Thermal Stability of Microstructure and Mechanical Properties of Ultrafine-Grained Pure Titanium

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Abstract Thermal stability of microstructure was investigated of ultrafine-grained (UFG) pure titanium subjected to anneal at 300°C, 400°C, 500°C and 600°C. Hardness variety was used to evaluate the effect of annealing temperature on mechanical properties in UFG titanium. Microstructural observation showed that the average grain size slightly increased in UFG pure titanium annealed below 400°C, while the dislocation density significantly decreased. As the annealing temperature increased to 500°C and 600°C, the average grain size dramatically increased and dislocation density sharply reduced. The hardness almost remained constant, when the annealing temperature was 300°C, while it gradually decreased, as the annealing temperature increased to 400°C. The hardness dramatically decreased in the samples annealed at 500°C and 600°C. The relationship between the microstructural stability and mechanical property variety in UFG Ti during annealing is discussed.

Keywords Ultrafine-grained pure titanium, Thermal stability, Grain size, Hardness

1. Introduction

Research on the ultrafine processing of grains in metals and alloys has attracted a lot of attentions since Prof Valiev and co-workers firstly produced the refined microstructures of less than 1 micron, which has a superior high strength, in copper via equal channel angular pressing (ECAP) technique two decades ago [1-4]. Ultrafine-grained (UFG) metals and alloys have demonstrated the higher combination of strength and ductility than the coarse-grained counterparts. The UFG titanium shows higher corrosion resistance than coarse-grained one due to rapid passivation [5]. Several severe plastic deformation (SPD) techniques have been used to fabricate UFG or nano-grained metals. For example, high pressure torsion (HPT) used for Cu, Cu-Zn, and Cu-Sn alloys [6,7], cumulative roll bonding (ARB) used for some fcc and hcp metals [8-10]. Thermal stability is a big concern for ultrafine-grained materials. Majid Hoseini et.al found that UFG pure titanium had a good thermal stability of microstructure below 450°C [11]. The grain growth activation energy (Q) is close to the self-diffusion activation energy in Ti [12,13]. Although the ultrafine processing of pure titanium has been well studied, little information is available on the thermal stability of mechanical properties and microstructure in UFG pure Ti. The objective of this paper is to study microstructural stability and mechanical property variety in UFG titanium subjected to anneal at different temperatures.

2. Experimental procedure

The UFG pure Ti sheets of 0.2mm in thickness were produced by repeat-rolling with a reduction of 97.5% in thickness at 450°C. Then these sheets were isothermally annealed at 300°C, 400°C, 500°C and 600^{o}C respectively for 20 minutes in the VTHK-350L vacuum furnace under 10^{-3} Pa. The heating rate was 8-10^{o}C/min. The samples of 30mm×30mm were cut along rolling direction of plates for microstructural examination and micro-hardness measurement. TEM foils were thinned to about 70-80μm by mechanical grinding, and then electronically thinned with a twin jet electropolisher at 25V and -30^{o}C. TEM examinations were carried on with a JEOL JSM-200CX TEM at 160KV. Texture was determined using an X'Pert PRO XRD equipped with a Cu-Kα ray source. Micro-hardness was measured in Tukon 2100B tester with a load of 20g holden for 10 seconds.

3. Results and discussion

3.1 Microstructure evolution of UFG Ti at different annealing temperatures

The optical microstructure in UFG pure Ti samples subjected to anneal at different temperatures is shown in Fig. 1. The as-rolled deformed microstructure remained (Fig.1(a)) and grain boundaries became obscure when the annealing temperature was below 400^{o}C, as shown in Fig. 1(b) and (c). However, as the annealing temperature increased to above 500^{o}C, equiaxed grains were observed, as shown in Fig. 1(d) and (e). Furthermore, the grain size increased with the annealing temperature. The average grain size increased to 3μm and 7μm, respectively, when the annealing temperature increased to 500^{o}C and 600^{o}C.

