

Origins and importance of fine secondary α in slowly cooled Ti6Al4V

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Abstract

It has been known for sometime that slowly cooled Ti6Al4V (Ti64) contains only about 10vol% of β and that this β takes three different forms. Firstly, there are thin volumes of interfacial β , between the grains of α , which are clearly the remains of the β present at high temperatures left when α replaces the β present at high temperatures, by diffusion and boundary migration. Secondly, there are regions of roughly equiaxed β at triple points, but some of this β contains very fine secondary α , whereas the other grains are retained β . Analytical transmission electron microscopy has been used in an attempt to understand the origin of these two different types of equiaxed β grains. It has been found that grains, which are retained β contain about 20wt%V, but the β grains that contain secondary α originate from parent β grains that have only about 14wt%V. The origin and the significance of these observations will be discussed in the presentation.

1. Introduction

Fine secondary α can be formed in transformed β grains when Ti alloys are slowly cooled at about 5°C/min after high temperature processing. The main aim of the present work has been to understand the mechanism which gives rise to this fine secondary α in slowly cooled samples of Ti64. The fraction of fine secondary α can be increased by cooling more rapidly. The cooling rate in a HIP can be increased to about 50°C/min by jet cooling and this increases the fraction of fine α and slightly increases the strength [1]. It has also been shown that air-cooling Ti64 at rates in excess of 200°C/min from just below the transus, leads to a very high fraction of coarser secondary α and to significant improvements in properties. It is clear therefore that increasing the cooling rate can increase the fraction of fine α , as would be expected, but the origin of fine secondary α is currently not understood. If it could be understood perhaps the amount of fine, rather than coarser secondary α , could be increased and stronger alloys developed. The work described in this paper attempts to investigate this possibility.

2. Experimental

The Ti64 alloy used was supplied by TLS as gas atomised powder, with powder particles between about 50 and 180 microns. The powder was HIPped (Hot Isostatically Pressed) in an EPSI Lab facility at 930°C for 4h at 100MPa and slowly cooled in the HIP at about 5°C/min.

Analytical transmission electron microscopy (TEM) was carried out on a JEOL 2100, which was interfaced to an INCA EDX system and on a Thermo Fisher Scientific Talos F200X with a Super-X EDS system. Both microscopes were operated at 200kV. Scanning electron microscopy (SEM) was carried out using a TESCAN MIRA-3 SEM equipped with an Oxford Instruments XMax silicon drift detector (SDD) for energy dispersive X-ray spectroscopy (EDS), which was operated at 10kV. Conventional electropolishing was used to prepare some samples but most of the β is polished away using this method and when β was to be characterised focused

ion beam SEM (FIB/SEM, FEI Quanta 3D FEG) was used to prepare specimens. This technique also allows site-specific examination of samples.

3. Results

A typical SEM image of an as-HIPped sample is shown in Figure 1(a) where the bright phase is β . The β is present in three different forms; as regions of interfacial β , the remnants of the β grains that were present at the HIP temperature and is also present as two different types of roughly equiaxed regions at triple points; some are featureless, whereas others clearly contain a second phase. This difference between the β at triple points is not obvious in this figure, but higher magnification images, such as that shown in Figure 1(b) clearly show fine secondary α within the β grain.

