Materials Science and Engineering A 398 (2005) 93–98

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A study on the hardness variation of α- and β-pure titanium with different grain sizes

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Received 11 October 2004; received in revised form 1 March 2005; accepted 1 March 2005

Abstract

In this study, the hardness and the microstructure of α- and β-pure Ti treated with different grain sizes were investigated. The α-Ti specimens were obtained by heating pure Ti at 750 °C for various periods and then quenched in water, while the β-Ti specimens by heating at 1000 °C for various periods and then quenched in water. Experimental results show that the hardness of α-Ti decreased with increasing the grain size; whereas, the hardness of β-Ti tends to increase with increasing grain size. From the microstructures examined with transmission electron microscope, it can be realized that the strengthening mechanisms of α- and β-Ti are quite different. Grain boundary could be the main strengthening microstructure of α-Ti and the smaller grain size the higher hardness. The major strengthening microstructure of β-Ti was martensitic twin with a very high dislocation density. The amount of martensitic twin increased with increasing the grain size of β-Ti and, therefore, the larger grain size the higher hardness.

Keywords: CP α- and β-titanium; Grain size; Martensite strengthening

1. Introduction

Pure Ti is widely used as the construction material in chemical and biomedical engineering owing to its superior corrosion resistance and appropriate mechanical properties [1–3]. Generally, pure Ti can exist in two crystalline structures [4], namely hexagonal close-packed (hcp) and body-centered cubic (bcc) structures. At room temperature, pure Ti has a hcp crystal structure. This structure tends to transforms to a bcc crystal structure at the temperature above 883 °C, which named as the β-transus temperature. A quasi-equilibrium martensite structure could be obtained as Ti being heated higher than β-transus temperature and then rapidly cooled to low temperature [5,6].

Due to the convenience in measurement, hardness is generally used to represent the strength and wear-resistance level of a material; the higher strength and the higher wear resistance are usually attendant on the higher hardness. It is accepted that refining the grain size, increasing the dislocation densities and inducing the hardening precipitates in the material are the generally recommended methods to increase the strength and the hardness of a given material [7–9]. However, so far we know that scare literature has been mentioned about the hardness variation of α- and β-Ti specimens with respect to the grain size. In present work, this hardness variation is investigated together with the results of the microstructure examination with transmission electron microscope (TEM). Especially, a strengthening microstructure of β-Ti is introduced in this study.

2. Experimental procedure

Commercial pure Ti (ASTM Grade 2) bar, 16 mm in diameter and 100 mm in length, was utilized in this study with its chemical composition given in Table 1. The as-received pure Ti bars were further heat-treated to obtain α- and β-Ti. The α-Ti specimens with different grain sizes can be achieved by

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doi:10.1016/j.msea.2005.03.004

Table 1
The chemical composition of pure titanium (ASTM Grade 2) used in this study

Elements	wt%
C	0.1
H	0.015
O	0.25
N	0.03
Fe	0.3
Ti	BAL

heating the as-received bars in the air furnace at 750 °C for different times varying from 1 to 240 h and then quenching in a 25 °C water bath. Similarly, the β-Ti specimens with different grain sizes were obtained by heating the as-received bars at 1000 °C for 1–240 h and quenching in water. Although α-Ti structure was still developed when specimens were quenched from 1000 °C into water, we name them as β-Ti in this study to differentiate the α-Ti specimens, which were quenched from 750 °C.

After heat treatment, the bar specimens were sectioned in disc type, 16 mm in diameter and 10 mm in length, for hardness test, grain size measurement and microstructure examination. The hardness test was conducted using Matsuzawa Digital Microhardness Tester (Model MXT-α7e) with an indenting load of 500 g. The mean hardness and its standard deviation were calculated through six measurements made in arbitrary positions, roughly–evenly distributed, on the surface of the disc specimen.

To develop the grain morphology, the disc specimen was mechanically polished to 1200 grid finish and then immersed in an etchant composed of 10 mL KOH (40%), 5 mL H_2O_2 and 20 mL H_2O at 60 °C for 30 min. The grain size of α- and β-Ti specimens were statistically evaluated by linear-intersection method [10,11], by which OM micrographs were taken on five, evenly separated, positions from the middle surface of the disc specimen. A set of parallel lines with equal distance of 1 cm was then drawn on these five micrographs. The grain size of the specimen was calculated based on the number of intersected points obtained from the parallel lines and grain boundaries. Then, the length of the total parallel lines was divided by the total intercepted points, the as-obtained length then multiplied the magnification of the micrograph and gave the mean grain size. The metallographically etched surfaces of α- and β-Ti specimens were also examined with scanning electron microscope (SEM, Hitachi S-3500N).

