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Preparation, microstructure and mechanical properties of porous titanium sintered by Ti fibres

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Abstract Open-cell porous Ti with a porosity ranging from 35 to 84% was successfully manufactured by sintering titanium fibres. The microstructure of the porous titanium was observed by SEM and the compressive mechanical properties were tested. By adjusting the spiral structure of the porous titanium, the pore size can be controlled in a range of 150–600 μm. With the increasing of the porosity, compressive yield strength and modulus decrease as predicated. However, high mechanical properties were still obtained at a medium porosity, e.g. the compressive yield strength and the modulus are as high as 100–200 MPa and 3.5–4.2 GPa, respectively, when the porosity is in the range of 50–70%. It was suggested that the porous titanium be strong enough to resist handing during implantation and in vivo loading. It is expected to be used as biocompatible implant, because their interconnected porous structures permit bone tissues ingrowth and the body fluids transportation.

Introduction

Titanium and its alloy have been widely used in the areas of orthopedic and dental implants owing to their excellent biocompatibility [1]. The fixation of titanium alloy implants has been challenged by the mismatch in mechanical properties between the implant and the natural bone, which can severely limit the long-term performance, particularly in load-bearing situations. As one of solutions, porous implant, which permits new bone tissues to ingrow into the interconnected pores structure and form a porous ingrowth (biological) fixation, shows a great potential for the bone implant application [2, 3]. In addition, the porous structure in implant reduces the mechanical properties of titanium implant [4], which makes them more close to that of the natural bone [5]. The design of the porous implant must be on the base of several requirements. First, materials must be biocompatible and bioactive. Second, the pore must be interconnected and the pore size should be in a suitable range, usually 150–600 μm, which allows the ingrowth of cells and the transportation of the body fluids [5–8].

Many processes have been developed to prepare porous titanium, such as slurry foaming [9], powder metallurgy (PM) with space holder [10], plasma spray technique [11]. There exist two types of the pores in porous titanium prepared by these methods. One is macro-pore with the size of 70–600 μm. The other is the micro-pore in the walls of the macro-pores resulted from the volume shrinkages during the sintering process. These micro-pores have the size of microns, and the amount of these micro pores is very large [9, 11, 12]. These micro-pores could induce the formation of micro cracks under the loading condition, which lowers the mechanical properties of porous titanium [13, 14]. The purpose of present study is to develop a method to fabricate porous titanium without micro pores in order to improve the mechanical properties of porous titanium.

Experimental

Commercially available Ti fibres (99.9% purity, 200 μm in diameter, from ShangHai LiZu Company, Shanghai,

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China) were used as raw materials. Figure 1 shows schematically the preparation processing steps of the porous Ti by titanium fibres. In the process, titanium fibres were first curved into helixes lines with special screw pitches and screw diameters. Then, these lines were arranged across in vertical and horizontal directions in a plane. After this, the plane was rolled to form a cylinder and compacted into a cylinder sample. The porosity of the porous titanium was controlled by adjusting the compacting pressure. At last, the compressed cylinder sample was sintered in vacuum atmosphere at 1250°C for 2 h.

The sintered neck and the pore structure of porous titanium samples were observed on Hitachi S–4700 scanning electron microscopy (SEM). The porosities of porous titanium were measured according to the international standard ISO2738 [15, 16]. The pore size distributions were measured by quantitative image analyses using an Image-Pro Plus soft-ware. Specimens with a dimension of Ø13 mm × 15 mm were used for the compression tests. Compression tests were conducted on a servo-hydraulic testing machine. The compressive rate was 1 mm/min, corresponding to a strain rate of 1.0×10^{-3} s^{-1}. An extension meter was used to measure the displacement. In order to measure the Young's modulus accurately, the samples were loaded and unloaded three times before yield. The modulus was determined according to the slop of the linear part of the nominal stress-nominal stain curves. At least five samples were used for compressive testing.

