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Mechanical and biological behavior of ultrafine-grained Ti alloy aneurysm clip processed using high-pressure torsion



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ABSTRACT

Severe plastic deformation (SPD) has recently been advanced as the main process for fabricating bulk ultrafine grained or nanocrystalline metallic materials, which present much higher strength and better bio-compatibility than coarse-grained counterparts. Medical devices, such as aneurysm clips and dental implants, require high mechanical and biological performance (e.g., stiffness, yield strength, fatigue resistance, and bio-compatibility). These requirements match well the characteristics of SPD-processed materials. Typical aneurysm clips are made of a commercial Ti-6Al-4V alloy, which has higher yield strength than Ti. In this work, Ti and Ti-6Al-4V workpieces were processed by high-pressure torsion (HPT) to enhance their mechanical properties. Tensile tests and hardness tests were performed to evaluate their mechanical properties, and their microstructure was investigated. The hardness and yield stress of the HPT-processed Ti are comparable to those of the initial Ti-6Al-4V due to significantly refined microstructure. Finite element analyses for evaluating the opening performance of a specific geometry of the YASARGIL aneurysm clip were carried out using mechanical properties of the initial and HPT-processed Ti and Ti-6Al-4V. These results indicate that SPD-processed Ti could be a good candidate to substitute for Ti-6Al-4V in aneurysm clips.

1. Introduction

Many industries are developing rapidly these days, particularly medical science and engineering, in response to the unprecedented advance of technology. As medical science advances, new medical devices are needed to perform new medical treatments. These new medical devices still need to be improved regarding size (smaller is better), and compatible with the conditions under which they will be used (Chen and Thouas, 2015; Niinomi, 1998). For example, aneurysm clips and dental implants are usually small but require excellent mechanical performance (e.g., high yield and tensile strength, corrosion and fatigue resistance, and biological compatibility).

The mechanical properties of metallic materials, such as yield strength, hardness, fatigue resistance, and elongation; vary with their chemical composition, microstructure, temperature, and strain rate. Among various processes controlling microstructural features, in particular, grain size, heat treatment is most widely used. Recently, severe plastic deformation (SPD) techniques have been advanced for use in the modification of microstructure. The SPD processes make it possible to produce ultrafine grained (UFG) or nanocrystalline (NC) microstructures out of metallic materials. The strength of SPDprocessed materials is significantly greater than those of the initial materials due to grain refinement and increased dislocation density. It should be noted that grain refinement increases strength, according to the Hall-Petch relation.

The SPD technique has been developed to provide ways to process workpieces of various shapes. Some SPD processes, such as equalchannel angular pressing (ECAP) (Furukawa et al., 2001; Ji et al., 2011; Kim et al., 2014; Quang, 2016; Valiev et al., 2000; Zhu and Lowe, 2000), high-pressure torsion (HPT) (Jiang et al., 2000; Song et al., 2016; Zhilyaev and Langdon, 2008; Zhilyaev et al., 2003), accumulative roll bonding (ARB) (Tsuji et al., 1999), and twist extrusion (TE) (Beygelzimer et al., 2009; Varyukhin et al., 2006) are bulk workpiece processes; whereas, the cone-cone method (CCM) (Bouaziz et al., 2009; Lapovok et al., 2010), high-pressure tube twisting (HPTT) (Lapovok et al., 2010; Toth et al., 2009), and hollow-cone high pressure torsion

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(HC-HPT) (Um et al., 2014) are tube workpiece processes. Both types of SPD processes are used to apply large plastic deformations into the workpieces to manufacture UFG/NC materials. However, the general size of an SPD-processed workpiece is too small to apply for many industrial parts. To solve this problem, it is necessary to approach this problem in two new ways: to develop a new SPD process that can deform a large workpiece, or to apply the SPD-processed workpiece to new fields that require small structural parts, e.g., biomedical devices, which need superior mechanical properties.

This study was focused on solving the problems in conventional aneurysm clips, such as yielding during clip opening and potential health hazards induced by harmful alloying elements in conventional Ti alloy, e.g., vanadium and aluminum in the Ti-6Al-4V alloy. To solve these problems, several essential issues are suggested below,

- To prevent yielding during the clip opening procedure, increased yield strength is needed.
- (2) To increase the closing force, increased Young's modulus is required.
- (3) Proper biocompatibility, corrosion resistance, and purity of the material (without harmful alloying elements) are necessary for implantation into human body.

