The Ultrafine-Grained Titanium and Biomedical Titanium Alloys Processed by Severe Plastic Deformation (SPD)

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Received: November 08, 2013; Accepted: November 25, 2013; Published: November 29, 2013

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Abstract

The ultrafine-grained materials processed by severe plastic deformation (SPD) can be tailored to achieve superior properties and performances. Recently, the SPD methods, emerging as an effective way to grain refinement, have become attractive for fabrication of the ultrafine-grained biomedical materials, which can be adjusted to possess both favorable mechanical properties and excellent biocompatibility. Biomedical titanium alloys have become one of the most promising biomedical metallic materials, due to their high strength, low density, good biocompatibility and excellent corrosion resistance. Compared with traditional titanium alloys, the ultrafine-grained biomedical titanium alloys possess higher strength, better corrosion resistance and fatigue performance. Moreover, the ultrafine-grained biomedical titanium alloys, which are usually used for orthopedic and dental implants, can induce in-growth of bone tissues, increase the interfacial strength and accelerate the repair process.

This review examines recent developments related to fabricating ultrafine-grained titanium and biomedical titanium alloys by various kinds of SPD methods, such as equal channel angular pressing (ECAP), high pressure torsion (HPT), accumulative roll bonding (ARB) and friction stir processing (FSP). More specifically, the mechanical properties and performances of biomedical titanium alloys processed by ECAP, HPT, ARB and FSP have been investigated. It can be expected that in the near future, these techniques will be utilized as methods for continuous productions of the UFG biomaterials in large scale industrial applications.

Keywords: Severe plastic deformation; Ultrafine-grained; Biomedical titanium alloys

Introduction

The ultrafine-grained titanium and biomedical titanium alloys processed by Severe Plastic Deformation (SPD)

It's well known that biomedical metal materials are widely employed in a various kinds of implants, devices and process equipment that contacts biological systems, which improve

the function of our tissues and organs. Among the biomedical materials, titanium alloys have received attractions for dental and orthopedic implants due to their high strength, great corrosion resistance and biocompatibility, and low density [1,2]. Recently, a series of new β titanium alloys have been developed with low elastic modulus, containing non-toxic alloying elements such as Nb, Ta, Zr and Mo in order to tackle the stress shielding effect caused by stiffness mismatch between implants and bones [3-6].

Several literatures [7-9] have reported that compared with traditional titanium alloys, the ultrafine-grained (UFG) biomedical titanium alloys possess higher strength, better corrosion resistance and fatigue performance. Moreover, the ultrafine-grained biomedical titanium alloys, which are usually used for orthopedic and dental implants, can induce in-growth of bone tissues, increase the interfacial strength and accelerate the repair process. An effective technique to introduce the ultrafine-grained structure is called the Severe Plastic Deformation (SPD).

The Severe Plastic Deformation (SPD) results in significant grain refinement by imposing high plastic strains on metallic materials. For two decades, several SPD methods, such as equal channel angular pressing (ECAP) [10,11], high pressure torsion (HPT) [12,13], accumulative roll bonding (ARB) [14,15] and friction stir processing (FSP) [16,17], have been developed to produce the ultrafine-grained materials. This paper examines recent developments related to fabricating ultrafine-grained biomedical titanium alloys by SPD methods. More specifically, the mechanical properties and performances of biomedical titanium alloys processed by ECAP, HPT, ARB and FSP have been investigated.

Techniques for SPD Methods

Equal channel angular pressing (ECAP)

The ECAP method is the most developed SPD techniques at

present [11]. As illustrated in Figure 1 [18], samples are extruded through a die constrained within an equal channel which is bent at an arbitrary angle. Samples are subjected to severe shear deformation in the channel, leading to grain refinement. The advantage of this process is that large uniform plastic strains can be imposed repeatedly on samples with the unchanged cross-sectional dimensions, which is significantly important for orthopedic implants by the net shape process.