The microstructures of Ti specimens with different grain sizes were examined with transmission electron microscope (TEM, Jeol 2000 FX). The TEM samples were prepared using a twin-jet electrochemical cell (Fischion Instruments Inc.). The jet-polishing was conducted in an electrolyte composed of 60 vol% methyl alcohol, 35 vol% *n*-butanol and 5 vol% perchloric acid at 30 V across electrodes until a tiny hole was produced in the middle of the specimen, around which the sample was so thin that made TEM examination and analyzing feasible.

3. Results and discussion

3.1. Microstructural characteristics

Fig. 1a and b shows the SEM and OM micrographs of etched surface morphologies of the α-Ti specimen heat-treated at 750 °C for 1 and 240 h, respectively. The etched surface with deeply etched gain boundary attack and relatively shallow grain etching of α-Ti can be observed in Fig. 1. Besides, it can be seen from Fig. 1, the grain sizes of α-Ti specimens increase with increasing the heating time. It means that the metallographic etching method can be successfully used to reveal the grain size of α-Ti specimen. Micrograph of β-Ti heat-treated at 1000 °C for 240 h is shown in Fig. 2a, in which only very large equiaxed grains in β-Ti specimen can be found. Fig. 2b and c shows the SEM micrographs of etched surface morphologies of β-Ti specimen heat-treated at 1000 °C for 1 and 240 h, respectively. The SEM

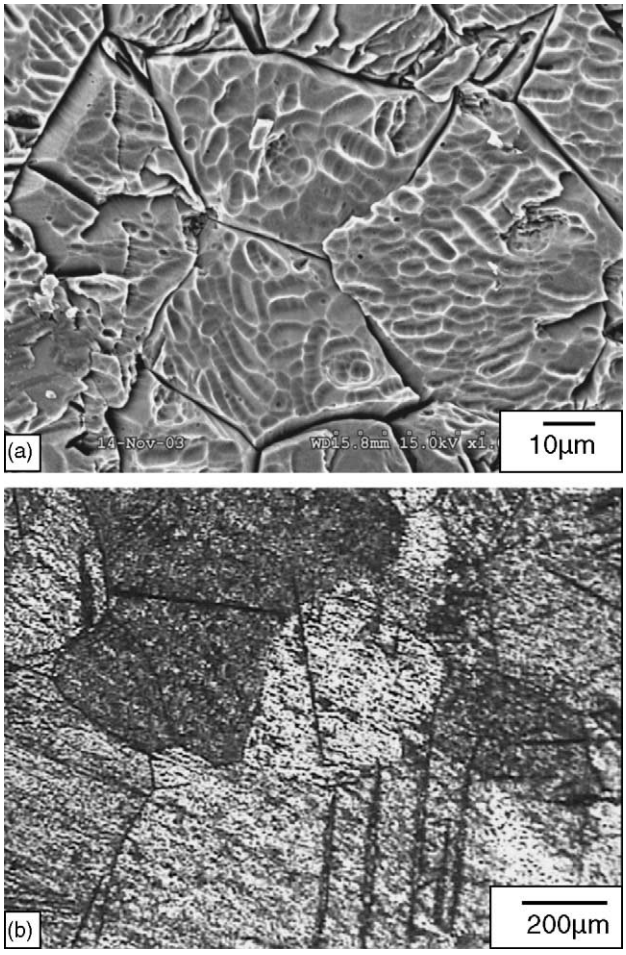

Fig. 1. Micrographs of α-Ti for specimens heat-treated at 750 °C for (a) 1 h, SEM and (b) 240 h, OM (etched specimen).

micrographs show only part of the grain interior and apparently each β-Ti grain comprised many elongated α-Ti laths and the width of the lath in β-Ti grain increased with increasing heating time at 1000 °C. In addition, it can be clearly seen in Fig. 2c, the α-Ti laths presented in a β-Ti grain intersected each other and assumed many shaped triangle morphologies. Researches [5,15] have indicated that the high cooling rates would lead to a martensite transformation of the Ti-alloys. The lath thickens relatively slowly than grows along lath axis during cooling, leading to the metallographicaly orientated interlaced lath morphology.

