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Microstructure refinement of a dual phase titanium alloy by severe room temperature compression

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Abstract: Microstructure refinement of a dual phase titanium alloy, Ti−3Al−4.5V−5Mo, by severe room temperature compression was investigated. Nanocrystalline grains were observed in the sample with 75% reduction, in which the grain sizes of α phase and β phase were approximately 50 and 100 nm. Conversely, the average thicknesses of α phase and β phase in as-received microstructure were measured to be 0.7 and 0.5 μm, respectively. TEM and XRD methods were used to analyze the microstructure and texture changes after severe deformation. Microstructure refinement was deduced to the complex interaction among slip dislocations in the α phase, the complex interaction among slip dislocations and martensites in the β phases. In addition, the interaction between the α phase and the β phase also contributed to the microstructure refinement.

Key words: dual phase titanium alloy; Ti−3Al−4.5V−5Mo alloy; severe plastic deformation; microstructure refinement; nanocrystalline grains; texture

1 Introduction

It has already been established that the material strength increases with the reducing of grain size according to the famous Hall-Petch equation. This trend of thought has led to an invariable interest in producing materials with extremely fine grains. Recently, severe plastic deformation (SPD) techniques, which have attracted increasing attentions in material fields, have been widely used to produce ultrafine grains and nanocrystalline grains materials. Numerous available literatures have described the application of SPD technologies in a serial of pure metals and simple alloys [1,2].

Titanium alloys have been extensively utilized in aerospace and automobile industries due to their low density, high strength, high toughness, good corrosion resistance and fatigue resistance. Grain refinement has continuously been a very important scientific and research topic in titanium alloy fields. However, only micron grain-sized titanium alloys can be got by traditional hot working technologies. Therefore, it should be a very meaningful work to refine titanium alloy materials by new technologies. Some researchers have tried to refine commercially pure titanium (CP-Ti) and Ti−6Al−4V alloy by SPD technologies in order to improve their strength or enhance the feasibility of superplastic processing [3−8]. However, due to the low ductility and difficulty to deform at ambient temperature, the deformation of Ti−6Al−4V alloy is generally conducted at higher temperature and needs more processing turns.

Recently, some heavily stabilized β titanium alloys have been investigated to decrease the grain size by severe cold deformation due to their higher room temperature ductilities. The grain size of Ti−24Nb−4Zr−7.9Sn titanium alloy can be decreased to less than 50 nm after 90% reduction in thickness by rolling, during which highly localized deformation plays an important role in grain refinement [9]. The Ti−25.4Nb−7.1 Ta−1.2In titanium alloy, a less stabilized β titanium alloy with low stacking fault energy, can be severely plastic deformed to nanocrystalline grains in conventional uniaxial compression due to the formation of stress-induced fine α'' martensite [10].

However, few reports could be found to describe the $\alpha+\beta$ typed dual phase titanium alloys by severe cold deformation at ambient temperature due to their limited room temperature ductilities. In this work, we used an $\alpha+\beta$ typed dual phase titanium alloy, Ti−3Al−4.5V−5Mo, which has higher room temperature ductility, to

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investigate the feasibility of producing ultrafine and nanocrystalline grains by conventional room temperature uniaxial compression.

2 Experimental

Ti−3Al−4.5V−5Mo alloy samples with 6.2 mm in diameter used in this investigation were machined from hot rolled and annealed rods. The measured composition of this alloy (in mass fraction, %) was 3.48 Al, 5.23 Mo and 4.68 V. The beta-transus temperature was measured to be 865 °C by metallographic method. Figure 1 shows the typical as-received microstructure of the annealed Ti−3Al−4.5V−5Mo alloy, which is homogeneously composed of lamellar α phase and β phase. The average thicknesses of α phase and β phase in the lamellar structure were measured to be 0.7 and 0.5 μm, respectively. The cylindrical samples for the present compression tests had a length-to-diameter ratio of 2:1. Uniaxial compression deformation was carried out at a constant ram speed of 7.44 mm/min on a Shimadzu testing machine. The reduction ratios in height of 50% and 75% were selected. Microstructure observations were performed using an Axiovert200 MAT optical microscope and Tecnai G^2 20 transmission electron microscope operating at 200 kV. The phase constitutions were detected on a Rigaku D/max−2400PC X-ray diffractometer using Cu K_α radiation, at a voltage of 56 kV and a current of 182 mA. The TEM foils were first mechanically thinned to about 50 μm in thickness and further thinned using a Tenupol-5 twin-jet electrolytic polisher.

