resulted in a significant softening accompanied with the \(O \rightarrow \alpha_2\) transition. The hardness decreased to \((-260 \text{ Hv or 2.6 GPa})\) when the sample contained mainly the equiaxed \(\alpha_2\) grains. This observation is in agreement with the findings reported in\[9\]: the yield strength of the O phase is greater than that of the \(\alpha_2\) phase. Here the estimated yield strength of the \(\alpha_2\) phase is \(-870 \text{ MPa}\).

Finally, some comments are made here regarding the ordered orthorhombic (O) phase. The O phase in the \(\text{Ti}_3\text{Al+Nb}\) system is not the stoichiometric compound \((\text{Ti}_2\text{AlNb})\) which has frequently referred in literatures\[9-11\]. Instead, the O phase should be regarded as an ordered phase which has a wide range of Nub solubility: \(-10 \text{ at.\%} \text{ - 25 at.\%}\). Furthermore, the critical temperature (below which the O phase is stable) is dependent upon the Nub content of the O phase. For instance, the O phase \((\text{Ti}_2\text{AlNb})\) which contained 25at.% Nub was reported to be stable up to \(-1000^\circ\text{C}\[11\].

Conclusions

Time-temperature-transformation behavior of rapidly solidified \(\text{Ti-24Al-11Nb}\) between 500° and 700°C was studied using X-ray diffraction, transmission electron microscopy and Vickers microindentation methods. The results showed consistently that an age hardening/softening behavior occurred during isothermal phase transformations. Age-hardening was found to occur during the stage of \(B2 \rightarrow O\) transition. The hardness increased to a maximum \((-5.4 \text{ GPa})\) when the aged sample contained mostly lath-like O phase. Subsequent aging resulted in a softening during the \(O \rightarrow \alpha_2\) transition. The hardness decreased to a minimum \((-2.6 \text{ GPa})\) when the sample contained mostly equiaxed \(\alpha_2\) phase. The results also revealed that there existed a critical temperature \((-500^\circ\text{C})\) above which the \(\beta(B2) \rightarrow T \rightarrow O \rightarrow \alpha_2\) transitions occurred, and below which the \(O \rightarrow \alpha_2\) reaction became very sluggish. Accordingly, the O phase \((-11 \text{ at.\%Nub})\) was stable when the RS \(\text{Ti-24Al-11Nb}\) alloy was aged below \(-500^\circ\text{C}\).

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References

Figure 6. Relationships between the variation of hardness and microstructure during isothermal aging at 650°C.

- Time (sec)
- Hardness (Hv)
- Temperature = 650°C

Images showing microstructural changes with time and hardness variations.