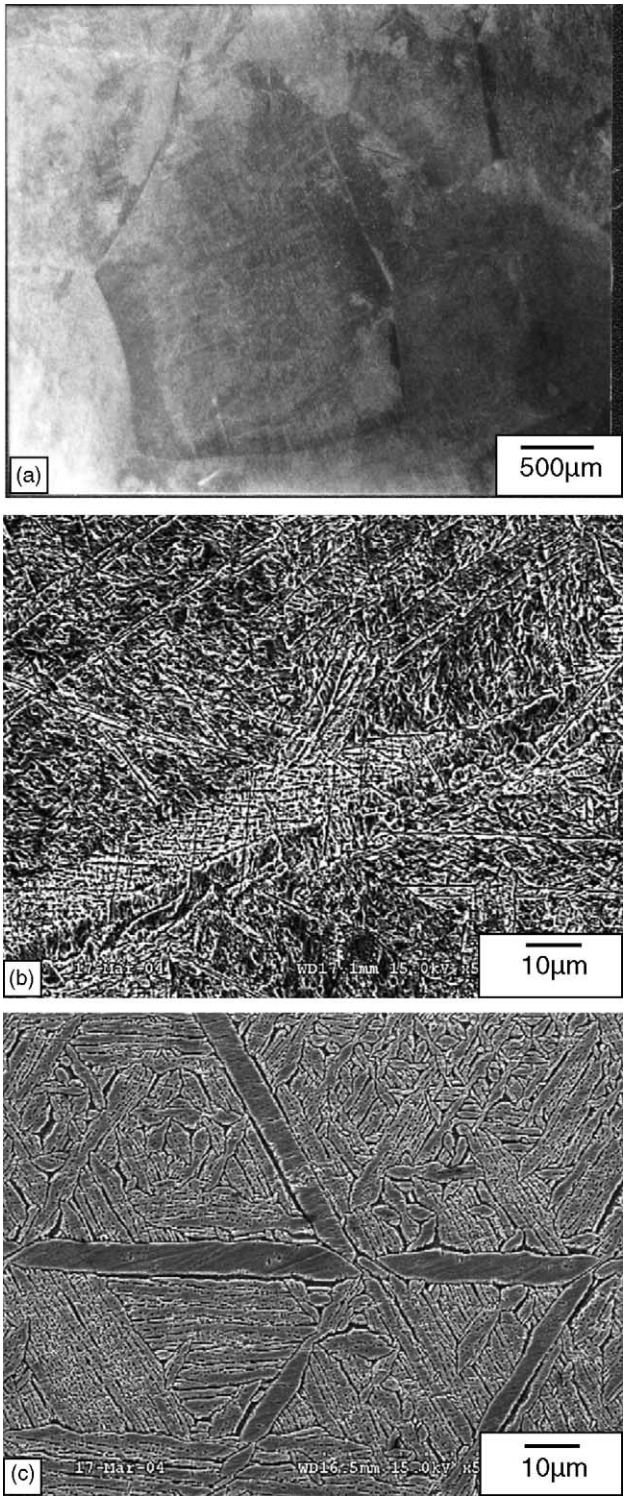

Fig. 2. Micrographs of β-Ti for specimens heat-treated at 1000 °C for (a) 240 h, OM (b) 1 h, SEM and (c) 240 h, SEM (etched specimen).

3.2. Grain size determination

Fig. 3 shows the grain size variation of α- and β-Ti specimens heat-treated for different times at 750 and 1000 °C. As-expected, the grain sizes of both α- and β-Ti specimens increase with increasing the heating time. The grain size of 30 μm can be detected when the as-received Ti specimen was heated at 750 °C for 1 h and 80 μm for that of 240 h. When the as-received Ti specimens were heated at 1000 °C, the grain size of β-Ti reaches as high as 700 μm for 1 h and 2250 μm for 240 h. The much larger grain size of β-Ti can be ascribed to the higher grain growth rate of β-Ti in the thermal activated process at 1000 °C. Furthermore, Gil et al. [9] has proved that the activation energy for grain growth of β-Ti was relatively low when heated at temperatures higher than β-transus temperature. However, it must be noted that each β-Ti specimens were composed of many elongated laths (Fig. 2b and c). That is, the elongated laths were developed from a β-Ti grain during quenching from 1000 °C, and the smaller grain, the finer width of the elongated lath.