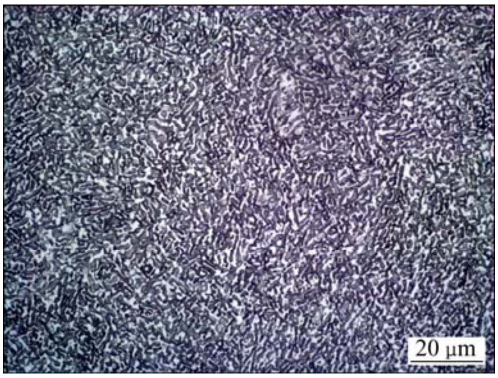

Fig. 1 Optical microstructure micrograph of as-received Ti−3Al−4.5V−5Mo titanium alloy

3 Results and discussion

XRD patterns before and after deformation are presented in Fig. 2. There exists some orthorhombic α'' martensite in patterns of 50% reduction and 75% reduction besides α phase and β phase. This indicates that stress-induced α'' martensite transformation had occurred in β phase during compression deformation. Deformation-induced texture evolution of α phase is measured using the peak intensity ratio $I_{0002}/I_{10\bar{1}0}$. The ratios for the as-received, 50% reduced and 75% reduced samples are 0.23, 4.13 and 7.80, respectively, which indicates a transformation from $(10\bar{1}0)$ plane texture to the (0001) plane texture with increasing in strain level. The ratios of I_{110}/I_{200} in β phase for as-received, 50% reduced and 75% reduced samples are 36.84, 1.58 and 2.36, respectively, demonstrating that (110) plane texture decreases while (200) plane texture increases with increasing the strain level. The observed stress-induced α'' martensite presents a (022) texture. The lattice correspondence between β and α'' phases can be expressed as follows [11]: $[100]_\beta//[100]_{\alpha''}$, $[010]_\beta//[01\bar{1}]_{\alpha''}$ and $[001]_\beta//[011]_{\alpha''}$. Therefore, it could be deduced that the $\{022\}_{\alpha''}$ plane is the principal transform plane when the $\{200\}_\beta$ plane is perpendicular to the compression direction. As shown in the XRD patterns in Fig. 2, the diffraction intensities of α'' martensite decrease when the deformation increases from 50% up to 75% reduction. This may be caused by the α'' martensite reversible transformation backward to the β phase, due to the localization temperature rising. The broadening of the XRD peaks after compression deformation in Fig. 2 indicates that grain refinement occurred.

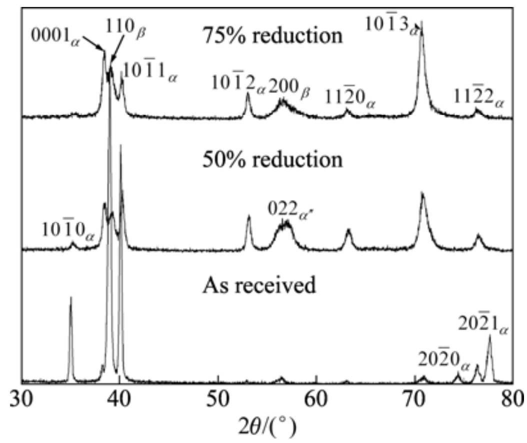

Fig. 2 XRD patterns of as-received, 50% reduced and 75% reduced samples after compression deformation

