Ti-2007 Science and Technology, edited by M. Ninomi, S. Akiyama, M. Ikeda, M. Hagiwara, K. Maruyama
The Japan Institute of Metals (2007)

Anisothermal Alpha Phase Precipitation in Ti-3Al-8V-6Cr-4Zr-4Mo(Ti-38644)

Basak Yazgan-Kokuoz, JoAn Hudson, Henry J. Rack

School of Materials Science and Engineering, Clemson University, Clemson, SC 29634, USA

The present investigation has examined the influence of grain size on α phase formation during cooling from above the β transus in Ti-3Al-8V-6Cr-4Zr-4Mo. X-ray diffraction, optical and transmission electron microscopy has shown that the critical cooling rate required to suppress α phase formation is relatively insensitive to grain size, at least within the coarse grained samples examined. Precipitation of the α phase in Ti-3Al-8V-6Cr-4Zr-4Mo with decreasing cooling rate involved heterogeneous nucleation and growth at prior β grain boundary triple points, grain boundary edges, grain surfaces and silicide-matrix interfaces. Not all grain boundaries served as α precipitation sites, their capacity to serve in this function apparently being related to the relative misfit/misorientation between two adjacent β grains. Finally the insensitivity of the critical cooling rate to suppress α phase formation on grain size in Ti-3Al-8V-6Cr-4Zr-4Mo appears to be related to early nucleation site saturation, precipitate growth being the primary factor controlling the subsequent increase in the volume fraction of α with decreasing cooling rate.

Keywords: metastable beta titanium, hardenability, alpha precipitation

1. Introduction

The development of advanced high strength-low density materials for high sections, e.g. landing gears and bulkheads, remains a prime objective of the aerospace industry. Among the many titanium alloys that have been examined for this purpose metastable beta titanium alloys remain of particular interest because of their potential for achieving uniform properties through heavy sections. This hardenability is directly controlled by the ability to suppress α phase precipitation during cooling from the α/β or β solution treatment temperature.

A previous study of Ti-6.8Mo-4.5Fe-1.5Al (TIMET LCB) [1], a "solute-lean" metastable β titanium alloy, has shown that cooling rates greater than 8 Ks must be attained in order to suppress α phase precipitation. With progressively slower cooling rates heterogeneous nucleation and growth of the α phase commenced at prior grain triple points, grain boundary edges, grain boundary surfaces and inclusion-matrix interfaces. Finally general intragranular α precipitation was observed at the slowest cooling rates examined, 0.3 Ks^{-1}. The present study has examined the precipitation of the α phase during cooling of Ti-3Al-8V-6Cr-4Zr-4Mo (Ti-38644), a "solute-rich" metastable β alloy[2]. The precipitation response of this alloy during cooling was characterized by x-ray diffraction, optical and transmission electron microscopy.

2. Experimental Procedures

The Ti-38644 examined had a composition of 3.22Al, 7.77V, 5.88Cr, 3.85Zr, 4.18Mo, 0.08Fe, 0.09O, 0.02C, 0.01N, 72 ppm H, with a beta transus of 1013K, and was supplied as 31.75 mm diameter hot rolled and solution treated bar. Optical and x-ray pole figure examination of the as-received material indicated a hot worked single-phase β-microstructure, with a fiber texture. The influence of the cooling rate on α-phase formation was examined utilizing a modified Jominy end quenching technique[2]. Specimens (25. 4mm in diameter and 101.6mm in length) were machined from the as-received bar, coated with boron nitride (BN) to minimize oxidation during solution treatment and insulated with Kaowool, the latter to promote one-dimensional heat flow during subsequent cooling. Two solution heat treatments were examined, the first a single solution heat treatment at 1228 K for 4 h followed by quenching. The second solution heat treatment procedure included a hold at 1323 K for 2 h, followed by furnace cooling to 1228 K, holding for anadditional 2 h and quenching. These procedures resulted in an average mean linear intercept grain size of 160 and 295 μm, respectively.

Water and helium gas cooling was conducted in accord with ASTM A255-88. Temperatures during cooling were continuously monitored by ten K-type thermocouples cold-welded at a pre-determined distances from the quenched end on the Jominy periphery[3]. Following cooling the Jominy bars were sectioned at specified distances from the quenched-end using electro-discharge machining.