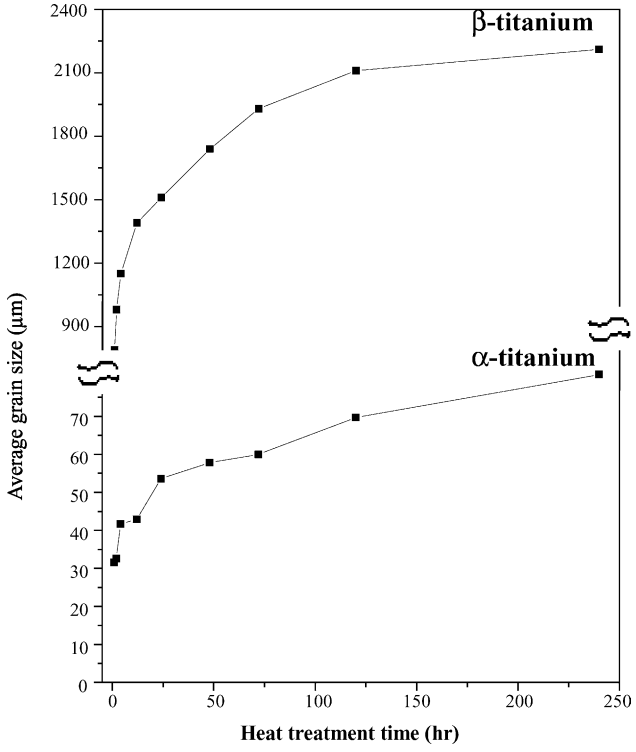

Fig. 3. Grain sizes of α- and β-Ti specimens after heat-treating at 750 and 1000 °C for different times then quenching.

3.3. Microhardness

The hardness variation of α- and β-Ti after different heating times was presented in Fig. 4. It can be clearly seen from the figure that the hardness of α-Ti specimen decreases rapidly initially then slowly with the increasing heating time at 750 °C. From the results shown in Figs. 3 and 4, it can be seen that the α-Ti specimen with larger grain size has relatively low hardness than that with relative small grain size. This result is in agreement with the well-known strengthening mechanism through grain refinement [12,13]. Contrary to the hardness variation of α-Ti specimen, the hardness of the β-Ti specimen increases with increasing heating time at 1000 °C. Although the grain sizes of β-Ti specimens are much larger than those of α-Ti specimens (Fig. 3), the hardness of β-Ti specimens is much higher than that of α-Ti specimens. This can possibly be attributed to the formation of many elongated fine laths, which developed during rapid cooling from a temperature above the β-transus temperature and the elongated lath boundaries can also impede dislocation migration, as grain boundary does, leading an increase of hardness. However, the increase in lath width with increasing heating time (Fig. 2) would reduce the total lath boundary area, leading theoretically to a decrease in hardness, this obviously contradict to the result shown in Fig. 3 for β-Ti. Consequently, there would possibly be other strengthening microstructure presented in β-Ti specimens, especially in the specimens with larger grain sizes. It would therefore be helpful to conduct TEM investigation of the microstructure of Ti specimens with different grain sizes.

3.4. TEM examination

Fig. 5a and b shows the TEM micrographs of α-Ti, with distinct grain sizes, heat-treated at 750 °C for 1 and 240 h, respectively. Only low dislocation density can be observed (Fig. 5); that is, there are no other possible strengthening

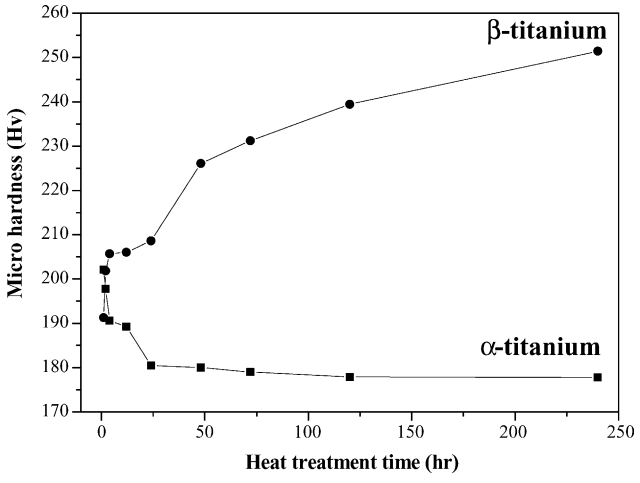

Fig. 4. The microhardness variation of α- and β-Ti specimens after heating at 750 and 1000 °C for different times then quenching.