A homogeneous microstructure of 50% reduced sample is shown in Fig. 3. A great number of grain boundaries in 50% reduced sample are wavy and not well delineated. There are many dislocation tangles and dislocation cells which subdivide α phase and β phase. Especially, the dislocation tangles in β phase are less than those in α phase. The selected area electron diffraction (SAED) patterns of α phase and β phase with 50% reduction deformation are presented in Figs. 3(b) and 3(c). The diffraction spots have elongated into diffraction arcs in both phases, indicating that the presence of low-angle grain boundaries and lattice distortions with high level of internal stress.

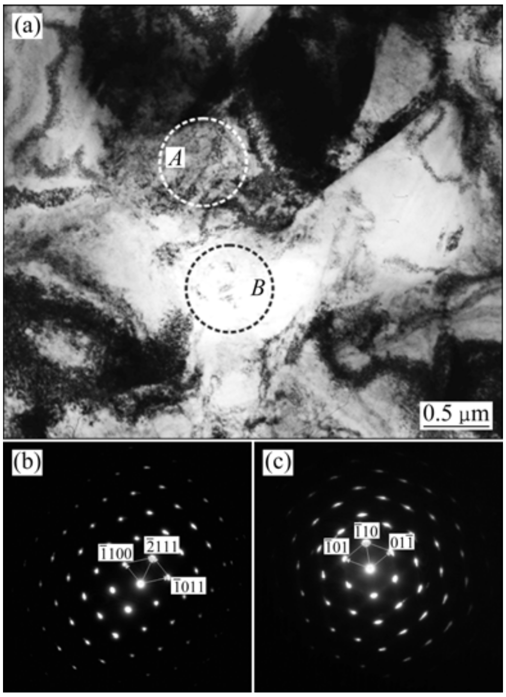

Fig. 3 TEM image of central part of sample after 50% reduction in height (a), SAED patterns taken from dashed circular areas *A* (b) and *B* (c), which are corresponding to *α* phase and *β* phase, respectively

In virtue of limited slip systems in Ti, the deformation twinning has an important influence on the plastic deformation and can effectively refine the grains. However, there was also report that little twin was observed in the largely deformed Ti under 75% reduction in thickness by rolling. The reason is that the deformation twin was suppressed when heavy deformation was imposed. However, no twin was found in the *α* phase in the present experiment. This may be related to that smaller lamellar *α* phase has suppressed the twin formation. For this reason, slip dislocations accommodated the deformation strain, resulting in a deformation substructure of dislocation tangles and cells and subsequently resulting in the formation of subgrains upon increasing strain.

Orthorhombic α'' martensite, which has been identified in Fig. 2, is not observed in the TEM image. This may be because that α'' martensite had transformed to *β* phase during the thinning process of thin-foil TEM specimens due to bulk constraint relaxation. Such explains were reported by other researchers [12,13].

Compared with *β* phase, the strength of *α* phase is relatively low so that it firstly plastically deforms [14,15]. In the initial stage of deformation, dislocation slip would occur in favorable orientation in *α* phase; with the increase in strain level, much more unfavorable *α* phase will rotate to favorable orientation, as shown in the XRD patterns, and then more slip systems could be activated, the dislocations would be multiplied into restricted regions and penetrated into regions as yet substantially free of mobile dislocations; dislocation segments at grain boundaries could be bowed out inside *α* phase, dislocation cells would be formed, and the grain size would be decreased with the increase in strain level.

Interface stress between *α* phase and *β* phase would be induced owing to their different deformation moduli. With the increase in strain level, the interface stress at interface between *α* phase and *β* phase would be increased. This would promote the bowing of dislocation segments and then the decrease in grain size in *α* phase. When sufficient interface stress was accumulated, stress-induced martensite would be formed in *β* phase, and then the previous grains were subdivided. The α'' martensite transformation was the primary deformation mechanism in *β* phase at the initial stage of deformation. With the increase in volume fraction of transformed martensite, induced stress would be raised. When the induced stress exceeded the yield strength of *β* phase, the stress-induced martensite transformation would be finished, and the dislocation slip would be dominant in the subsequent plastic deformation.