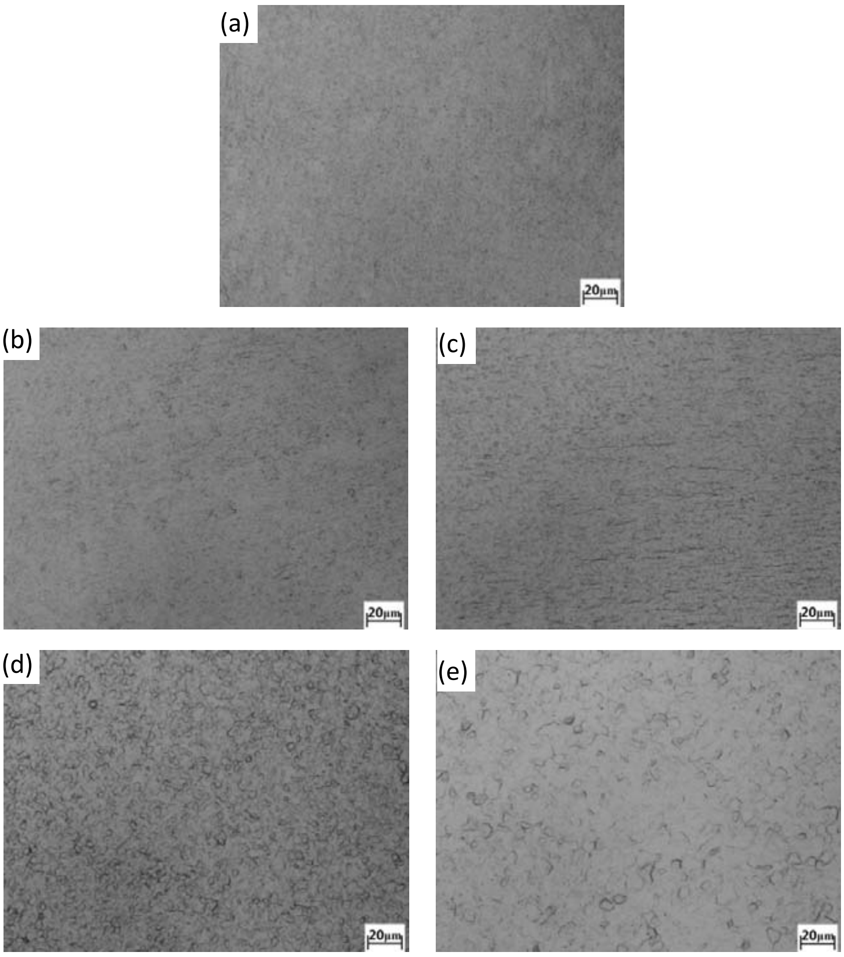

Figure 1. Optical microstructure in UFG Ti samples annealed at different temperatures: (a) as-repeat rolled; (b) 300^{o}C; (c) 400^{o}C; (d) 500^{o}C; (e) 600^{o}C.

TEM examination shows the variety of deformed substructure and grain size with the annealing temperature, as shown in Fig. 2(a) to (e). The ultrafine-grains with a high density of dislocations were produced in the as-rolled samples (Fig.2(a)). The less well-defined grain boundaries and high density of dislocation tangles were the predominant features in the samples subjected to anneal below 400^{o}C, as shown in Fig.2(b) and (c). High density of dislocation cells were formed at 300^{o}C (Fig. 2(b)). Grain boundaries became distinguishable at 400^{o}C (Fig.2(c)). When the annealing temperature was higher than 500^{o}C, well-defined grain boundaries were observed. Most dislocations in the interior of grains disappeared, as shown in Fig.2 (d) and (e). The diameter of grains in the sample annealed at 400^{o}C is larger than that annealed at 300^{o}C. Grain obviously grew and grain size significantly increased as the annealing temperature increased to 500^{o}C and 600^{o}C. The effect of annealing temperature on grain sizes of UFG Ti are summarized in table 1. There is not an obvious increase of grain size under 400^{o}C. It indicates that the deformed microstructure remained stable under 400^{o}C. However, grains apparently grow when the annealing temperature increased to 500^{o}C and 600^{o}C. In other words, microstructural stability was lost over 500^{o}C.