Results and discussions

SEM observation

Open-cell porous Ti with the porosity ranging from 35 to 84% was successfully manufactured by sintering titanium fibres. The scanning electronic micrograph of the porous titanium with the porosity of 67% was shown in Fig. 2.

Figure 3 shows the distribution of pore size. The pore size mainly concentrates in a range of 150–600 μm. A kind of interconnected structure was clearly found. Porosity measurement shows all pores are interconnected, i.e. open porosity is about 100%. The interconnected structure is not regular, which is made up of the spiral structures of the fibres. Cleaning sintering necks with the size in the range of 20–30 μm were found between titanium fibres (see Fig. 4).

It has been reported that the pores which allow the ingrowth of cells must be interconnected and the pore size should be in a suitable range, for example 150–600 μm [6–8, 17]. The pore size in the range of 150–600 μm in the present experiment is suitable for bone ingrowth and the transportation of the body fluids.

Compressive properties

Compressive stress

Figure 5 shows the typical nominal compressive stress-nominal strain curves of the porous titanium with a porosity of 35–84% under a quasi-static compression, which reveals the influence of porosity on the compressive properties of porous titanium.

Two different deformation behaviours were observed. The overall compressive response of the porous titanium with a porosity of 35–67% shows a bilinear behaviour while that of the porous titanium with a porosity of 72–84% follows a smooth power law pattern. This bi-linear behaviour was also observed very evident in a previous study on sintered irons and bronzes with 11–33% porosity [18] and the sintered copper with 5.9% porosity [19]. The power law behaviour was widely observed in porous materials with a high porosity, such as metal form [5]. This seems indicate that the porosity of the porous metal determines the compression behaviour: the porous metal with low porosity seems more likely to have bi-linear behaviours than the porous metal with high porosity. However, no bilinear behaviour was found in porous titanium with 72–84% porosity. As suggested in the previous study [19], the structure of the porous materials, such as the pore shape and the pore size might also plays a very important role in determining the compression behaviours. In Fig. 5, not all porous titanium shows a compressive plateau, so the compress yield stress was determined as showed in Fig. 5 by drawing two tangent lines along the stress-train curve. The stress at the intersection point was considered to be the compressive yield strength. Figure 6 shows the effect of the relative density on the compressive

