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Journal of the Mechanical Behavior of Biomedical Materials

Research Paper

Flexural and compressive mechanical behaviors of the porous titanium materials with entangled wire structure at different sintering conditions for load-bearing biomedical applications

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ARTICLE INFO

Article history:
Received 4 June 2013
Received in revised form
13 August 2013
Accepted 15 August 2013
Available online 28 August 2013

Keywords:
Entangled titanium materials
Porous titanium
Biomaterials
Implants
Elastic modulus

ABSTRACT

The entangled titanium materials with various porosities have been investigated in terms of the flexural and compressive mechanical properties and the deformation and failure modes. The effect of the sintering parameters on the mechanical properties and the porosity reduction has been comprehensively studied. The results indicate that both the flexural and compressive mechanical properties increase significantly as the porosity decreases. In the porosity range investigated the flexural elastic modulus is in the range of 0.05–6.33 GPa, the flexural strength is in the range of 9.8–324.9 MPa, the compressive elastic modulus is in the range of 0.03–2.25 GPa, and the compressive plateau stress is in the range of 2.3–147.8 MPa. The mechanical properties of the entangled titanium materials can be significantly improved by sintering, which increase remarkably as the sintering temperature and/or the sintering time increases. But on other hand, the sintering process can induce the porosity reduction due to the oxidation on the titanium wire surface.

1. Introduction

Porous titanium materials that can provide space for bone ingrowth, vascularization and nutrient delivery are beneficial for osteoconductive potential and capable of osseointegration with bone when used as implant for bone repair and regeneration (Dolder et al., 2003; Marin et al., 2010). Their mechanical properties such as strength, stiffness, toughness, flexibility, as well as the fatigue lifetime in the body environment are key factors for the surgical implants, which should well and truly imitate the natural bone and have adequate strength to bear the external loading. Unfortunately, the porous titanium materials fabricated by conventional metallurgical methods cannot integrate the accredited strength and the sufficient toughness with the prerequisite porous structure that contributes to the osteoconduction. The powder sintered porous titanium (Oh et al., 2003) not only exhibited very limited toughness, but also its porosity was restricted in a relatively low range. This severely weakened the osteoconduction of the porous titanium. When the H_2O_2 foaming reagent was introduced in the powder

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1751-6161/$ - see front matter
http://dx.doi.org/10.1016/j.jmbbm.2013.08.016

sintering process, the porosity of the porous titanium could be enlarged up to 76% (Chen et al., 2009). But the connectivity of pores was defective; its toughness was also very low, so that the materials showed brittle nature. By using space holder technique (Wen et al., 2001; Garrett et al., 2006) one could adjust the size and shape of pores and the porosity of the porous titanium. Niu et al. (2009) reported that the porous titanium fabricated by this method had about 10 MPa in compressive plateau stress at 75% porosity and about 35 MPa at 55% porosity. A careful investigation of the spacer size on the architecture and mechanical properties of porous titanium (Nihan et al., 2011) revealed that the compressive strength of this material was in the range of 45–90 MPa, and the elastic modulus in the range of 3.8–6.2 GPa at 64% porosity. It was out of question that these materials were brittle because the sintered struts were full of contaminations (e.g., high oxygen content (Nihan et al., 2011)) and defects. In order to make an ideal homogeneous porous structure, Jung et al. (2012) prepared the porous titanium with 3-dimensional periodic macrochannels by machining. The thickness of the titanium wall (or struts) for such structure was larger than that of other porous titanium materials with the same porosity, thus a relatively higher strength and a higher elastic modulus should be expected. However, its compressive properties (e.g., the compressive strength: 100 MPa; elastic modulus: 3.5 GPa at 64% porosity) did not show any superiority compared to other porous titanium produced by using various manufacturing techniques (Nihan et al., 2011; Takemoto et al., 2005; Ryan et al., 2008). Its ductility and toughness were also expected to be low due to its powder sintering processing. In addition, some additive manufacturing techniques (Parthasarathy et al., 2011), such as the rapid prototyping (Ryan et al., 2008; Lopez-Heredia et al., 2008a, 2008b), electron beam melting (EBM) (Parthasarathy et al., 2010), selective laser melting (SLM) (Parthasarathy et al., 2011), direct metal laser sintering (DMLS) (Hussein et al., 2013), and direct 3D printing (Wiria et al., 2010; Xiong et al., 2012; Maleksaeedi et al., 2013), were recently used for fabrication of the porous titanium, by which the porous titanium structure could be designed beforehand by using computer assisted design software. The additive manufacturing process enabled quick fabrication of porous titanium with desired 3D structure by fusing fine titanium powders together layer upon layer. However, the real compressive strength and the elastic modulus of the porous titanium fabricated by the additive manufacturing techniques were only comparable to that of the porous titanium fabricated by the space holder technique (Wen et al., 2001; Garrett et al., 2006; Niu et al., 2009; Nihan et al., 2011), although the macroscopic pore structure could be well controlled in perfect morphology. Because titanium is highly reactive and has an extremely high affinity for oxygen, it is difficult to form defect-free struts during the additive manufacturing process. The contaminations and defects introduced during the laser or electron beam melting must be the predominant factors to deteriorate the mechanical properties. Although the key data (e.g., tensile strength, toughness, impact resistance, fatigue lifetime, etc.) of the porous titanium fabricated by the above additive manufacturing techniques has been infrequent in the literature, it is easy to imagine that the ductility and toughness of the above well-designed porous titanium materials are very poor according to the general metallurgical knowledge.