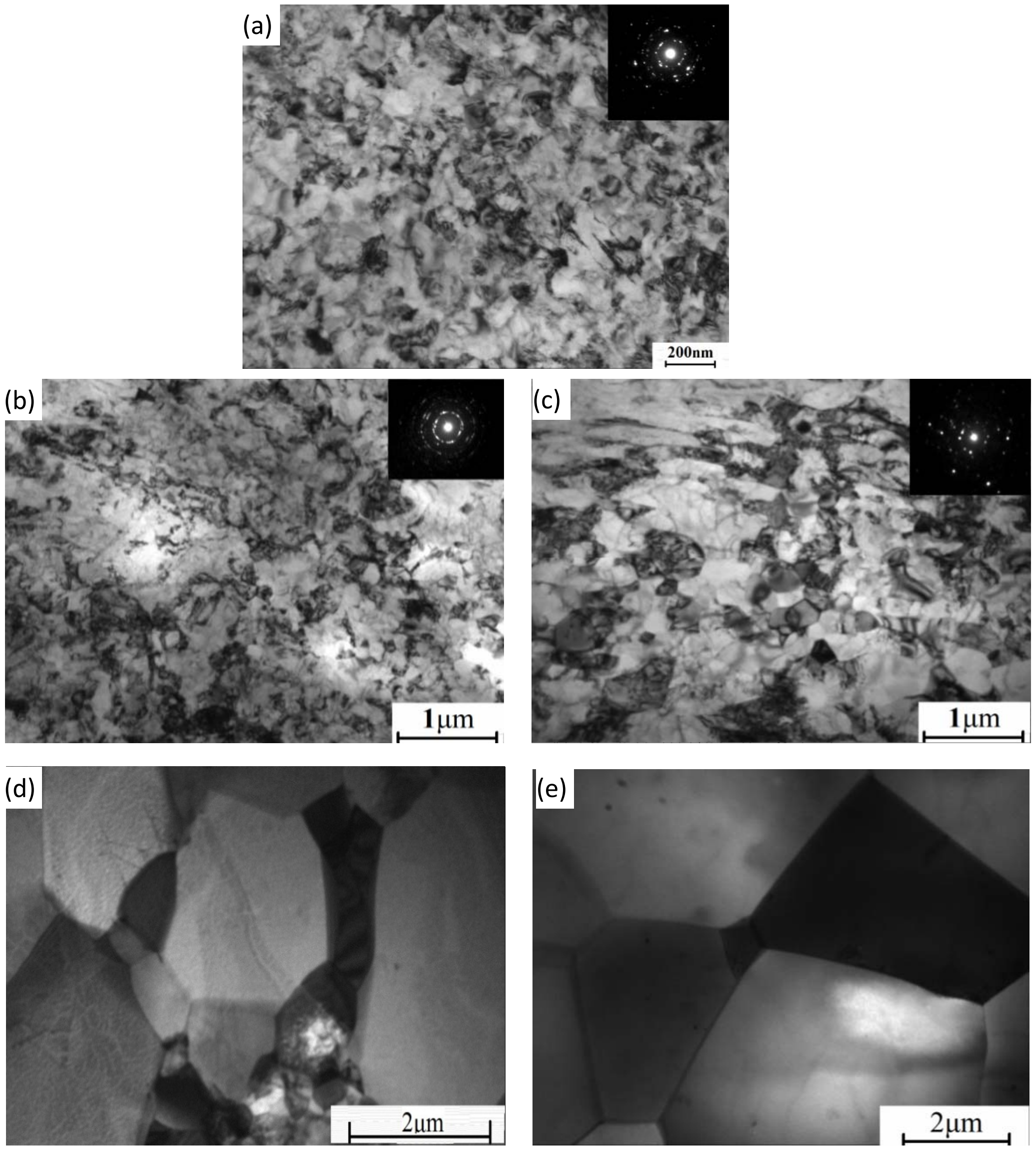

Figure 2 . TEM micrographs in UFG pure titanium annealed at different temperatures: (a) as-repeat rolled; (b) 300^{o}C; (c) 400^{o}C; (d) 500^{o}C; (e) 600^{o}C.

Table 1. The average grain size in the UFG pure titanium samples annealed at different temperatures

Annealing temperature (^oC)	25	300	400	500	600
Average grain size (μm)	0.1	0.2	0.2	3	7

Microstructural observation showed that the average grain size slightly increased when the annealing temperature was less than 400^{o}C. However, the grain size dramatically increased when the annealing temperature was higher than 500^{o}C, as shown in Fig 3. When the annealing temperature is below 400^{o}C, grain size remains constant; however, the distortion and defects in crystal lattice disappear leading to a decrease of the internal stress. When the annealing temperature was higher than 500^{o}C the recrystallization took place in the deformed UFG pure Ti. As a result, the dislocation density remarkably decreased and well-developed grain growth occurred.