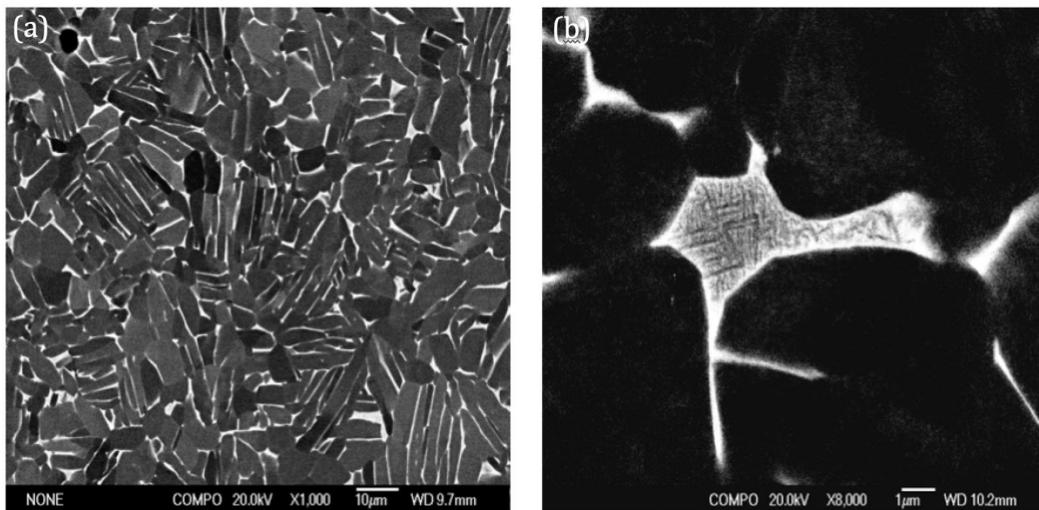


Figure 1 (a) Low magnification back-scattered SEM image; (b) High magnification image of HIPped Ti64 cooled at 5°C/min in the HIP.

High magnification images, such as that shown in Figure 1(b), show very clearly a high density of fine secondary α in the roughly equiaxed β and that the thin regions of interfacial β do not contain any secondary α , but show uniform bright contrast. Bright contrast is also shown along the perimeter of the β that contains secondary α . FIB has been used to prepare thin samples for TEM of regions that contains fine secondary α and detailed EDX measurements carried out [2]. In order to obtain the composition of the parent β grains that contain secondary α the beam was spread so that a large part of the grain was analysed, which included fine α and the β separating the fine α . Analyses showed that whilst the β that was free of fine α contained about 20wt%V, whereas grains with fine α contained only about 14-15wt% V. The β in these grains was also analysed and also contained about 20wt%V.

In addition to the EDX data, diffraction patterns and dark field images using selected diffracted beams were obtained from the β grains that contained secondary α and from the β grains that contained no secondary α . Diffraction patterns and a dark field image, obtained from a grain that contained secondary α , are shown in Figures 2(a) and (b). As expected the pattern, shown in Figure 2(a), which was obtained using a small diffraction aperture that covered several α laths and the inter-lath β is complex and obviously as well as β maxima, shows maxima from several habits of α . The dark field image shown in Figure 2(b) using closely spaced α reflections, confirms that the pattern contains maxima from α .

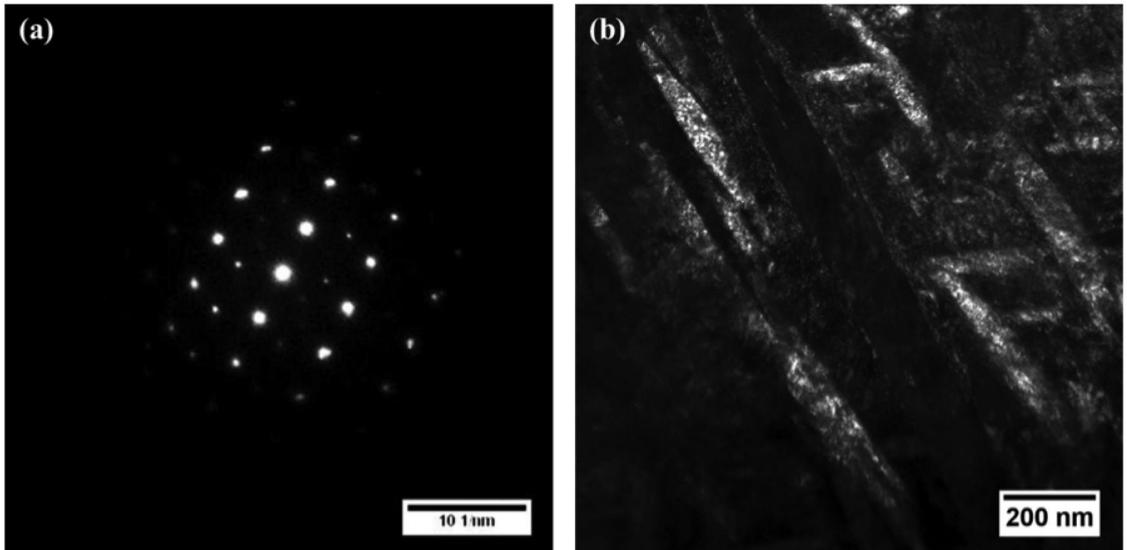


Figure 2 (a) Selected area electron diffraction pattern taken along $\langle 110 \rangle_{\beta}$ from a grain containing secondary α ; (b) dark field image taken using α reflections.

