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Microstructure of titanium component in hydroxyapatite-Ti asymmetrical functionally graded biomaterial ①

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Abstract: The microstructure of the titanium component in hydroxyapatite (HA)-Ti asymmetrical functionally graded biomaterial (FGM) fabricated by powder metallurgical process was investigated. It is found that the main substructure of the Ti component in the FGM consists of screw dislocations whose Burgers vectors are 1/3⟨11$\bar{2}$0⟩ and there are not deformation twins. Screw dislocations are straight and regularly distributed, and cross slip can be observed. The density of the dislocations in the Ti component increases with the rise of the content of the HA component in the FGM. The subgrain boundaries of the Ti component consist of dislocation network walls. Some microbands with bamboo-leaf-shape distribute regularly in Ti grains, which exhibit a specific orientation relationship with α-Ti parent phase.

Key words: functionally graded biomaterial; microstructure; hydroxyapatite; titanium
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1 INTRODUCTION

Hydroxyapatite (HA)-Ti asymmetrical functionally graded biomaterial (FGM) with an asymmetrical compositional profile varying gradually from the Ti side to the HA side, combining the excellent bioactivity of HA ceramic and the high strength of Ti metal, has been developed[1-5]. The fabrication, mechanical properties and the relaxation characteristics of thermal residual stress have also been reported for this material[2-6]. The perfect HA-Ti asymmetrical FGM can be prepared by the thermal stress relaxation design and structure optimization[7].

It is well known that the twinning mode offers an important contribution for the plastic deformation of hcp α-Ti phase under the allotropic transformation temperature (about 882.5 ℃) of α↔β-Ti phase[8,9]. However, HA-Ti asymmetrical FGM was fabricated by powder metallurgical process with hot pressing at 1 100 ℃[7]. Thus, the plastic deformation of the Ti component in the FGM above the allotropic transformation temperature of α↔β-Ti phase could not be avoided. The microstructure of titanium component in HA-Ti asymmetrical FGM fabricated by this method should be investigated since it is very important to the properties of the FGM and has not been reported.

In this paper, the microstructure of titanium component in HA-Ti asymmetrical FGM was investigated by transmission electron microscope (TEM) in detail. In the titanium component of such FGM, an interesting substructure consisting of screw dislocations and microbands distributing regularly is found and there are not deformation twins.

2 EXPERIMENTAL

The raw materials used were titanium powders and HA powders. The chemical composition of titanium was (mass fraction, %): Ti 99.3, Fe 0.039, O 0.35, N 0.035, C 0.025, Cl 0.034, H 0.024 and Si 0.0018. Hydroxyapatite powders were prepared by the reaction between $Ca(NO_3)_2$ and $(NH_4)_2HPO_4$. Its Ca/P ratio was 1.67% ±2.0%. Sizing by means of Laser Particle Sizer (OMEC LS-POP(Ⅲ)) showed Ti particles had an average size of 45.2 μm (93.64% of Ti particles were in the range of 37.0 - 60.0 μm), whereas the average size of HA particles is 1.75 μm (82.12% of HA particles were found to be between 0.35 μm and 3.70 μm). There are significant agglomerations of HA powders shown by scanning electron microscopy (SEM). The starting powders with different HA-Ti mixing ratios were first blended by ball milling for 12 h. Then the mixed powders were stacked layer by layer in a steel die according to the optimized compositional profile[7] and compacted at 200 MPa. Subsequently, the green compacts were hot-pressed at 1 100 ℃ under a pressure of 20 MPa in nitrogen atmosphere for 30 - 90 min with a heating rate of 10 ℃/min and a cooling rate of 6 ℃/min. The perfect HA-Ti asymmetrical FGM was fabricated; it has no residual bending de-

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formation or microcracks parallel to the graded direction of the FGM on the surfaces.

Disk specimens with 0.3 mm in thickness and 3 mm in diameter were cut from the regions of the sintered FGM sample with different HA-Ti mixing ratios, mechanically thinned to 0.1 mm and ion-thinned finally. The resulting specimens were examined in a JEOL-2000EX transmission electron microscope (TEM) equipped with a double-tilt holder and operating at 160 kV.