The TEM microstructure and SAED of Ti−3Al−4.5V− 5Mo alloy, obtained from the central part of the sample with 75% reduction, are presented in Fig. 4. Pronounced grain refinement is observed, and many grains are evolved into nearly equiaxed nanocrystalline grains with thick cell walls in both *α* phase and *β* phase. However, there are heterogeneous areas consisting of coarser grains. This phenomenon was also reported in severe deformation of other HCP alloys, such as Mg [16] and Zr [17]. Due to few dislocation slip systems in HCP alloys under the low strain and low temperature, some *α* phase in a favorable orientation prefers to deform while other *α* phase with unfavorable orientations can not deform. Therefore, only the favorable regions in the microstructure had been transformed to nanocrystalline grains; on the other hand, other regions had been not effectively refined and remained as coarser grains. Moreover, *β* phase in the favorable orientations had transformed to α'' martensite under the interface stress aiding, and then refined itself to nanocrystalline grains. Such inhomogeneous microstructure was commonly considered a transitional structure, and the coarser grains can rotate to a favorable orientation to deform and then refine to nanocrystalline grains when the deformation strain attains a higher amount.

Figure 4(b) shows the enlarged microstructures of both *α* phase and *β* phase in the nanocrystalline zones, respectively. Original grain boundaries between *α* and