Until now, several researchers have reported the ultrafine-grained CP-Ti and Ti-6Al-4V alloy processed by ECAP. Yang et al. [19,20] employed an die with a intersection angle 120° and route Bc to extrude CP-Ti by multi-pass ECAP. After the first and second pass, deformation twins appeared in the sample. The grains were refined to 200nm after the eighth pass and the microhardness and ultimate strength increased sharply to 2640MPa and 790MPa respectively, while retaining a ductility of 16.8%. Kim et al. [21] systematically investigated the shear deformation mechanisms of CP-Ti pressed at various temperatures from 473K to 873K. It could be concluded that the pressing temperature played a vital role in the deformation mechanisms. The deformed microstructures of ECAP-processed CP-Ti at 473K, 523K-873K, and 873K were thin shear bands (with a width of 0.3 μm), {1011} deformation twinning bands and fine recrystallized grains respectively. For the Ti-6Al-4V alloy, after the multi-passes uniform ultrafine grained structures were formed and the grain size was nearly 200nm and the ultimate strength enhanced to 1510MPa. Furthermore, the ECAP- processed Ti-6Al-4V alloy followed by applied low-temperature deformation contributed to additional enhancement of strength and keeping sufficient ductility [22]. Saitova et al. [23,24] studied fatigue performance and cyclic deformation behavior of the UFG Ti-6Al-4V alloy processed by ECAP. They found that the UFG Ti-6Al-4V alloy exhibited a pronounced improvement in the fatigue life compared to the conventional grain (CG) size counterpart. Moreover, at low plastic strain amplitudes the UFG structure was beneficial to the fatigue behavior of the alloy; when applied at higher plastic strain amplitudes, the UFG microstructure of the Ti-6Al-4V alloy played a detrimental role in fatigue lives due to its reduced ductility.

However, the UFG new β biomedical titanium alloys processed by ECAP have been rarely reported. Xu et al. [25] successfully fabricate the UFG Ti-24.6Nb-5Zr-3Sn β titanium alloys by the ECAP process. The equiaxed β grains (400nm) were obtained

and the authors pointed that β grains pronounced refinement resulted from stress-induced martensite transformation of β phase to α'' phase and its reverse transformation. Our group [18] reported the effects of the pressing temperature and accumulative deformation on microstructure evolution and mechanical properties of the UFG Ti-35Nb-3Zr-2Ta alloy. The results showed that after the fourth pass via route Bc, the ultrafine equiaxed grains of 300nm were obtained and the UFG Ti-35Nb-3Zr-2Ta alloy exhibited a tensile strength of 765MPa, ductility of 16.9% and low elastic modulus of 59GPa, which made it a promising candidate for biomedical implants.

In terms of medical implant applications, evaluations of performances of the biomaterials, such as corrosion resistance and biocompatibility, are of paramount importance. However, relevant literatures about the UFG titanium biomaterials are confined to the UFG CP-Ti and Ti-6Al-4V alloy. Preliminary investigations suggest that the UFG CP-Ti possessed better corrosion resistance than coarse-grained Ti due to the more uniform corrosion [26,27]. On the other hand, some reports have pointed an increased fibroblast cell adhesion and expedite proliferation of pre-osteoblast cells on the UFG CP-Ti [28,29].

High pressure torsion (HPT)

High pressure torsion refers to the processing of metals in which samples are subjected to compressive force and concurrent torsional straining under a high hydrostatic pressure (>2GPa). As shown in Figure 2, samples located between two anvils are imposed on a compressive applied pressure and plastic torsional straining is achieved through rotation of the lower anvil. Surface friction forces make samples shear deformation under a quasi-hydrostatic pressure [13].

Sergueeva et al. [30] obtained the UFG CP-Ti (grain size of

nearly 120nm) by HPT at 5GPa followed by short annealing at low temperatures, which exhibited high strength (>1200MPa) and adequate ductility (>20%). Therefore, it could be concluded that the UFG biomedical titanium alloys with both high strength and great ductility can be fabricated by development of SPD methods with subsequent appropriate heat treatment. Moreover, the UFG TiNi and Ti-6Al-4V alloys processed by ECAP and HPT were also produced [31]. Compared with coarse-grained counterparts, the UFG biomaterials had advantages in tensile strength and fracture toughness, as illustrated in Table 1. Furthermore, grain refinement caused by HPT was more effective than that of ECAP and conventional hot processing. Higher the strength of the UFG alloys by HPT was approximately 500 MPa than that of by ECAP.