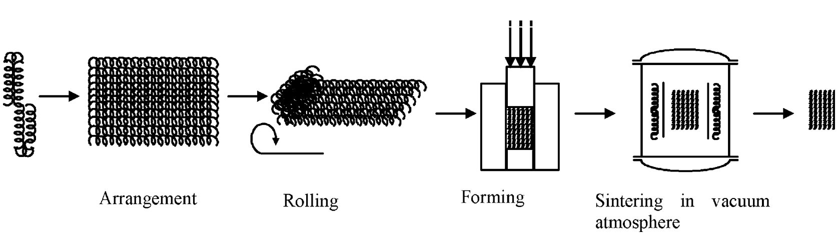

Fig. 1 Processing steps for fabricating porous titanium

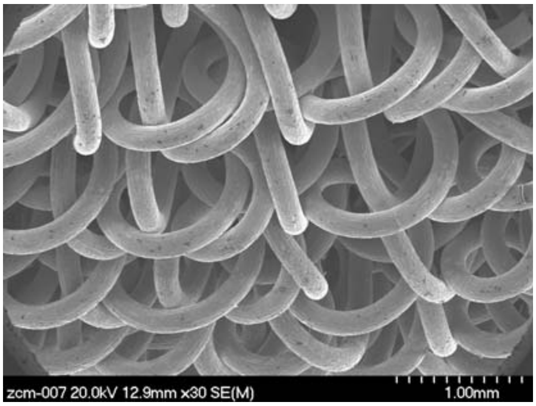

Fig. 2 Micrograph of porous titanium with an interconnected porous structure

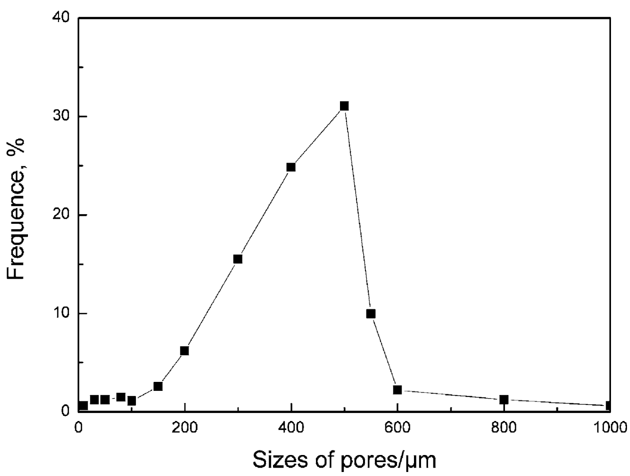

Fig. 3 Pore size distributions of porous titanium fabricated by fibres

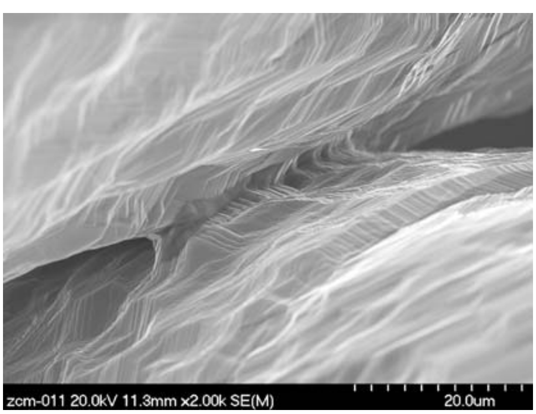

Fig. 4 A sintering neck between titanium fibres

yield strength. It can be seen that the yield strength increases with the increasing of the relative density of the porous titanium.

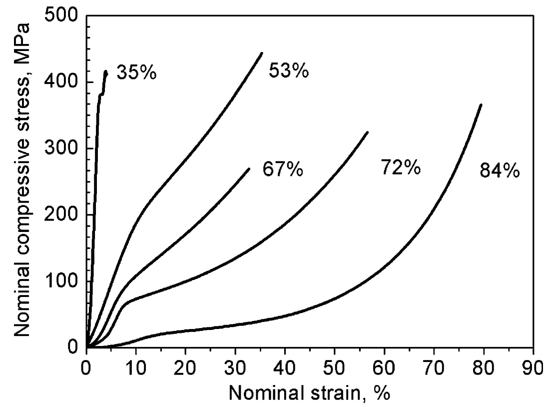

Fig. 5 Compressive stress-strain curves of porous titanium with different porosities

According to Gibson–Ashby beam-model [5], for metals, "plastic collapse model" dominates at all densities. The most important structural characteristic of a cellular material that influences the plastic collapse stress is its relative density, ρ^*/ρ_s (ρ^*-the density of the cellular materials, ρ_s the density of solid material). The relationship between the relative stress and the relative density is given by:

$$\sigma^*_{pl}/\sigma_{ys} = C_1(\rho^*/\rho_s)^{3/2} \quad (1)$$

where σ^*_{pl} and ρ^* are plastic collapse stress and the density of the porous titanium, respectively. The density of solid titanium is 4.503 g/cm^3, and C_1 is a constant which was determined to be 0.3. σ_{ys} is the yield stress of the solid fibre. The tensile yield stress of titanium fibre which was sintered in the same condition to porous titanium was tested to be 172 MPa in this paper. Due to the fact that compressive yield stress of titanium fibre can not be obtained, so the tensile yield stress is used as the compressive yield stress in Eq. 1. And then Eq. 1 can be rewritten as

$$\sigma^*_{pl} = C_1\sigma_{ys}(\rho^*/\rho_s)^{3/2} \quad (2)$$

The calculated results from Eq. 2 were showed in Fig. 6. In the present study, the compressive yield strength is obviously higher than the calculated results from Eq. 2. For example, the yield strengths of the porous titanium with the porosity of 53, 67 and 72% are approximately 200 MPa, 100 MPa and 60 MPa, respectively, while the calculated yield strengths are approximately 108 MPa, 64 MPa and 37 MPa respectively.