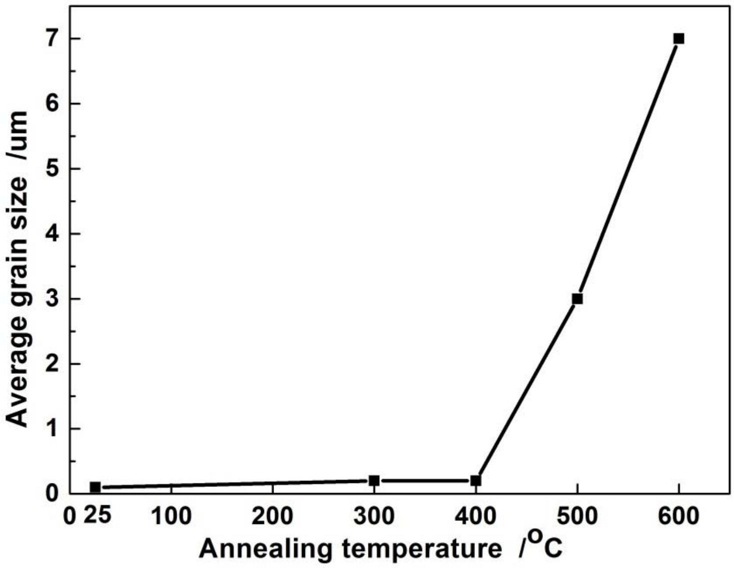

Figure 3. Average grain sizes in UFG pure Ti annealed at different temperatures.

The effect of annealing temperature on texture in UFG titanium is showed in Fig 4. The XRD curve in the initial UFG pure Ti shows that the highest peak is {0002} as shown in Fig 4(a). With increasing annealing temperature to 500°C, the highest peak becomes {10-10}, as shown in Fig 4(b). It means that the lattice rotation took place in UFG titanium during recrystallization.

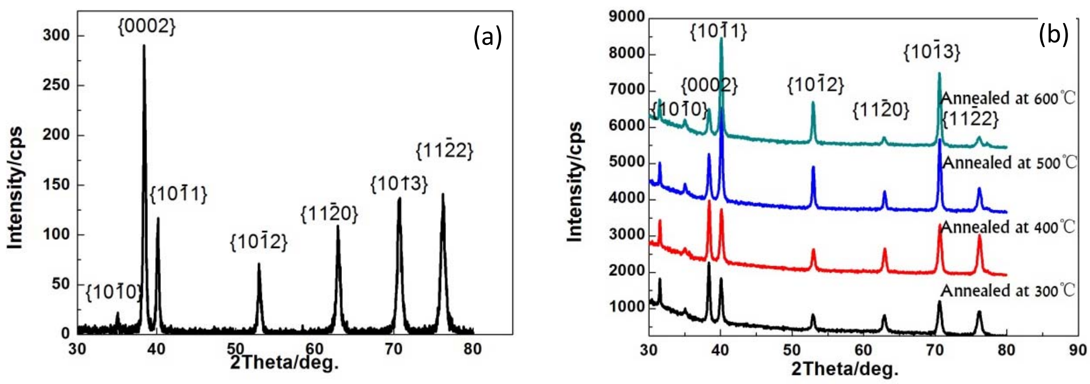

Figure 4. XRD curves of UFG pure titanium annealed at different temperatures

Based on microstructural observation with OM and TEM, it is demonstrated that the microstructure in UFG pure Ti remains a good thermal stability below 400°C.

3.2 Decrease in hardness during annealing

Hardness was measured in the UFG samples annealed at different temperatures, as shown in Fig 5. The hardness almost remained a constant below 300°C. However, the hardness dramatically decreased when the annealing temperature increased to above 400°C, especially 500°C. The hardness of sample annealed at 600°C is close to that of coarse-grained titanium. This means that the stability of mechanical properties could be kept in the UFG pure Ti below 400°C.