Diffraction patterns have been taken from regions of β that did not contain any secondary α , as shown in Figure 3, using either a small probe or using a defocussed beam. These patterns contain diffuse scattering, which is more obvious in Figure 3(a) and attempts have been made to obtain dark field images from the diffuse scattered intensity, but no useful images could be obtained. This diffuse scattering was not obvious in the grains that contained secondary α .

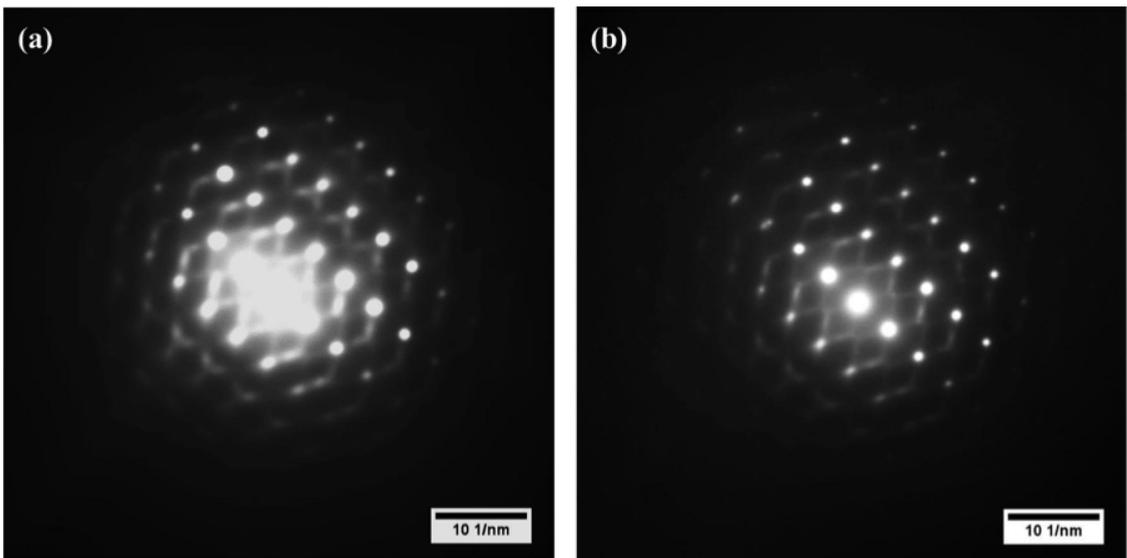


Figure 3 (a) Microdiffraction pattern and (b) selected area pattern from a β grain.

The analyses in the table show that the parent grain, which forms fine secondary α , has a composition of about Ti-3Al-15V-2Fe and an alloy of this composition has been made up and examined. An SEM image of a sample after being cooled at 5°C/min from about 930°C is shown in Figure 4. Clearly this alloy may have interesting properties. Work is underway to assess the tensile properties and the stability of this fine microstructure at elevated temperatures.