Pinheiro et al. [32] reported that HPT proved to be an effective way to produce the UFG Ti-6Al-7Nb alloy, manifested by the increase of micro hardness 78.7% after 5 turns. Meanwhile, the dynamic recovery of the deformed microstructure reached a quasi-equilibrium state, indicating that saturation of the grain refinement was attained and no new defects were formed with higher plastic strain. Yilmazer et al. [33,34] investigated the microstructure of Ti-29Nb-13Ta-4.6Zr β -type alloy processed by HPT and aging treatment (723K 259.2ks). The result showed that a heterogeneous microstructure of matrix and non-etched bands consisting of equiaxed grains and ultrafine elongated grains respectively was formed in the HPT-processed alloy. After 10 turns, the needle-like α phase and small amount ω phase precipitated in ultrafine β grains. The micro hardness in the peripheral region was higher than in the central region. On the other hand, an interesting discovery in the microstructure of the UFG Ti-20 wt.% Mo β titanium alloy was that HPT significantly changed the aging response of the Ti-20 wt.% Mo β titanium alloy, leading to a complete ultrafine-duplex ($\alpha + \beta$) structure in which equiaxed α phase precipitated in equiaxed β grains. This unique microstructure may result from three decisive factors: nanoscale β grains, abundant grain boundaries and enhanced atomic transport [35].

Accumulative roll bonding (ARB)

The technique of accumulative roll bonding (ARB) utilizes a conventional rolling facility. Figure 3 schematically represents

Figure 3: Schematic illustrations showing the principle of the Accumulative Roll Bonding process.

the ARB process. Two sheets surface treated in advance are stacked together, and then the stacked sheets are rolled to one-half thickness of a pre-rolled condition by a traditional roll bonding process. The rolled sheet is cut into two halves which are degreased and wire-brushed before being stacked again. Thus, a series of surface treatment, stacking, rolling, and cutting operations are repeated to achieve a large plastic strain in the sheet. The whole rolling process should be at an elevated temperature where there is no recrystallization to ensure the accumulative strain.

The CP-Ti was processed by ARB process up to eight cycles (equivalent strain of 6.4) at ambient temperature [36]. In ARB-processed samples, two kinds of the UFG microstructures were observed. One was the lamellar boundary structure elongated along rolling direction. The lamellar boundary interval decreased sharply with increasing the accumulative plastic strain; the other was equiaxed grains. Both the grain size reached approximately 80 nm after five cycles. Furthermore, the volume of equiaxed grains went an upward trend with the increase of strain and arrived at 90% after eight cycles. Milner et al [37] hot rolled CP-Ti 7 cycles by ARB at 450 and a predominantly-equiaxed ultrafine grain structure with an average grain size of 100 nm was finally formed. The tensile strength doubled of UFG alloy as the initial one, from 450 to 900 MPa. Furthermore, jump in tensile strength and grain refinement was obtained after the first cycle, indicating that the most effective ARB processing was attained at low cycles.

However, rare investigations on the UFG biomedical β titanium alloys processed by ARB can be seen. Raducanu et al. [14,38] compared the mechanical properties and corrosion resistance of the UFGTi-10Zr-5Nb-5Ta alloy with its as-cast alloy. After three ARB cycles, micro hardness of the ARB processed alloy is higher than that of the as-cast alloy while the elastic modulus was only 46GPa for the ARB processed alloy. With regards to corrosion resistance, lower corrosion and ion release rates, higher polarization resistance for the ARB processed alloy due to the favorable effect of ARB thermo-mechanical processing

Alloy	Processing method	Grain size/ μm	Hv/GPa	YS/MPa	UTS/MPa
CP-Ti	annealing	15	1.8	380	460
	ECAP(+rolling)	0.3(0.1)	2.8(3.2)	640(1020)	710(1150)
	HPT	0.1	3.2	790	950
Ti-6Al-4V	hot rolling	5-10	2.8	900	970
	ECAP	0.5	4.24	1100	1160
	HPT	0.08	5.5	1750	1750
TiNi	quenching	50	2.0	600	940
	ECAP	0.3	2.8	1360	1400
	HPT	30-40	6.0	>2000	>2000

