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Deformation mechanisms in surface nano-crystallization of low elastic modulus Ti6Al4V/Zn composite during severe plastic deformation

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ABSTRACT

Decreasing elastic modulus and simultaneously improving biological compatibility has been a major challenge in developing biomedical alloy. In this study, Ti6Al4V/Zn composite with surface nano crystallization is successfully fabricated by friction stir processing (FSP) with simultaneous addition of beneficial Zn element. Nanoscale microstructures in surface region include nanoscaled Zn rich particles (including $TiZn_2$), α nanograins, nanotwins and nano α + β lamellae. Dislocations slip and grain boundary sliding contribute much to the transformation of α-Ti nanograin, while dynamic overlapping of two partial dislocations is the main deformation mechanism for nanotwins. Interestingly, these nanoscale microstructures reduce the elastic modulus of this region.

As the most typical and frequently used α + β type titanium alloy, Ti-6Al-4V (TC4) alloy has attracted extensive attention in biomedical implant materials field because of its high strength to weight ratio, high corrosion resistance and biocompatibility [1,2]. For bone implants produced from TC4 alloy, exorbitant Young' s modulus and insufficient surface biological compatibility are two main issues, which could reduce their service life. On the one hand, the significant differences between implants and bone in elastic modulus can lead to "stress shielding" [3,4]. On the other hand, surface biological compatibility of the implant can directly affect the early cell adhesion, while cellular adhesion is the primary stage of the interaction of tissue and implants. Thus, as for practical biomedical application, it is highly desirable to obtain combination of lower elastic modulus and better surface biological compatibility of TC4 alloy [5].

In the past few years, extensive efforts have been made to decrease the elastic modulus of Ti alloy [6,7]. Main approaches include modifying microstructures of Ti alloy via alloying β phase stable elements [8], heat treatment and plastic deformation [9] etc. Matsumoto et al. [7] reported alloying Sn into Ti-V alloy could lead to its lattice expansion, thus decreasing elastic modulus of the alloy. While alloying β phase stable elements such as Mo, V, Nb and Ta etc. into Ti is beneficial to the formation of more β phase, leading to the decrease of elastic modulus of Ti alloy [10,11]. Plastic deformation is also an effective method to decrease elastic modulus of Ti alloy. Matsumoto et al. [12] reported that the texture formed after rolling of Ti-8%V can decrease its elastic modulus in the rolling direction. Wang et al. [13] also found that grain refinement of Ti-35Nb-2Ta-3Zr alloy processed by severe plastic deformation can also lower the elastic modulus of the alloy.

Currently, Zn is considered as an indispensable element in human body because it participates in the synthesis of a variety of enzymes, which can promote bone growth and maintain cell physiological function. Furthermore, Zinc oxide displays excellent antimicrobial properties and Zn can also effectively promote osteogenic differentiation of bone marrow stromal cells (BMSCs) [14,15]. It can be predicted that Zn is very promising to improve the biological compatibility of alloy. However, the addition of Zn into titanium alloys through metallurgical processing for implant application has been limited because of significant differences between the melting point of Ti (1660 °C) and the boiling point of Zn (907 °C) [16]. In order to solve this problem, FSP was used to add Zn into TC4 alloy for obtaining surface TC4/Zn composite in this work. On one hand, Zn element can be added into TC4 alloy due to lower peak temperature during FSP. On the other hand, the grains on the surface of TC4/Zn composite can be refined to nanoscale because of the severe friction between FSP tool and materials.

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In this paper, TC4/Zn composite with surface nano-crystallization was successfully fabricated by FSP. The nanoscaled microstructures includes nanoscaled Zn rich particles (including $TiZn_2$), nano α grains, nanotwins and nano α + β lamellae. The nanoscaled microstructures and their deformation mechanisms were systematically studied using transmission electron microscope (TEM) and atom probe tomography (APT) techniques. A new deformation mechanism of nano-microstructures formed on the surface of TC4/Zn composite prepared by FSP is put forward.

TC4 alloy plate with the thickness of 5 mm was used as matrix material. Holes with depth of 1 mm were prepared at a consistent interval of 2 mm. Commercial nano Zn powder with particle size of ~50 nm was used to add into TC4 alloy. After the powder was loaded into holes, FSP was applied on the TC4 plate at the traverse speed (υ) of 100 mm/min and at the rotation rate of 300 rpm. In order to prevent oxidation of the sample, argon gas was used to flow continuously around the tool during whole deformation processing.

A JEM-200 EX transmission electron microscope (TEM) and high resolution TEM (HRTEM) were used to observe the surface microstructures of the TC4/Zn composite. Atom probe tomography (APT) specimen was drawn from upper surface of the composite. Standard metallography techniques were firstly used to mechanically polish the upper surface. Then, the focused ion beam (FIB) lift-out method was used to obtain atom probe needles from the previous polished sample. The APT experiment was performed at Nanjing University of Science and Technology (Nanjing, China) and relative processing parameters were reported in previous investigation [21]. The elastic modulus of the composites were tested by nanoindentation tests using a NANO Indenter G200 Testing System. The maximum loading force and dwell time were 10 mN and 30 S respectively.

