Effect of yttrium addition on grain growth of α, β and α+β titanium alloys

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Abstract. In this study effects of small amount of yttrium addition on grain growth of C.P titanium (single α), Ti-14Mo-3Nb-1.5Zr (single β) and a newly developed Ti-4.5Al-6Nb-2Mo-2Fe (α+β) titanium alloy are investigated. By adding yttrium nano-sized Y₂O₃ particles are formed in ‘in-situ’ mode. These particles lead to significant suppression of grain growth in single α and β alloys rather than in the α+β alloy. The results are discussed with regard to conventional grain growth and grain boundary pinning models. It is concluded that yttrium is a potential micro alloying element in titanium alloys and could be used as a grain refining agent.

1. Introduction

The titanium and titanium alloys are used in a wide variety of industrial applications such as aerospace, marine, automotive, and so on [1]. Generally, hot working for breakdown of the cast structure of titanium ingots is conducted at high temperatures which is known as β-processing. On the other hand, the final stage of hot working is mostly conducted in the two-phase region at lower temperatures [2]. The main reason for selection of such a kind of processing route is that heating to temperatures above the β transus temperature of titanium alloys causes a very coarse microstructure due to rapid grain growth of the β phase, which cannot be refined by the final heat treatment [2].

Recently, Hotta et al. [3] have shown that small amounts of yttrium addition to SP-700 alloy resulted in a marked refinement of β grain size as well as retardation of β grain growth at elevated temperatures. Yttrium has a strong affinity to oxygen in titanium, and in-situ Y₂O₃ oxide particles are formed by a very small amount of yttrium addition around 0.02 to 0.05 mass%. These particles are able to control and suppress β grain growth by exerting a pinning effect on the grain boundaries (Zener Effect) [4]. The Zener equation, which was modified by Nishizawa et al. [5], described by Eq. (1), shows the relationship between the average grain radius R and the particle radius r, and its volume fraction f_p :

\[ R = \beta r / f_p^m \] (1)

where m and β are 2/3 [5] and 4/3 [4], respectively.

In the two-phase region of α+β titanium alloys with the volume fraction of the phases being comparable, the rate of coarsening is controlled by inter-diffusion between the phases over distances which are in the order of the phases’ size [6]. Generally, it is known that the grain growth rate in deformed metallic materials is much faster compared with un-deformed ones. This behaviour is known as dynamic or deformation grain growth [7-11].

Recently, some studies were carried out to control grain growth in titanium alloys using particles like TiB [12-15]. These studies are related to titanium matrix composites reinforced with ceramics. Such materials show superior high temperature strength. However, some disadvantages of these materials restrict their application. One is high production cost through conventional composite production methods like adding TiB particles into the molten material. The other is deterioration of elongation during superplastic deformation because of using a high fraction of hard particles [16, 17]. Production of in-situ formed Y₂O₃ particles by adding Y during the melting process showed to be very
effective in controlling β grain size during high temperature annealing [3]. Furthermore, Y is much cheaper than B and the production process is simpler.

In this study, the influence of yttrium on grain growth was investigated in single α, C.P titanium, new β, Ti-14Mo-3Nb-1.5Zr, and new α+β type titanium alloy, Ti-4.5Al-6Nb-2Mo-2Fe, in particular the pinning of grain boundaries by Y₂O₃ particles during isothermal heating at 1073K of all alloys and also during hot deformation of the α+β two-phase structure.

2. Experimental procedure

Button ingots of alloys were prepared by arc melting with a non-consumable electrode and a water-cooled copper mould. Raw materials used in this study were titanium sheets containing 0.02% oxygen, high purity metal sheets and yttrium powder. Static grain growth in these alloys was examined by isothermal heating of cylindrical specimens with a diameter of 10 mm and a length of 10 mm at 1073 K for time periods between 3.6 to 40 KS, followed by water quenching. Grain growth behaviour during hot deformation of α+β alloy was investigated by hot compression using a hot working simulator of THERMEC-Master Z. Specimens were compressed at a temperature of 1073K to a true strain of 0.7 followed by He-gas quenching. The deformation was conducted at strain rates of $2 \times 10^{-4}$ s⁻¹.