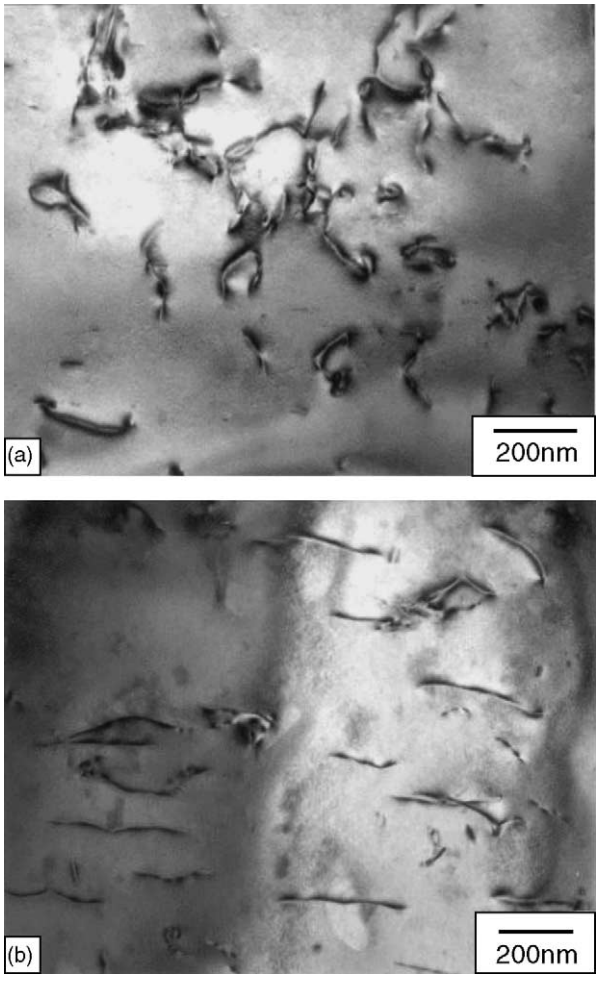

Fig. 5. TEM micrographs of α-Ti after heating at 750 °C in (a) 1 h and (b) 240 h, respectively (plane-view specimen).

microstructures except difference in their grain sizes. Thus, it can be reasonably recognized that the main strengthening factor for the α-Ti specimens is the amount of grain boundary area in the specimens; therefore, the smaller grain size, the higher hardness.

The TEM micrographs of β-Ti specimens with different grain sizes were presented in Fig. 6. As shown in Fig. 6a, no presence of specific strengthening microstructure in β-Ti specimens heated for a short time (≤12 h) at 1000 °C were detected. Surprisingly, some martensite with very high dislocation densities, as viewed from Fig. 6b and c, presented in the β-Ti specimens heated at 1000 °C for a longer time (≥24 h). It can be found the width of the martensite increased with increasing grain size of β-Ti. It means that a large grain size of β-Ti is required for the presentation of the martensite through rapid cooling from 1000 °C. The martensite was identified with selected area diffraction (SAD) pattern using electron beam. The SAD pattern shown in the corner of Fig. 6c presents an hcp structure, the same as alpha phase, in the zone axis of $[1\,\bar{2}\,1\,\bar{3}]$. Apparently, there was a very high dislocation density in