Fig. 1 shows the image of Zn rich particles formed in upper surface of TC4/Zn ocomposite (0–150 μm). In Fig. 1(a), nano Zn rich particles with grain size around 10 nm are observed. The selected area electron diffraction (SAED) pattern of the region containing Zn rich particles (Fig. 1 (a) inset) indicates that the Zn rich particles have various crystal structures containing $TiZn_2$ particles. Relatively coarser Zn rich particles with grain size around 50 nm are also found (see Fig. 1(b)). The SAED pattern (Fig. 1(b) inset) proves that the Zn rich particles are $TiZn_2$ and the misorientation angle of 3.6° also reveals the existence of subgrain boundaries in $TiZn_2$ particles. Brice et al. [16] reported that Zn has a slightly larger solubility in Ti-rich side of the Ti-Zn phase diagram. Therefore, during FSP added Zn can react with Ti to form the second phase ($TiZn_2$) or dissolve into the matrix to form solid solution. Due to more severe plastic deformation induced by shoulder drag effect, relatively coarser $TiZn_2$ particles are broken up, which leads to the formation of nano particles with grain size around 10 nm. It should be mentioned that a large number of TEM observations indicate that lots of stacking faults are formed around the $TiZn_2$ particles (as labelled by white arrows in Fig. 1(b) and inset). Fig. 1(c) shows the morphologies of α/$TiZn_2$ interface. It is apparent that there is a transition region between α phase and $TiZn_2$ particles (marked with white dashed circle in Fig. 1 (c)). Fig. 1(d) shows the HRTEM image of transition region in red frame of Fig. 1(c), in which high densities of stacking faults are seen clearly. It suggests that stacking faults are formed during FSP due to higher stress concentration in the α/$TiZn_2$ interface. As the local stress concentration exceeds the critical nucleation stress, twins may nucleate at the grain boundaries and grow into the grain interiors via partial dislocation emission [17,18]. We cautiously speculate that it is due to the gradient variations of Zn content in the transition region. Since Zn element has lower basal plane stacking faults energy, adding Zn into α-Ti can decrease the basal plane stacking faults energy of the whole system.

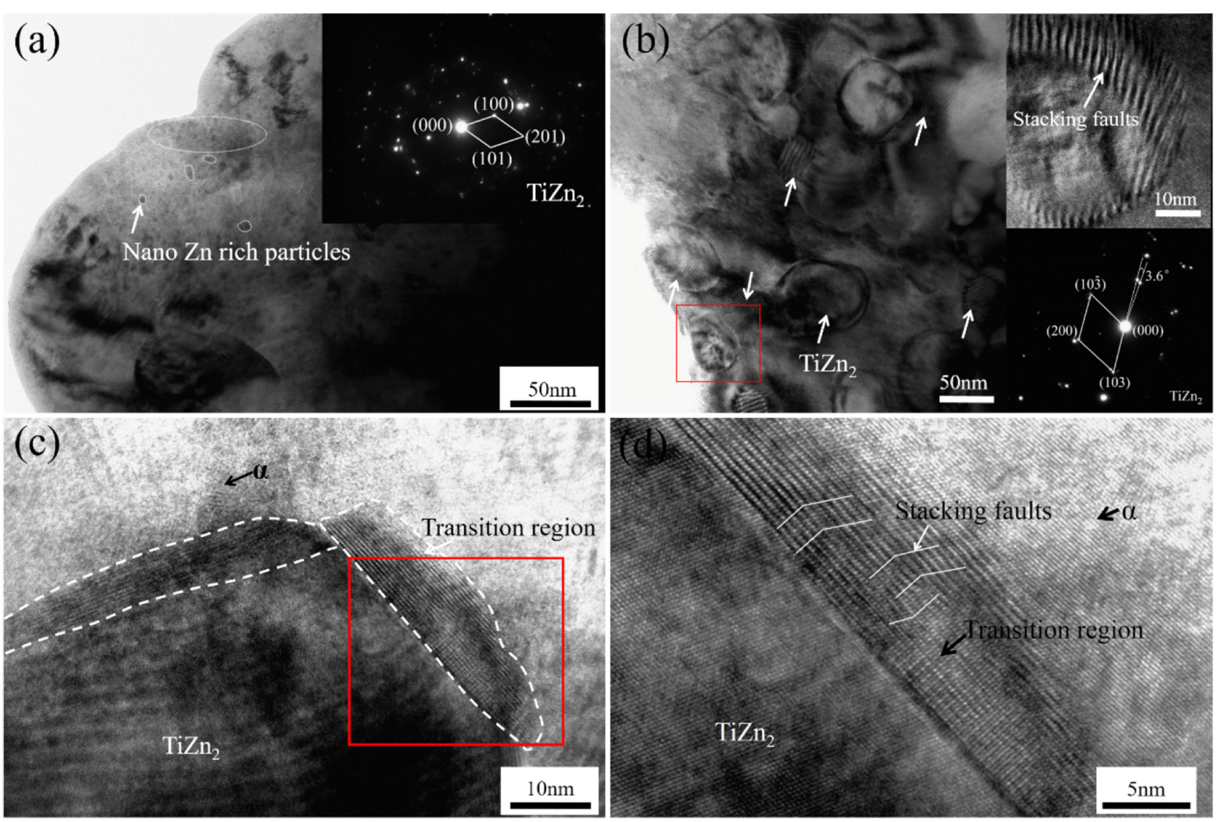

Fig. 1. TEM images of Zn rich particles in TC4/Zn composite: (a) nano Zn rich particles in the upper surface, (b) stacking faults around $TiZn_2$ particles and the inset is magnified image of red frame region in Fig. 1(b), (c) the transition region between α matrix and $TiZn_2$ particles, and (d) HRTEM image corresponding to the white frame region in Fig. 1(c). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)