The microstructure was observed by optical microscopy (OM) and scanning electron microscopy (SEM, PHILIPS XL30FEG). Average α grain size was measured from micrographs using the line intercept method. The presence and size of Y₂O₃ particles were identified by thin foil observation using transmission electron microscopy (TEM, JEOL-2000EX2) at 200kV.

3. Results and discussion

Figure 1 shows TEM micrographs of 0.05Y added Ti alloys subjected to heat treatment at 1073K for 3.6ks (a, b and d) BF images and (c) SAD pattern of single β alloy. Figure 2 shows changes of grain size with holding time of (a) single α, (b) single β and (c) α+β Ti alloys.

The Y₂O₃ particles seem to have spherical shape. Figure 2 shows changes of grain size in single α, single β and α+β alloys with holding time at 1073K. It is obvious that the addition of yttrium to single phase alloys leads to extensive refinement of the microstructure. Higher amount of yttrium addition causes lower average α and β grain size. By adding 0.05% Y grain growth is fully suppressed in these alloys.
On the other hand, in the two-phase alloy, which has a fine initial microstructure, grain growth is not suppressed. However, as shown in Fig. 2 (c), yttrium results in a slightly finer grain size. The reason could be attributed to the size and fraction of the Y$_2$O$_3$ particles. Figure 3 shows the grain size as a function of particle size for three different levels of particle volume fractions based on the Zener equation. It should be noted, that this figure shows the effect of particle fraction and size on grain size of the single phase microstructure. Here, it is assumed that if the particles could completely suppress grain growth in the single phase, this will result in complete retardation of grain growth in the two-phase microstructure. From Fig. 3 it is clear that for suppressing grain growth in the two-phase alloy with a volume fraction of 0.05% the size of the Y$_2$O$_3$ particles should be decreased to about 10nm. Meanwhile, it is possible to control the grain size by increasing the volume fraction of the Y$_2$O$_3$ particles with size of 0.1μm from 0.05 to 1%. This shows that for both controlling the grain size and complete suppression of grain growth optimization of the particle size and fraction is necessary.

The effect of deformation on the microstructure of the two-phase alloy used could be cleared more when comparing the microstructure after static and dynamic grain growth at the same heating time. Microstructures of the base and 0.05Y alloy after heating for 3.6ks at 1073K are shown in Fig. 4 (a) and (b), respectively. After 50% compression of the alloy at a strain rate of $2 \times 10^{-4}$s$^{-1}$ which corresponds to the same heating time (3.6ks), the microstructure obviously is coarser as shown in Figs. 4 (c) and (d). This difference in grain size is due to the difference between dynamic and static grain growth. Dynamic grain growth results in about two times larger grain size compared to static grain growth. Superplastic elongation of both alloys was obtained by hot tensile test. The base alloy yields very high superplastic elongation of about 2500%. However, addition of yttrium to the alloy reduces this value to 1500%. The achievement of large superplastic elongations in new alloys is dedicated to extremely fine two-phase microstructures as well as an optimum design of chemical composition. It is important to note that here very high elongation was obtained at much lower temperature than observed for conventional superplastic titanium alloys like Ti-6Al-4V [16]. Addition of yttrium caused a reduction in superplastic elongation which may be attributed to the presence of Y$_2$O$_3$ particles. These particles may act as strain accumulation sites during hot tensile deformation enhancing void nucleation and growth during superplastic deformation and resulting in a reduction of ductility [14, 15]. Therefore, it seems that increasing the volume fraction of Y$_2$O$_3$ particles is not an appropriate way to control grain growth of the
two-phase alloy. The only procedure remaining to control grain growth seems to be the decrease of particle size. In this regard, further studies are needed to achieve finer particle sizes.

4. Conclusions

In this study, the effect of yttrium addition to single α, C.P titanium, single β, Ti-14Mo-3Nb-1.5Zr, and new α+β type titanium alloy, Ti-4.5Al-6Nb-2Mo-2Fe, was investigated and the following results were obtained. Y$_2$O$_3$ particles with an average size around 100 nm are formed in an in-situ mode. Grain growth is suppressed by pinning of the grain boundaries by Y$_2$O$_3$ particles during isothermal heating of single α and single β alloys at 1073K while adding Y has a small effect on grain growth of the two-phase structure. Grain growth is accelerated by deformation, but 0.05% yttrium addition showed to be effective in suppression of dynamic grain growth. Y$_2$O$_3$ particles have detrimental effect on superplastic elongation.

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