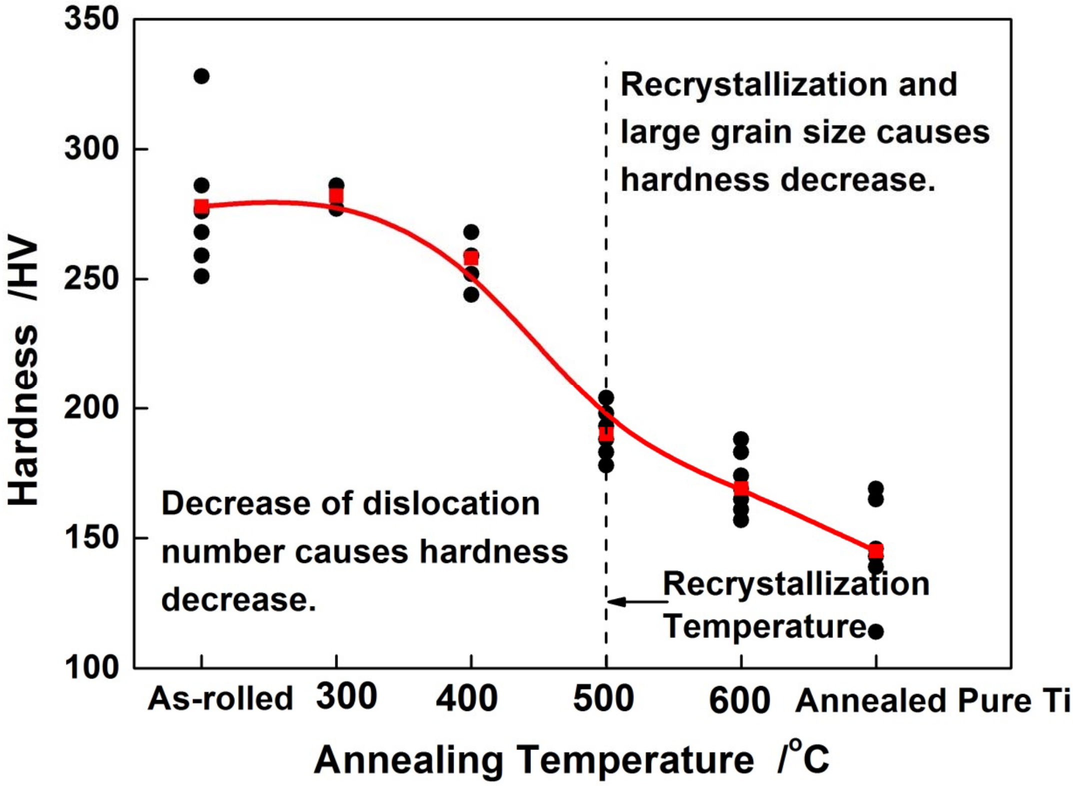

Figure 5. Effect of annealing temperature on micro-hardness in UFG pure titanium

The decrease of hardness in the UFG Ti could be attributed to the following two reasons. When the annealing temperature is below 500°C, the decrease of dislocation density is a predominant factor. While, as the annealing temperature increase to above 500°C, recrystallization and grain growth predominately contributed to the decrease of hardness.

4. Conclusions

1) The average grain size of UFG pure titanium slightly increased when the annealing temperature is lower than 400°C. The microstructure remains stable under 400°C. While the grain size dramatically increased as annealing temperature increased to 500°C or higher. Recrystallization and grain growth took place in ultrafine-grained Ti as annealing temperature is above 500 °C.
2) The rolling texture orientation of UFG pure Ti is {0002} and it disappears when the sample is annealed at 500°C and 600°C.
3) The hardness of UFG Ti slightly decreased when the annealing temperature was lower than

400^{o}C. While, the hardness dramatically decreased as the annealing temperature increased to above 500^{o}C. In former, the decrease of hardness is attributed to the decrease of dislocation density. In the latter, dramatic decrease in hardness results from the recrystallization and grain growth.

Acknowledgements

This project was financially supported by the National Natural Science Foundation of China (51071118, 51271136 and 50831004) the 973 Program of China (2010CB631003), and the 111 Project of China (B06025).

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