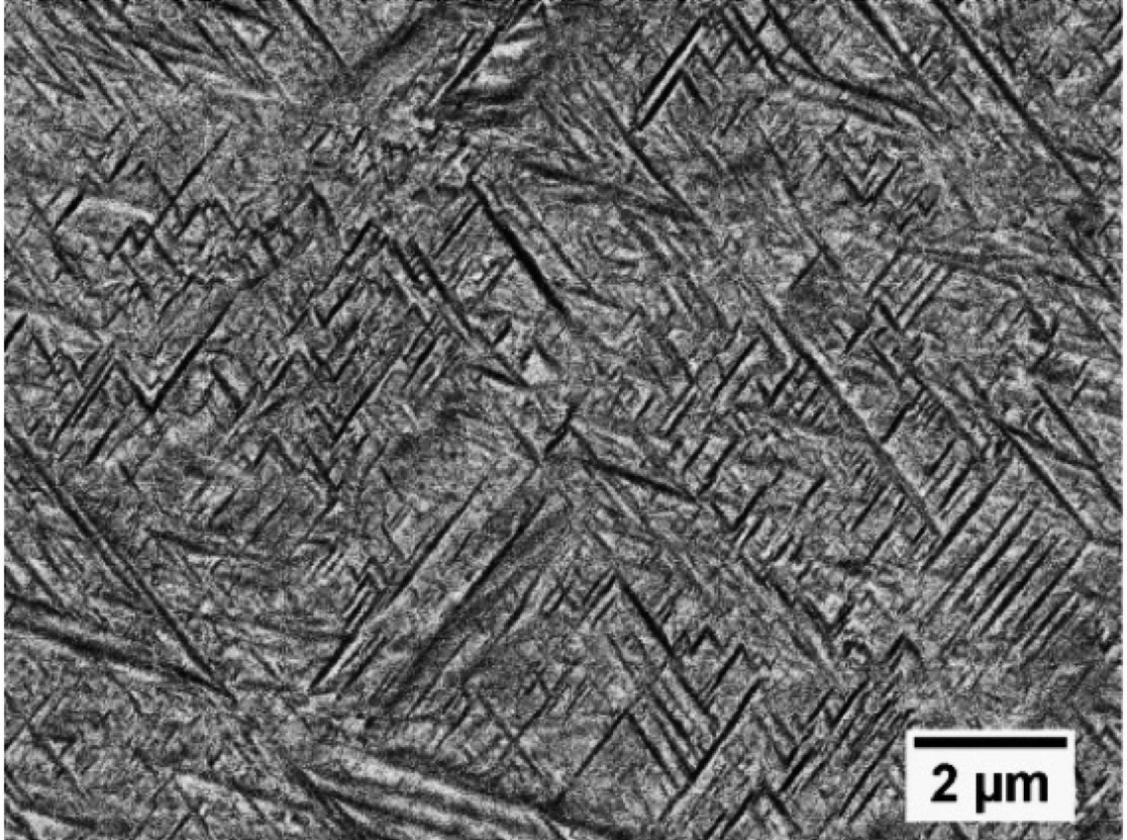


Figure 4 Back scattered SEM image of an alloy of composition Ti3Al15V2Fe, slowly cooled from well above its transus, showing fine α .

4. Discussion

The main aim of the work reported in this paper was to understand why different β grains in slowly cooled Ti64 showed such different microstructures, varying from retained β grains to β grains that contain a high density of fine secondary α . The observations have shown that this difference is associated with the fact that the compositions of β grains present after slow cooling powder-HIPped Ti64 range in V-content from about 14wt% to over 20wt%. It is important to understand how these differences arise. It should be noted that β is preferentially polished away when samples are produced by electropolishing and the data obtained from β grains has been taken from samples prepared using FIB and very few grains have been analysed. If more were analysed other β grains of different mean compositions, which transform to form fine secondary α , would presumably be found and/or the range of compositions of retained β grains increased. The compositions of β , which have been analysed range from 19.5 to 20.5wt%. The analysis of parent β grains, that form fine α internally, is influenced by the extent of the scanned area since for

slight changes the amount of fine α contributing to the analyses would vary. It is thus not straightforward to detect small changes in the compositions of parent β grains, which contain fine α . There is the possibility that powders produced by the EIGA process will have liquid droplets of different composition. Because of this possibility, HIPped AP&C plasma atomised, Tekna plasma atomised and spheroidised powders and of TIMET PREP powder have been examined, and it was found that they all have both types of β grains; those that contain secondary α and those that are retained β .

The amount of retained β when slowly cooling Ti64 from 930°C at 5°C /min has been measured recently as 11% on the same HIPped powder as used in the present work [1]. CALPHAD calculations predict that about 4% of β is the equilibrium amount at room temperature and that 11% of β is present at 870°C. Assuming that CALPHAD calculations are not too far in error it thus follows that the proportions of α and β present in Ti64 at 870°C are frozen in. However, it is clear that the composition of β can change very significantly during cooling below 870°C since although some grains have about 13%V (the amount expected at 870°C), some have over 20%V, which corresponds to the equilibrium concentration at about 790°C.