Samples were mechanically polished using a multi-step polishing procedure involving grinding through 1000 grit SiC followed by vibratory polishing with Al_2O_3 and colloidal SiO_2. Initial x-ray diffraction experiments utilizing these samples were undertaken using a Scintag XDS 2000 θ-θ diffractometer operated at 40 kV and 30 mA, the diffraction spectra being collected with a Mostek detector. Step scans with 2θ ranging between 20 and 100 were conducted at the rate of 1.2°/min using CuKα radiation. Following data acquisition raw data files were analyzed using DMSNT version 1.37 software, under Windows NT. Each peak was analyzed by "profile fitting" using a Pear 7-type of curve with an acceptable error of ±10%. Rocking scan analysis was also conducted to establish the existence of the α phase when at low volume, the presence of the (10-10) α peak at 2θ angles between 35.0 and 35.75 degrees being examined with ω angles varied from 5 to 35 degrees.

Microstructure analysis of these samples was done using a Nikon inverted optical microscope. Examination of single phase β was performed following etching with 5 v/o HF — 5 v/o HNO_3 — bal. H_2O, whereas two-phase (α+β) microstructures were etched with 3 vol. % (HF) — 6 vol. % (HNO_3) — bal. (H_2O). Finally selected solution heat-treated and cooled samples were examined using a Hitachi 9500 transmission electron microscope. Samples for this purpose, ~0.35 mm thick, were initially wafered

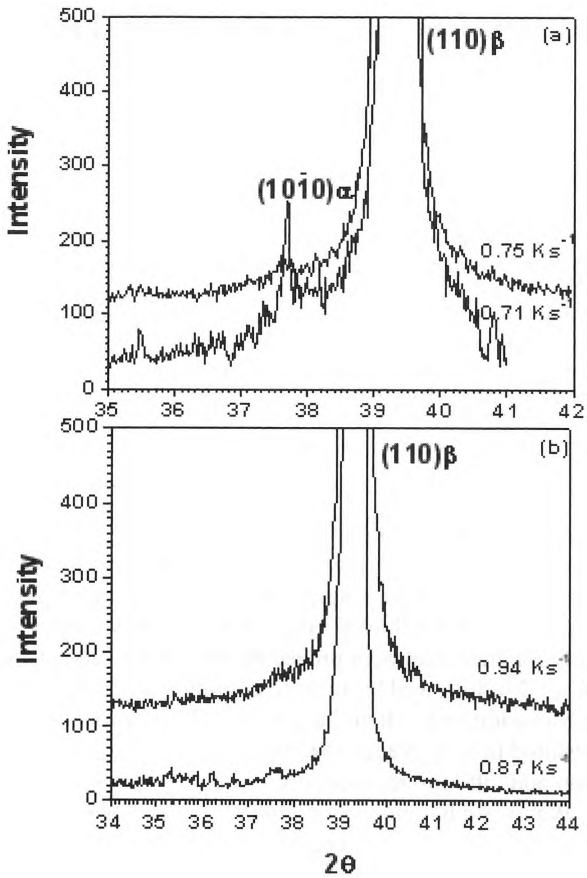

Figure 1. Wide-angle x-ray diffraction results for Ti-38644 having grain size of 160 and 295μm cooled at indicated rates.

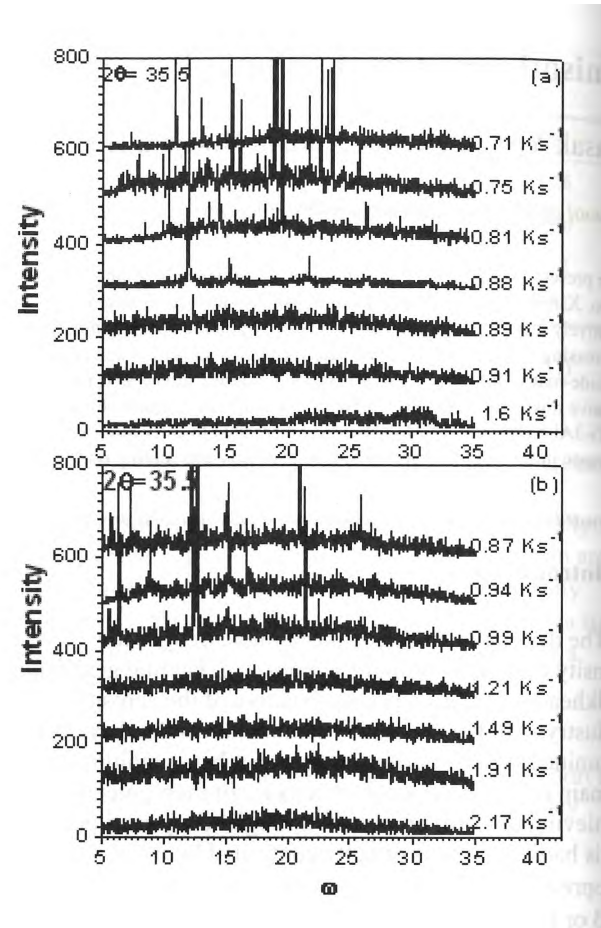

Figure 2. Rocking curve x-ray diffraction results for Ti-38644 having grain size of 160 and 295 μm cooled at indicated rates.

using a precision diamond saw, manually ground to 150μm on a 600 grit paper and thinned to 10μm using a GATAN dimple grinder. These samples were then ion milled using a GATAN dual mill at 4 kV and 1 mA/13 degree angle utilizing a liquid nitrogen cold-finger.