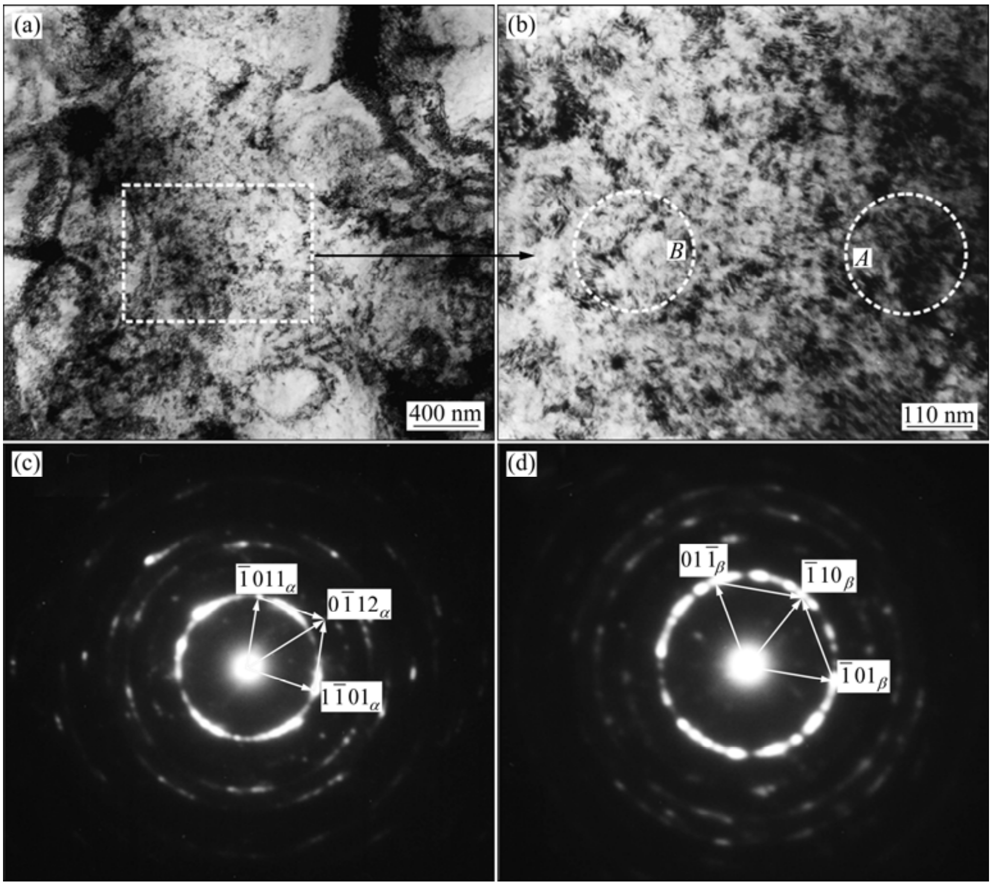

Fig. 4 TEM image of central part of sample after 75% reduction in height (a), an enlarged image of dashed rectangle region (b), and SAED patterns taken from dashed circular areas *A* (c) and *B* (d) (where *A* corresponds to *α* phase and *B* corresponds to *β* phase, respectively)

β phases completely disappear, and the nanocrystalline grains of both phases agglomerate respectively. The grain size of *β* phase is about 100 nm while that of *α* phase is approximately 50 nm. Many grain boundaries of the nanocrystalline grains are not well defined. The SAED patterns for *α* and *β* phases are presented in Figs. 4(c) and (d), respectively. The diffraction spots arranged in circles reveal that large fraction of grain boundaries became high angle. Similar to the nanocrystalline grain in *α*-Ti and *β*-Ti alloys processed by SPD, the microstructure of the dual phase titanium alloy also displayed some complicated moire fringe patterns.

With the increase in the strain level, the dislocation segments at previous formed cell boundaries in *α* phase would continue to bow out, and then new cells would be formed, and the size of cells decreased to nanocrystalline size. Owing to the low-symmetrical orthorhombic structure, α'' martensite is short of sufficient independent slip systems, and then would impede the dislocation in *β* phase to pass through [10]. Thus, the dislocations would intensely interact with α'' martensite in *β* phase and accumulate at the interface between *β* phase and α'' martensite to subdivide the region into nanocrystalline blocks.

4 Conclusions

Nanocrystalline grains were observed in the severe plastic deformed Ti−3Al−4.5V−5Mo alloy. The lower-strength *α* phase with favorable orientation initially plastic deformed by slipping. The interface stress between *α* phase and *β* phase will be induced, owing to their different deformation moduli. When sufficient interface stress was accumulated, α'' martensite transformation occurred in *β* phase; with the increase in strain level, slip would be dominant in subsequent *β* phase plastic deformation. Microstructure refinement to the nanocrystalline size was deduced to the complex interaction among slip dislocations in *α* phases, the complex interaction among slip dislocation, martensites in *β* phase. In addition, the interaction between *α* phase and *β* phase also contributed to the microstructure refinement. These results present an approach for producing full nanocrystalline high performance dual phase titanium alloy using traditional severe compression deformation techniques.

References

[1] VALIEV R Z, ISLAMGALIEV R K, ALEXANDROV I V. Bulk nanostructured materials from severe plastic deformation [J]. Prog Mater Sci, 2000, 45: 103−189.

[2] VALIEV R Z, LANGDON T G. Principle of equal-channel angular pressing as a processing tool for grain refinement [J]. Prog Mater Sci, 2006, 51: 881−981.

[3] ISLAMGALIEV R K, KAZYHANOV V U, SHESTAKOVA L O, SHARAFUTIDINOV A V, VALIEV R Z. Microstructure and mechanical properties of titanium (Grade 4) processed by high-pressure torsion [J]. Mater Sci Eng A, 2008, 493: 190−194.

[4] ZHU Y T, HUANG J Y, GUBICZA J, UNGAR T, WANG Y M, MA E, VALIEV R Z. Nanostructures in Ti processed by severe plastic deformation [J]. J Mater Res, 2003, 18: 1908−1917.