The fact that β grains exist at 790°C which have very different V-concentrations, is clear evidence that diffusion occurs very differently in different volumes of a sample below 870°C. In some β , the V-content increases by diffusion from the adjacent α , to 20wt% so that it forms retained β , although the temperature at which it reaches this composition is not known. In other grains, of average V-content of about 14wt%, the composition of the β between the α laths of 20wt%V is reached by diffusion within those grains, associated with the precipitation of fine α , but this must occur below about 760°C, the estimated transus of this alloy. It thus appears that the rate of diffusion of V from α to β is very different in different β grains, because some β grains reach 20wt% by diffusion from the surrounding α and others by diffusion within the β grain. The factors that could lead to the differences in the extent of diffusion, which underlie the different microstructures observed, are considered below.

There are several factors that could locally influence the diffusion rate. Thus, the work of Elmer et al [3] showed that significant changes in unit cell volumes of β occur during heating or cooling as the V-content changes, leading to high internal stresses, which, in the present work, may lead to a high dislocation content in some β grains. If these are formed by internal stresses during cooling, they could change local diffusion rates; the extent of the influence depending upon the dislocation density. Diffusion will also be influenced by the misorientations between the β and the α grains that replace the β during cooling and by the corresponding structures of the boundaries, which migrate during the transformation.

These factors, different dislocation densities, different misorientations between different α and β grains and the associated difference in boundary structure appear to be the factors which could lead to compositional differences between different α grains and between different β grains. Because of their different stability during cooling, these differences in composition between β grains lead to the different microstructures observed of retained β grains and β grains that contain fine secondary α . Thus it should be understood, that any initial small differences in V-content between different β grains would tend to increase during cooling because any grain that has a slightly low V-content will have a higher transus and form some α in that grain, increasing the V-content of the β in that grain. Other grains, with a slightly higher V-content, will have a lower transus and will, as they cool, continue to increase their V-content from the remaining α , thus further increasing their stability. These differences will continue to increase on further cooling until no further diffusion can occur and the final microstructure will contain β grains, with an average composition of about 14wt% V that have formed fine α , and retained β grains containing 20wt% V. The large difference in V-contents observed, thus develop gradually during cooling and the different grains are locally in equilibrium.

This sequence of events could lead to the perimeter of grains that contain secondary α being rich in V and depleted in Al due to diffusion to/from the surrounding α , whilst the fine α precipitated internally. Once fine α is totally formed, the β within that grain has a V-content of about 20wt% so that further diffusion to/from the surrounding α will cease leaving the Al-depleted and V-enriched perimeter, which would explain the presence of the bright perimeter visible in Figure 1(b).

The diffusion to/from the surrounding α makes local precipitation of α at the boundary less likely because it is effectively β -stabilised, leading to the zone depleted of α at the boundary. If the diffuse scattering shown in Figure 3 is due to the presence of fine omega, the secondary α may then be nucleated on this omega as observed in β alloys [4]. Whether that is possible depends upon the temperature at which fine α is formed during cooling at 5°C /min and the stability of omega in this alloy. We know neither

of these facts and at this stage we cannot decide whether or not omega can play a part, although with a transus of about 750°C it may be that the α forms at a temperature above that at which omega is stable.

The only β remaining at room temperature, when cooled at 5°C /min, has a V-content close to 20%. If this formation of secondary α , which occurs in β of about 14wt%V, did not occur, X-ray diffraction and even electron diffraction would reveal a spread in lattice parameter of β at room temperature, because as noted above, changes in V-content give rise to significant changes in the volume of the unit cell of β ; such a spread in the lattice parameter of β in Ti64 at room temperature is not observed [3].

The microstructure of an alloy of composition defined by that of the parent β grain that forms fine secondary α in slowly cooled Ti64, is fine α which offers a new way of designing Ti alloys.

5. Conclusions

- (1) The parent β grains that transform to fine secondary α contain about 14wt% V.
- (2) The grains, in Ti64, which did not contain any secondary α , contain about 20wt% V and are thus stable as β .
- (3) The diffraction patterns from V-rich grains show diffuse scattering, which may mean that omega acts as nuclei for the secondary α .
- (4) An alloy has been prepared of composition defined by that of the parent β grain that forms secondary α in Ti64. The microstructure of this alloy, when slowly cooled is a very fine α/β .

Acknowledgements

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