As has been pointed in above, not all porous titanium shows a plastic collapse. For the porous titanium with high relative density or low porosity, a bilinear behaviour rather than plastic collapse is observed, indicating that the plastic

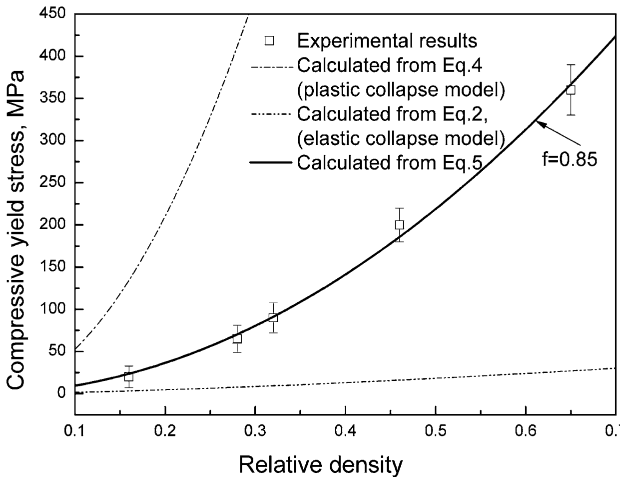

Fig. 6 Effect of the relative density on the compressive yield stress of porous titanium

collapse mode cannot describe completely the compressive behaviour of porous titanium with different porosities. The bilinear behaviour indicates that the elastic collapse also contributes to the compression behaviours to some extent. According to Gibson–Ashby results [5], for a non-linear elasticity mode, the elastic collapse stress can be written as following.

$$\frac{\sigma^*_{el}}{E_s} \approx C_2\left(\frac{\rho^*}{\rho_s}\right)^2 \quad (3)$$

where σ^*_{el} is elastic collapse stress, C_2 is a constant which was determined to be 0.05 by Gibson and Ashby, and E_s is the Young's modulus of the titanium fibre, 103 GPa, which was determined from the tension testing of titanium fibre sintered at the same condition to porous titanium fibre. Then, elastic collapse strength can be described as following

$$\sigma^*_{el} \approx 0.05{*}E_s * \left(\frac{\rho^*}{\rho_s}\right)^2 \quad (4)$$

The calculated results from Eq. 4 is also showed in Fig. 6. It can be found that there is still large difference between experimental results and the calculated results.

From Fig. 6, it can be clearly found that neither the plastic collapse model nor the elastic collapse model can describe completely the compressive behaviour of the porous titanium. Microstructure observation in the above section has shown that the pore size distributes in a large range of 150–600 μm. The inhomogeneous microstructure and the spiral structure of the porous titanium contribute to the large difference between the experimental data and the predicted data in Fig. 6. However, if we combine the elastic collapse model with the plastic collapse model, in the other words, we consider that both the elastic deformation and plastic deformation contribute to the compression, then a new equation can be obtained as following

$$\sigma^*_{cy} \approx fC_1\sigma_{ys}(\rho^*/\rho_s)^{3/2} + (1-f)C_2E_s\left(\frac{\rho^*}{\rho_s}\right)^2 \quad (5)$$

where σ^*_{cy} is compressive yield stress and f is the fraction of the plastic collapse in the compression. When *f* is 0.85, the predicted date fit the experimental very well. The high *f* value of 0.85 indicates that in compression, the plastic collapse plays the most important role and the porous titanium behaves most likely in a plastic collapse model.

Young's modulus

Figure 7 shows the effect of relative density on the compressive Young's modulus of the porous titanium.