[5] TERADA D, INOUE S, TSUJI N. Microstructure and mechanical properties of commercial purity titanium severely deformed by ARB process [J]. J Mater Sci, 2007, 47: 1673−1681.

[6] MISHRA R S, STOLYAROV V V, ECHER C, VALIEV R Z, MUKHERJEE A K. Mechanical behavior and superplasticity of a severe plastic deformation processed nanocrystalline Ti−6Al−4V alloy [J]. Mater Sci Eng A, 2001, 298: 44−50.

[7] YAPICI G G, KARAMAN I, LUO Z P, RACK H. Microstructure and mechanical properties of severely deformed powder processed Ti−6Al−4V using equal channel angular extrusion [J]. Scripta Mater, 2003, 49: 1021−1027.

[8] SEMENOVA I P, RAAB G I, SAITOVA L R, VAILIE R Z. The effect of equal-channel angular pressing on the structure and mechanical behavior of Ti−6Al−4V alloy [J]. Mater Sci Eng A, 2004, 387−389: 805−808.

[9] HAO Y L, LI S J, SUN S Y, ZHENG C Y, HU Q M, YANG R. Super-elastic titanium alloy with unstable plastic deformation [J]. Appl Phys Lett, 2005, 87: 091906.

[10] XU W, KIM K B, DAS J, CALIN M, RELINGHUAS B, ECKERT J. Deformation-induced nanostructuring in a Ti−Nb−Ta−In alloy [J]. Appl Phys Lett, 2006, 89: 031906.

[11] KIM Y Y, IKEHARA Y, KIM J I, HOSODA H, MIYAZAKI S. Martensitic transformation, shape memory effect and superelasticity of Ti−Nb binary alloys [J]. Acta Mater, 2006, 54: 2419−2429.

[12] SPURLING R A, RHODES C G, WILLIAMS J C. The microstructure of Ti alloys as influenced by thin-foil artifacts [J]. Metall Trans, 1974, 5: 2597−2600.

[13] LUETJERING G, WILLIAMS J C. Titanium [M]. 2nd ed. Heidelberg: Springer-Verlag, 2003: 141−145.

[14] JAWORSKI A, ANKEM S. Influence of the second phase on the room-temperature tensile and creep deformation mechanisms of $\alpha-\beta$ titanium alloys: part I. Tensile deformation [J]. Metall Mater Trans A, 2006, 37: 2739−2754.

[15] LI X W, LU M X, SHA A X, ZHANG L. The tensile deformation behavior of Ti−3Al−4.5V−5Mo titanium alloy [J]. Mater Sci Eng A, 2007, 490: 193−197.

[16] FIGUEIREDO R B, LANGDON T G. Principles of grain refinement and superplastic flow in magnesium alloys processed by ECAP [J]. Mater Sci Eng A, 2009, 501: 105−114.

[17] WANG Y B, LOUIE M, CAO Y, LIAO X Z, LI H J, RINGER S P, ZHU Y T. High-pressure torsion induced microstructural evolution in a hexagonal close-packed Zr alloy [J]. Scripta Mater, 2010, 62: 214−217.

双相钛合金室温大压缩变形下的组织细化

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摘　要：研究了室温大压缩变形下双相 Ti−3Al−4.5V−5Mo 钛合金的组织细化。原始片层组织中 α 相和 β 相的平均宽度分别为 0.7 和 0.5 μm；当压下量达到 75%时，在 α 相和 β 相中均发现了纳米晶，晶粒尺寸分别约为 50 和 100 nm。采用 TEM 和 XRD 对大压缩变形后的显微组织和织构变化进行分析。结果表明，α 相中滑移位错间的交互作用、β 相中滑移位错间及与应力诱发马氏体的复杂作用是显微组织细化的原因。此外，α 相和 β 相之间的相互作用对于晶粒细化有影响。

关键词：双相钛合金；Ti−3Al−4.5V−5Mo 合金；大塑性变形；晶粒细化；纳米晶；织构

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