The requirements for aneurysm clips and dental implants match well with characteristics of SPD-processed materials and the small workpieces best processed by SPD are well-matched to the manufacture of small medical devices. Therefore, application of SPD to production of medical devices would be a good solution to adapt the limitations of SPD methods (Mishnaevsky et al., 2014; Valiev et al., 2008) to current industrial developments.

2. Materials and methods

Inductively coupled plasma (ICP) analysis was performed to determine the composition of the clip and about 6.5% of aluminum and 4.8% of vanadium were detected in result. As shown, the aneurysm clip includes three major elements: Ti, Al, and V; and some minor elements (e.g., Fe). To define each type of alloy, minor elements were ignored and the composition of the major elements, Al and V, were measured. Based on this result, commercial purity grade 2 Ti and Ti-6Al-4V grade 5 were prepared. Both Ti and Ti-6Al-4V alloy were annealed at 800 °C for 1 h and then furnace-cooled to homogenize the microstructure before the HPT process.

Fig. 1(a) represents a schematic of the HPT process. Following this procedure, the HPT process was performed by five revolutions under a compressive force of 48 t of pressure to enhance the mechanical properties of the Ti and Ti-6Al-4V workpieces. This was achieved by refining the grain size of microstructures and homogenizing the microstructures of the HPT-processed workpieces. The compressive force of 48 t corresponds to the average compressive stress of 6 GPa on a disk 10 mm in diameter.

Hardness was measured in the radial direction of the initial and HPT-processed workpieces from the center to the edge part of the disk, using a micro-hardness tester (Future-tech FM-700). A tensile test was performed at a strain rate of 10^{-3} s⁻¹ to measure the tensile behavior of the initial and HPT-processed workpieces using sub-size dog-bone shaped workpieces, a universal testing machine (INSTRON 1361) and digital image correlation methods (Yoon et al., 2016). Optical microscopy (OLYMPUS BX51M) and transmission electron microscopy (JEOL JEM-2100F with Cs-Corrector on STEM) were used to characterize microstructures. Specimens for the tensile test and microstructure observations were prepared from the HPT-processed workpiece, and the measuring locations are shown in Fig. 1(b).

After aneurysm surgery, the installed clips are exposed to two types of environments. The first one is blood vessel cells (biocompatibility) and the second one is cerebrospinal fluid around the brain (corrosion). Although both the HPT-processed and initial Ti alloys show excellent biocompatibility and corrosion resistance, verification tests under the similar conditions, where the aneurysm clips are exposed, need to be performed. A comparison of biocompatibility between the HPT-processed and initial Ti alloys is necessary because proliferation and attachment of the blood vessel cell could proceed to the tong contact part. However, it is hard to acquire the blood vessel cells from aneurysm patients due to safety reasons. Hence, human umbilical vein endothelial cells (HUVECs) was selected. Next, a comparison of corrosion resistance between the HPT-processed and initial Ti alloys is also necessary. Average pH values of cerebrospinal fluid are about 7.3-7.4 (Reed et al., 1967; Siesjö, 1972), where both the HPTprocessed and initial Ti alloys are hardly corroded. Also, dissolved ions, e.g. sodium chloride, could affect the corrosion behavior of the HPT-processed and initial Ti alloys. Cerebrospinal fluid from aneurysm patients is dangerous, and the NaCl solution with medical saline of the similar concentration of mild pH and ion of the cerebrospinal fluid were selected as corrosion media.

A Cu disk was attached to ensure the electrical conductivity of Ti and Ti-6Al-4V alloy, using carbon tapes. To measure the corrosion resistance, 0.9% NaCl water solution (the same as medical saline), was used as a corrosion medium and a polarization scan was initiated from -1.0 to 1.0 V with a scan rate of 0.167 mV/s, using a saturated calomel electrode (SCE). Specimens 20 mm in diameter were prepared for the potentiodynamic test using the HPT process under 2 GPa pressure and ten revolutions. A cell proliferation assay was also performed to characterize biocompatibility and to measure the proliferation of HUVECs on the surface of each specimen. Specimens 10 mm in diameter were prepared using the HPT process under 6 GPa pressure and five revolutions. Proliferation results after 1 to 3 days for the Ti, Ti-6Al-4V alloy, HPT-processed Ti, and HPT-processed Ti-6Al-4V alloy were measured.

The finite element method (FEM) simulations were performed to investigate the deformation behavior of the clip during the opening, using the commercial software ABAQUS ver. 6.9. The geometry of the YASARGIL aneurysm clip was obtained using a 3D scanning device,



Fig. 1. (a) High-pressure torsion procedure and (b) Location of the tensile specimen on the HPT-processed disk.

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