Table 1: [31] Mechanical properties of the alloy in the various processing state.

made it a promising material for bone substitute, as shown in Figure 4 [14]. The Ti-25Nb-3Zr-3Mo-2Sn alloy processed by ARB 4 cycles exhibited ultrafine β grains heavily elongated in the rolling direction and nanocrystalline α phase precipitated on the β grain boundaries, leading to 70% enhancement in the ultimate tensile strength (1220MPa) and double improvement in the 0.5% proof stress (946MPa) [39].

Friction stir processing (FSP)

The FSP technique, based on the principle of friction stir welding (FSW), is an effective solid-state processing method providing localized modification of the surface layers of biomedical materials. As illustrated in Figure 5, a rotating tool with pin and shoulder is inserted in a single piece of materials. The tool heats the samples and localized heating softens the material around the pin and the combination of tool rotation and translation gives rise to the movement of material from the front to the back of the pin [40]. The heats rise from friction between tool and work pieces and plastic deformation of the samples. The advantage of the technique is that we can optionally control the depth of processed zone by adjustment of the length of pin tool.

Figure 4: [14] Potentiodynamic curves for as-cast and ARB processed Ti-10Zr-5Nb-5Ta alloy in the neutral ringer solution at 37°C.

Figure 5: [40] Schematic drawing of friction stir welding.

Figure 6: [42] SEM images taken from stir zone (SZ) in samples: (a) 900 RPM/11IPM and (b) 900RPM/4IPM.

Until now, literatures about the FSP-processed biomedical alloys have been only confined to the TiNi and Ti-6Al-4V alloys. Barcellona et al. [41] investigated the feasibility of the FSP-processed TiNi alloy. The reduction of ductility and shape memory capability was observed, which was strongly influenced by the effective depth of processed zone. By subsequent heat treatment (450, 5mins), the shape memory capability could preserve 62.5% of the base material. Su et al. [42] reported microstructure and mechanical properties of the FSP-processed Ti-6Al-4V alloy. When the tool rotational speed and traverse speed were 900 RPM and 4 IPM, the yield strength and ultimate strength of the UFG Ti-6Al-4V alloy were 1067 and 1156MPa respectively with a ductility of 21.7%. Furthermore, as shown in Figure 6, in the stir zone (SZ) of the FSP-processed alloy, the typical basket-weave lamellar α/β structure was observed, indicating that temperature in the local SZ was above the β -transus temperature. When the tool rotational rate was lower or traverse speed was higher, fine β grains and smaller α colonies were formed, giving rise to higher tensile strength. Also, Atapour et al. [43] compared the corrosion behavior of the as-cast and FSP-processed Ti-6Al-4V alloy (above and below the β -transus temperatures). They concluded that the α/β -FSP Ti-6Al-4V alloy exhibited shorter activation time and higher corrosion rate than the as-cast alloy and the β -FSP alloy.

Summary

Processing through SPD techniques is attractive for the fabrication of the UFG biomedical titanium alloys. These biomaterials can be adjusted to possess both superior mechanical properties and excellent biocompatibility. At the present, the SPD techniques are emerging as methods for production of bulk UFG materials at relatively low costs. And scaling to larger UFG billets is also feasible by SPD methods. In the long term, when continuous production methods are well developed, it is reasonable to anticipate that these techniques will be utilized as methods for continuous productions of the UFG biomaterials in large scale industrial applications.

Acknowledgements

This work is financially supported by the 973 Program under Grant No: 2012CB619600, the Shanghai Natural Science Foundation under Grant No: 12ZR1445500, the New Teachers' Fund for Doctor Stations, Ministry of Education under Grant

No: 20120073120007, the National Science Foundation under Grant No: 81171738, the Medical Engineering Cross Research Foundation of Shanghai Jiaotong University under Grant No: YG2011MS23 and the Excellent Academic Leaders of Shanghai under Grant No: 12XD1402800.

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