3 RESULTS AND DISCUSSION

3.1 Dislocation substructure

The typical dislocation substructure in pure titanium graded layer of HA-Ti asymmetrical FGM observed from [0001] direction is shown in Fig. 1(a). Fig. 1(b) is the diffraction pattern of [0001] corresponding to Fig. 1(a). It is found that there are many straight dislocations that lie mainly along $\langle 11\bar{2}0\rangle$ directions and their cross slip phenomena can be observed. Figs. 1(c) and (d) show the dislocation substructures of Ti component in Ti-40% HA and Ti-80% HA (volume fraction) composite graded layers of HA-Ti asymmetrical FGM. Obviously the dislocation configurations of the Ti component in both the composite graded layers are consistent with those in pure titanium layer. However, the density of the dislocations in Ti component increases with the rise of the content of HA ceramic component in graded layers of the FGM, which may be due to the introduction of the additional microscopic stress in the Ti component of the composite graded layers by reason of the elastic mismatches between the HA phase and the Ti one. As shown in Fig. 2, an interesting subgrain boundary consisting of a dislocation network wall with equidistant dislocation lines in the same direction was found in the Ti component of HA-Ti asymmetrical FGM.

The character and Burgers vectors $\boldsymbol{b}$ of straight dislocations lying along $\langle 11\bar{2}0\rangle$ directions could be determined by the condition of dual beam and the invisibility condition of dislocation. It is found that the Burgers vectors $\boldsymbol{b}_1$ of the dislocation along the direction of $[\bar{1}2\bar{1}0]$ should be $1/3[\bar{1}2\bar{1}0]$. Because the Burgers vectors $\boldsymbol{b}_1$ is parallel to the dislocation line, the dislocations along this direction must be screw dislocations. In the same way, it could be determined that the dislocations lying along $[11\bar{2}0]$ and $[\bar{2}110]$ directions must be screw type with the Burgers vectors $\boldsymbol{b}_2$, $1/3[11\bar{2}0]$, and $\boldsymbol{b}_3$, $1/3[\bar{2}110]$ respectively. The cross slip phenomenon shown in Fig. 1(a) is one of the important characteristics that distinguish between screw and edge type dislocations[11].

The screw dislocations have been verified as the main substructure of the Ti component in the

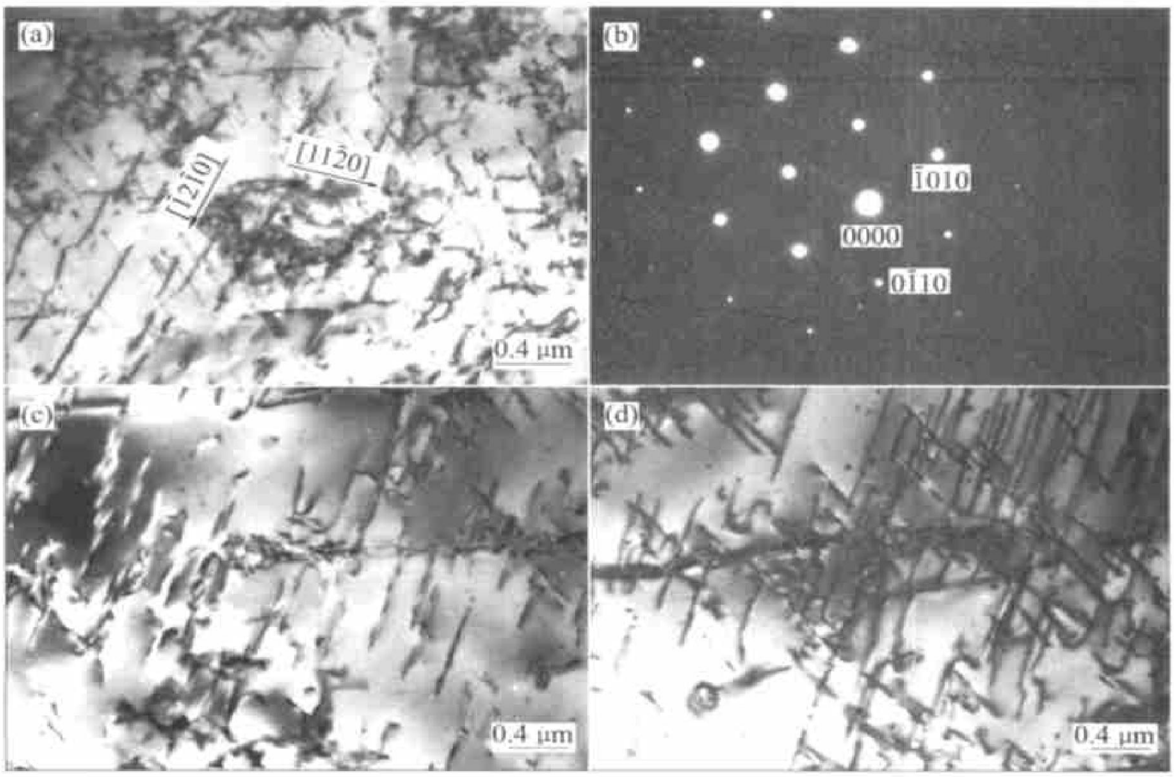

Fig. 1 TEM photographs of dislocation substructure of Ti component in HA-Ti asymmetrical FGM
(a) —Pure titanium graded layer; (b) —Diffraction pattern of [0001] corresponding to (a);
(c) —Ti-40% HA graded layer; (d) —Ti-80% HA graded layer