3. Experimental Results

Wide-angle x-ray diffraction results, illustrated in Figure 1, indicate that the critical cooling rate required for retention of a single phase β microstructure in Ti-38644 is relatively insensitive to grain size. These results suggest that retention of a single phase β microstructure requires cooling at rates ≥ 0.7 Ks^{-1} when solution treated at 1228K (grain size = 160 μm) , while a single phase microstructure was retained at all cooling rates examined when solution treated at 1323K/1228K(grain size = 295 μm).

Rocking curve analysis, Figure 2, showed however that α phase formation following solution treatment at 1228K actually occurred at cooling rates ≤ 0.9 Ks^{-1} while formation of α after double solution treatment occurred at rates ≤ 1 Ks^{-1}. Optical microscopy indicated that α phase precipitation, independent of grain size, involved heterogeneous nucleation and growth progressing with decreasing cooling rate from prior β grain boundary triple point to grain edges and grain surfaces, Figure 3. Moreover, as shown in Figure 3, α precipitation did not occur on all prior β grain boundaries, suggesting that the mis-orientation between neighboring prior β grains and the local boundary curvature, as shown in Figure 4, were additional factors controlling the rate of α phase nucleation and growth. Further study also indicated that once initial α grain boundary nucleation and growth occurred, side-branching of α along selected planes into the neighboring prior β matrix commenced, the degree of side-branching increasing with decreasing cooling rate, Figure 5. Finally no generalized α phase precipitation within the prior β matrix was observed, the only evidence of intra-granular α phase precipitation being associated with the heterogeneous nucleation and growth of α/ silicide-matrix interfaces[4], Figure 6.

4. Discussion

The present investigation has shown that the cooling rate required to suppress α phase formation in Ti-38644 following solution treatment is relatively insensitive to grain size, at least for the two large grain sizes, 160 and

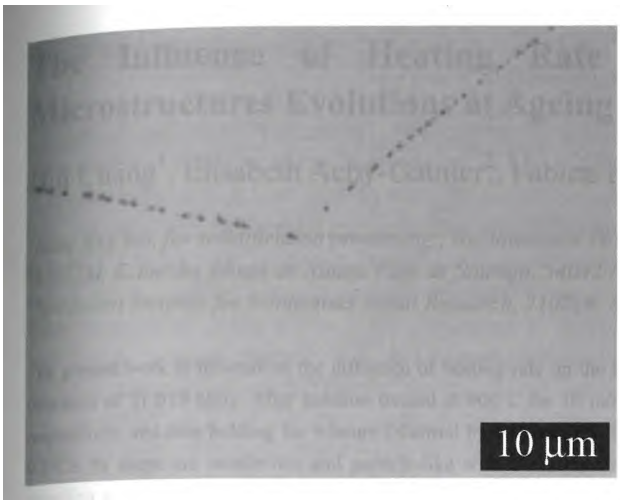

Figure 3. Precipitation of α phase on selected grain boundaries in Ti-3Al-8V-6Cr-4Zr-4Mo solution treated at 1323 K for 2 h, furnace cooled to 1228 K, held for 2 h and quenched at 0.99Ks^{-1}.

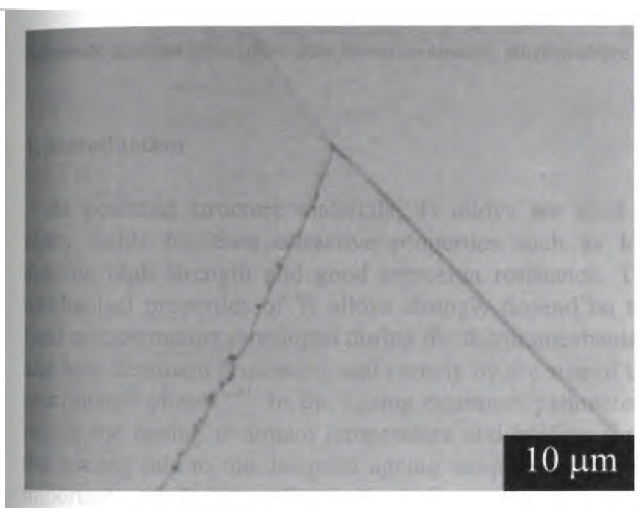

Figure 4. Grain boundary curvature driven precipitation of α in Ti-3Al-8V-6Cr-4Zr-4Mo solution treated at 1228K for 4 h and quenched at 0.89Ks^{-1}.