In order to toughen the porous titanium, the entangled materials with porous structure have been developed recently years (Liu et al., 2010; He et al., 2012), which not only have good balance between the low elastic modulus and the toughness but also contain the interconnected pores through the bulk materials. These characteristics promise their potential applications in orthopedics as implants. Previously, the mechanical behavior of the entangled titanium materials under uniaxial tensile loading was reported (He et al., 2012), which exhibited the yield strength in the range of 24–75 MPa, the ultimate tensile strength in the range of 47.5–108 MPa, and the elastic modulus in the range of 0.33–1.05 GPa when the porosity is in the range of 57.9–44.7%. Compared with other porous titanium (Wen et al., 2001; Oh et al., 2003; Xiong et al., 2012; Maleksaeedi et al., 2013), such materials are very tough under the tensile stress. In addition, the previous work (Liu et al., 2010) demonstrated that the compressive yield strength of such entangled titanium materials was in the range of 2.6–31.1 MPa and the Young's modulus was in the range of 0.14–0.82 GPa (corresponding to the porosity range of 77.6–47.8%). The compressive response of the materials obeyed the typical three-stage stress–strain response which was similar with that of other porous (or cellular) materials (Pippan, 2002). All these available data published are very helpful for the R&D of the porous titanium implants. In this paper, the mechanical behavior of the entangled titanium materials under flexural and compressive loads will be comprehensively investigated. Some relationships between the mechanical properties and the porosity will be established. For practical reference, some detailed flexural and compressive data will also been reported. It is believed that all the findings presented in this paper, together with the data published previously (Liu et al., 2010; He et al., 2012), constitute a complete database that is useful for understanding this new type of the porous titanium materials.

2. Materials and methods

The entangled titanium materials were prepared by using commercial pure titanium (Grade 1) wires with diameters of 0.08 and 0.15 mm, (purchased from Shanghai ZuLi Company, Shanghai, China). The impurities in the titanium wires were chemically determined to be N: 0.02%, O: 0.15%, H: 0.015%, and C: 0.008% (all in weight percent). Their mechanical properties were tested to be 300 MPa yield strength, 370 MPa ultimate tensile strength, and 116 GPa Young's modulus. The fabrication procedure of the entangled titanium materials was described in detail in our previous papers (Tan et al., 2009; Liu et al., 2010; He et al., 2012). Since the mechanical properties of the entangled titanium materials could be significantly influenced by the entangled wire architecture, only the non-woven entangled structure was focused in this study. The green compact porosity of the entangled materials could be directly calculated by using the following formula:

$$P = \left(1 - \frac{M_{Ti}}{V_0 d_{Ti}}\right) \times 100\% \quad (1)$$

where M_{Ti} is weight of the specimen; V_0 is volume of the specimen; d_{Ti} is the density of the titanium wire which is