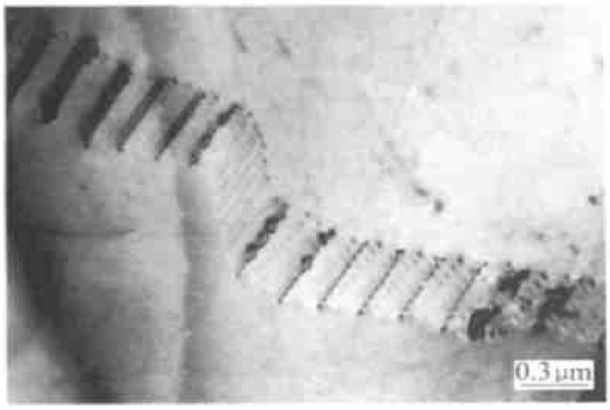

Fig. 2 TEM photograph of subgrain boundary consisting of dislocation network wall with equidistant dislocation lines in same direction in Ti component of HA-Ti asymmetrical FGM

FGM by TEM observation. Thus the slip mode of dislocation is the chief plastic deformation mechanism for the Ti component of the FGM. Plastic deformation of the Ti component in the FGM mainly arises in the high temperature zone of bcc β phase and the screw dislocation is often the chief dislocation configuration which controls the slip characteristics of bcc metals[12], so it is indicated that the screw dislocation configuration above-observed reflects the plastic deformation characteristics of high temperature β-Ti phase. Because it is difficult for the dislocations in bcc metals to form thermo - bending and thermo - folding[12], screw dislocation lines observed present the linear configuration.

3.2 Microband substructure

Figs. 3(a) and (b) show bright-field image and dark-field image of microbands in the titanium component of HA-Ti asymmetrical FGM observed from [0001] direction respectively. Fig. 3(c) is [0001] diffraction pattern of α-Ti phase matrix corresponding to Fig. 3(a). It is found that microbands with bamboo-leaf shape are parallel to each other. Considering the magnetic deflection angle of 189° between diffraction pattern and morphological image, the major axis of microbands is verified to lie along $\langle 11\bar{2}0 \rangle$ directions. Obviously the major axis directions of microbands shown in Fig. 3(a) are corresponding to the slip directions of screw dislocations shown in Fig. 1(a). In fact, screw dislocations could also be found in Fig. 3(a). There are large numbers of dislocations existing at the interfaces between microbands and α-Ti phase matrix. Fig. 3(d) is the composite diffraction pattern taken from the interface between the microband and the α-Ti phase matrix in Fig. 3(a). The following orientation relationship between α-Ti phase matrix and microband (B) could be found:

$$[17\bar{8}\bar{9}]_{\alpha} \parallel [1\bar{2}1\bar{3}]_{B} \quad (1)$$

$$(10\bar{1}1)_{\alpha} \parallel (01\bar{1}\bar{1})_{B} \quad (2)$$

Based on this study, it is difficult to determine

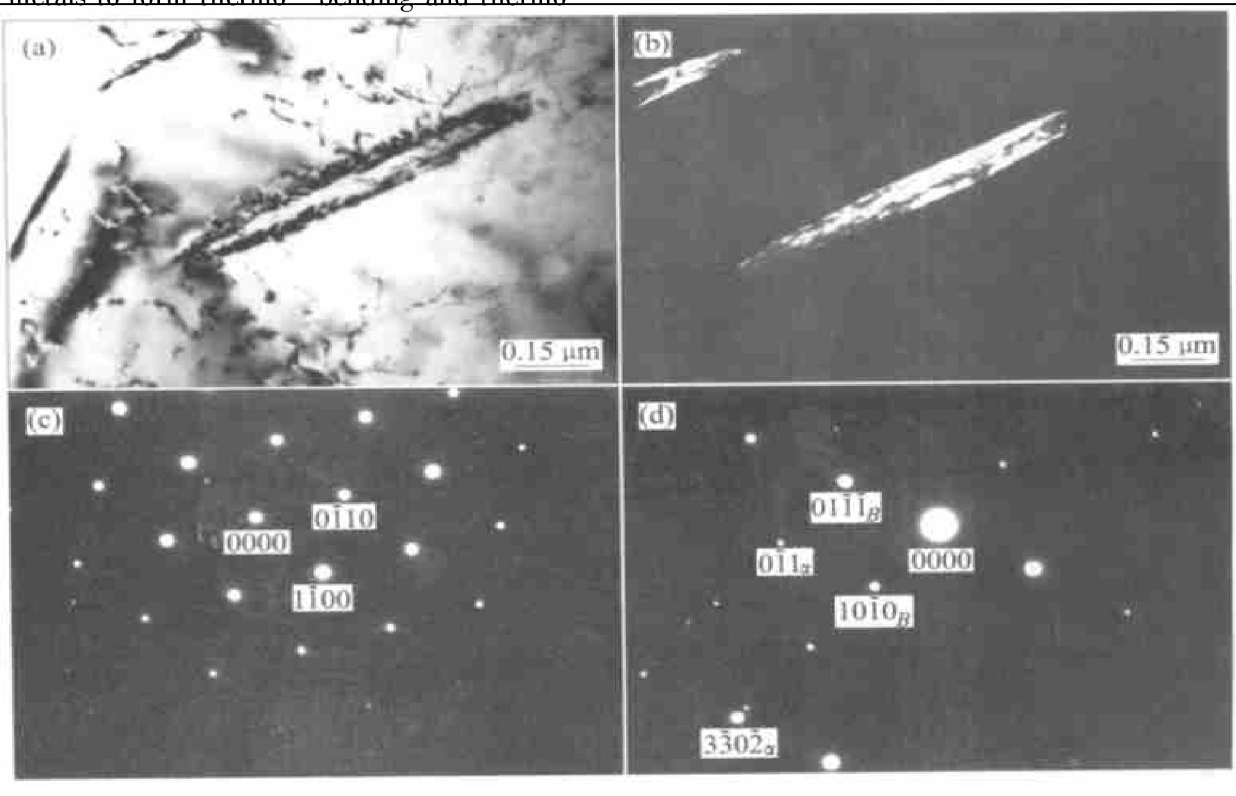

Fig. 3 TEM photographs of microband substructure of Ti component in HA-Ti asymmetrical FGM
(a) and (b) —Bright-field image and dark-field image of microbands observed from [0001] direction respectively;
(c) —[0001] diffraction pattern of α-Ti phase matrix corresponding to (a);
(d) —Composite diffraction pattern taken from interface between microband and α-Ti phase matrix in (a)

the mechanism and conditions for the formation of microbands in the titanium component of the HA-Ti asymmetrical FGM. However, a very good corresponding relationship between microbands and screw dislocations could be found as discussed above, which indicates that the formation mechanism of microbands may be related with the movement of dislocations. In general, the motion of individual dislocation is the translational slip on its slip planes without leading to rotation of the crystal lattice, while the collective motion of dislocation ensembles can lead to local rotations of the substructural elements[13]. Some studies have found that many metals including α-Ti have translational and rotational dislocation substructure simultaneously after plastic deformation under certain conditions, of which the latter is so-called dislocation band substructure[13-15]. Microbands observed in titanium component of HA-Ti asymmetrical FGM may be a special variant of dislocation band substructure. HA-Ti asymmetrical FGM was fabricated by powder metallurgical process with hot pressing at 1 100 ℃ under a pressure of 20 MPa. Therefore, the plastic deformation of the titanium component mainly occurred in the high temperature bcc β-Ti phase region. Large plastic deformation at a high temperature can lead to the formation and the movement of high density of dislocations more easily. As a result, local rotational deformation of the substructural elements in grains occurs, which could lead to the formation of microband finally. Microbands change the periodicity of crystal lattice and can interact with dislocations. Their resistance to the movement of dislocations can lead to an increase of strength and hardness and a decrease of plasticity for the titanium component in HA-Ti asymmetrical FGM.

4 CONCLUSIONS

1) The main substructure of the Ti component in HA-Ti asymmetrical FGM consists of screw dislocations whose Burgers vectors are $1/3\langle 11\bar{2}0\rangle$ and there are not deformation twins. Screw dislocations are straight and regularly distributed, and cross slip can be observed. The density of the dislocations in the Ti component increases with the rise of the content of HA ceramic component in graded layers of the FGM.

2) The subgrain boundaries of the Ti component in HA-Ti asymmetrical FGM consist of dislocation network walls with equidistant dislocation lines in the same direction. Some microbands with bamboo-leaf-shape distribute regularly in Ti grains, which exhibit a specific orientation relationship with α-Ti parent phase.

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