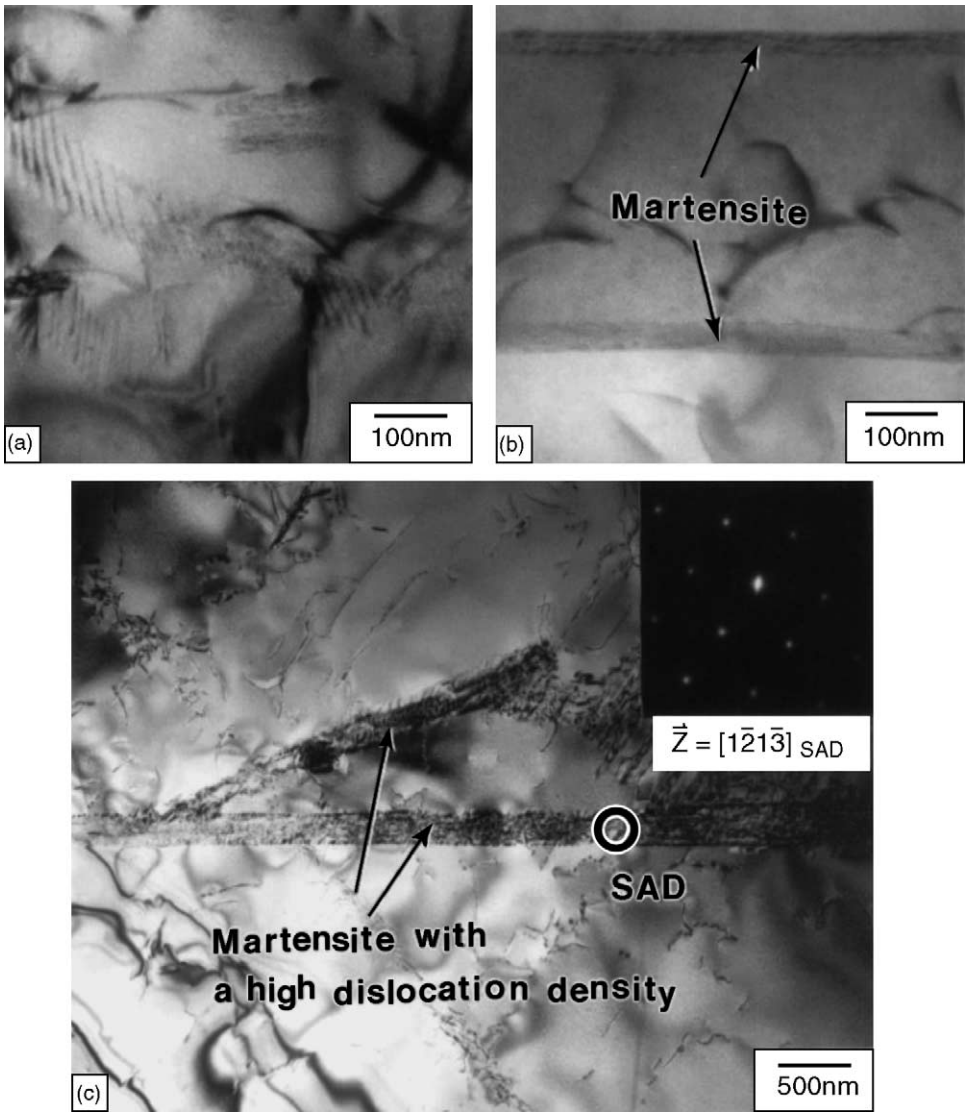

Fig. 6. TEM micrographs of β-Ti after heating at 1000 °C in (a) 1 h, (b) 72 h and (c) 240 h, respectively (plane-view specimen).

the martensite alpha phase (Fig. 6c). It is interesting to find that the hardness of these specimens was raised apparently (Fig. 4). It is well known that the microstructure with a very high dislocation density can effectively impede dislocation migration. Thus, a reasonable interpretation of the hardness increase of β-Ti specimens with large grain sizes is the presence of these martensite twins in the specimens.

Several researchers [5,14,16] have reported that martensite structure can be achieved in Ti–6Al–4V alloy when it was rapidly cooled from the temperature above β-tranus temperature. In this study, however, we confirm that the martensite structure with a very high dislocation density can also be formed in pure titanium, which is rapidly cooled from 1000 °C and transformed into elongated lath within a very large grain size. The width and amount of martensite increased with increasing the heating treatment time at 1000 °C (Fig. 6b and c). In summary, moreover, the martensite with high dislocation density was reckoned as the main strengthening microstructure of the β-Ti specimens.

4. Conclusion

In this study, the main strengthening microstructures of pure α- and β-Ti specimens with different grain sizes are elucidated and differentiated. The former is strengthened by grain refinement; while the latter by martensite formation in the elongated lath structure. The martensite structure and high dislocation density can be detected in β-Ti specimens with relative large grain sizes by heating at 1000 °C then rapidly quenching in water.

Acknowledgement

The authors thank the National Science Council of Republic of China for financially supporting this work under the Contract No.: NSC 90-2216-E-182-006.

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