It can be found that the modulus increases with respect to the relative density, as observed in foam materials. According to Gibson–Ashby model, the relative density is the most important factor influencing not only the plateau stress but also the young's modulus. For open-cell foam, the Young's modulus E is proportional to the density raised to the second power, which is given by [5]

$$E/E_s = C_1(\rho/\rho_s)^2 \quad (6)$$

where *C* is a constant that approximates 1 for metals. The calculated results were also shown in Fig. 7. It can be seen that there exists a large difference between the calculated modulus and the measured one, especially for the samples with low porosities, although both have the same trend of change: increasing with the increasing of relative density, or decreasing with the increasing of porosities. For examples, the measured Young's modulus with porosity of 53, 67 and 72% are 4.3, 3.5 and 2.0 GPa, respectively, while

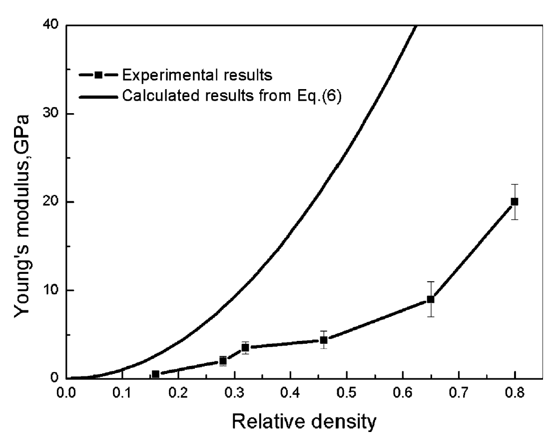

Fig. 7 Effect of the relative density on the Young's modulus of the porous titanium

the calculated Young's modulus according to Eq. 3 are 19, 10, and 8 GPa, respectively. This difference was also found in Wen's researches [20, 21]. There were differences between the Wen's experimental data and the calculated data when the porosities of the titanium foams were smaller than 70%.

Compared with the porous titanium prepared by powder metallurgy, the porous titanium sintered by titanium fibre shows higher open-cell porosity. For example, the porous titanium with lower porosity, such as 53%, is still of complete open-cell structure while the porous titanium prepared by titanium powder with the porosity of 80% was not complete open-cell structure because of isolated micropores in the pore wall [20, 21]. Also the porous titanium prepared by sintering titanium fibres shows relative high compressive yield strength. For example, the compressive yield strengths of the porous titanium with the porosity of 53, 67 and 72% are approximately 200, 100 and 60 MPa, respectively, while the compressive yield strengths of porous titanium with 60 and 77% porosity prepared by powder metallurgy (sintered at 1400°C for 1 h) are about 70 and 6 MPa, respectively [22]. In addition, preparing porous titanium by sintering titanium fibres avoids the use of space holder which was used in powder metallurgy, and in turn eliminates the contamination produced by the space holder [22, 23].

In practice, the relative density of cancellous bone varies from 0.05 to 0.7, i. e. porosity of bone varies from 30 to 95% (technically, any bone with a relative density less than 0.7 is classified as 'cancellous'). Pore size of the cancellous bone varies from 20 to 1000 μm, and the pore structure is usually open. Cancellous bone has the compressive stress of 20–193 MPa and the Young's modulus of 2.3–20 GPa [5]. The process of sintering titanium fibres can produce porous titanium with similar mechanical properties, pore structures and porosities to those of natural cancellous bone. The compressive yield strength and Young's modulus of porous titanium with the porosity of 53–72% in this study range from 200 to 60 MPa and from 4.3 to 2.0 GPa, respectively, which is strong enough to resist handing during implantation and in vivo loading. This porous titanium is expected to be used as biomaterials, because open-cell structure with the pore size of 150–600 μm permits the ingrowths of new-bone tissue and transport of body fluids.

Conclusions

Open-cell porous titanium with a porosity of 35–84% and a pore size of 150–600 μm has been successfully manufactured by sintering titanium fibres. The compressive yield strength and Young's modulus of porous titanium range from 60 to 200 MPa and from 2.0 to 4.3 GPa, respectively. It was suggested this porous titanium should be strong enough to resist handing during implantation and in vivo loading and be expected to be used as biocompatible implant.

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