295 μm examined in this study. Once a cooling rate below approximately 1 Ks-1 is reached, α precipitation progresses, with decreasing cooling rate, in a manner similar to that previously observed in Ti-6.8Mo-4.5Fe-l.5Al(TIMET LCB) . It involves sequential formation of a at prior β grain boundary triple points, grain edges and grain surfaces with decreasing cooling rate. However the stages in α precipitation in Ti-38644 with respect to site selection do not appear to be as distinct as was previously observed in Ti-6.8Mo-4.5Fe-1.5Al(TIMET LCB). This suggests that heterogeneous nucleation site saturation may have occurred more rapidly in Ti-38644. Indeed Cahn[5,6] has suggested that site saturation will occur rapidly during cooling, that is the under cooling necessary from nucleation initiation to site saturation should be small. Under these conditions growth of the α phase in Ti-38644 will predominant and will control the rate of increase in α volume fraction with decreasing cooling rate.

Figure 5. Transmission electron micrograph of Ti-3Al-8V-6Cr-4Zr-4Mo showing the side-branching of α into neighboring prior β matrix following solution treatment at 1228K for 4 h and quenched at 0.71Ks^{-1}.

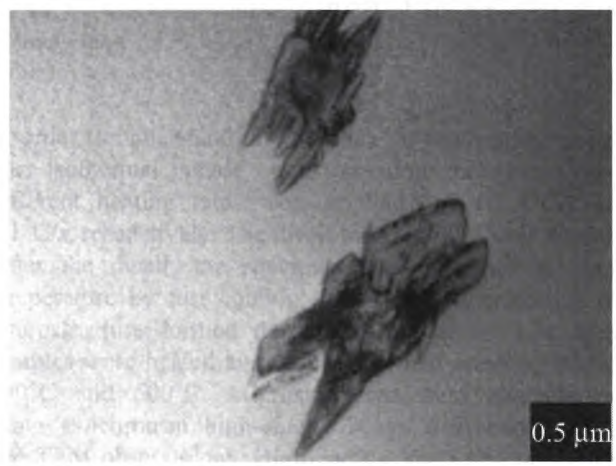

Figure 6. Transmission electron micrograph of α phase on silicide matrix interfaces following solution treatment at 1228K for 4 h and quenched at 0. 71Ks^{-1}.

The lack of sensitivity to grain size in establishing the cooling rate necessary to suppress α formation suggests that the β solution treatments examined in this study may have altered the grain boundary structure. It would, for example, be expected that the misoreintation between neighboring grains will decrease with increasing elevated temperature exposure, i.e., increasing grain size. Indeed, the importance of grain boundary structure and local boundary curvature on α phase precipitation can be seen in Figures 3 and 4. Finally the critical cooling rate required to suppress α phase precipitation in Ti-6.8Mo-4.5Fe-1.5Al(TIMET LCB)[1)] is approximately 8 times higher than that for Ti-38644. Once again this suggests that growth of the α phase controls nucleation and growth process, the increased β stabilizing solute content of Ti-38644 vis a vis Ti-6.8Mo-4.5Fe-1.5Al (TIMET LCB) tending to limit the diffusivity of elements, primarily Al, which favor the precipitation of the α phase. This decrease in diffusivity of α stabilizers with increasing β stabilizer content also

limits the nucleation and growth of α within the prior β matrix as observed in Ti-38644.

Acknowledgements

The authors wish to knowledge the assistance of Ms. M. Gheorghe during the initial phase of this program and Mr. Herbert Boeckels during the preparation of this manuscript. The kind donation of the Ti-38644 bar used in this investigation by RMI Titanium, Niles, OH as well as the partial financial support of the High Speed Research Program at NASA-Langley are also acknowledged with gratitude.

REFERENCES

1) M. Gheorghe, J. I. Qazi and H. J. Rack, in *Ti-2003, Science and Technology*, G. Leutering and J. Albrecht, eds.,Wiley-VCH Verlag GmbH & Co, KGaA, Weinheim, Geremany, 2004, pp.1155-1161.
2) T. J. Headley and H. J. Rack, Met. Trans., **10A,** 1979, pp. 909-924.
3) T. Ahmed and H. J. Rack, Mat. Sci & Eng., **A243,** 1998, pp. 206-216.
4) A. Vassel, in *Beta Titanium Alloys in the 1990*'s, D. Eylon, R. Boyer and D. A. Koss, eds., TMS, Warrendale, PA, 1993, pp. 173-185.
5) J.W. Cahn, Acta Met. **4,** 1956, pp. 449-459.
6) J. W. Cahn, Acta Met. **4,** 1956, pp. 572-575.