

Effect of sintering neck on compressive mechanical properties of porous titanium

ZOU Chun-ming¹, LIU Yan², YANG Xin¹, WANG Hong-wei¹, WEI Zun-jie¹

1. School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China;

2. Key Laboratory for Bionics Engineering, Ministry of Education, Jilin University, Changchun 130022, China

Received 28 August 2012; accepted 25 October 2012

Abstract: In order to study the role of sintering neck in porous titanium with helical pores, the effect of size and position of sintering neck on compressive mechanical properties of porous titanium single cell was studied by using numerical simulation method. The results show that the compressive mechanical properties of the porous titanium unit cell are determined by the helical pore structure and sintering neck. Contribution coefficient of sintering neck is approximately 3.5 times larger than that of helical pore structure. With the increase of the relative diameter of sintering neck, compressive yield stress and elastic modulus of the cell are constantly increased. The sintering point of C1 is the most important sintering position. Under the same condition, increasing the size of sintering neck at C1 is much effective to the increasing of compressive properties.

Key words: porous titanium; helical pore structure; single cell; sintering neck; compressive mechanical properties; contribution coefficient

1 Introduction

With their outstanding comprehensive performance, titanium and its alloys are applied to hard tissue medical materials such as artificial bone, artificial joints and teeth [1–3]. Among them, the introduction of pore structure of implant helps to reduce and even eliminate the phenomenon of “stress shielding”, improving the mechanical compatibility of the implants [4,5].

Many new researches have focused on the effect of porosity, pore size and distribution on the properties [6–12]. ZOU et al [9–12] proposed a new porous titanium sintered by titanium wire with helical pore structure. Its preparation process included regularly arranging the spiral coil, rolling the arranged helix network, forming in mold and sintering in vacuum atmosphere [12]. After surface bioactivation, it achieved some promising results from biological experiments, making it potential for practical application [10–12].

For porous materials, such as sintered powder materials, metal foams and entangled wire materials, the general theory believes that the mechanical properties of porous materials are mainly influenced by the porosity

[13–17]. The research of compressive mechanical properties of the porous titanium sintered by titanium wire also verifies this point [5]. In addition, studies on porous materials sintered by metal powder found that the sintering temperature, time and pressure also directly affect the mechanical properties [18–20]. In general, the increase of the sintering temperature, the sintering time or the sintering pressure will increase the size of sintering neck, which help to improve the mechanical properties of the porous materials when increasing the size of the sintering necks [18–22]. Therefore, the sintering neck will be another important factor to the mechanical properties.

For porous titanium sintered by titanium wire with helical pore structure, the sintering process was used for the connection of titanium wire [9–12]. So it can be expected that the sintering neck will be an important factor to the mechanical properties. In fact, it has been recognized that wire sintering process is difficult, so a layer of easily sintered metal is coated on the steel wire surface so as to ensure the sintering point on the quality and morphology [23]. However, when evaluating the contribution of sintering point on the mechanical properties, the friction behavior between wires causes the

interference, which makes the quantitative evaluation of the contribution of sintering point difficult extremely. The porous titanium with helical pores also faces the same problem [6]. So, in the present work, the typical single cell of porous titanium will be used to evaluate the contribution of sintering point by numerical simulation method, which is convenient for eliminating the friction interference.

2 Numerical simulation process

ANSYS Workbench was selected as an analytical environment which is one of the modules of the finite element software ANSYS. The main analysis progress of the numerical simulation can be briefly summarized as geometric modeling, the materials data setting, meshing, loading setting and results analysis.

Figure 1 shows the actually prepared porous titanium whose preparation process was introduced in Ref. [9]. It can be seen that the porous titanium is regularly arranged by a number of same cells which has a certain structure.

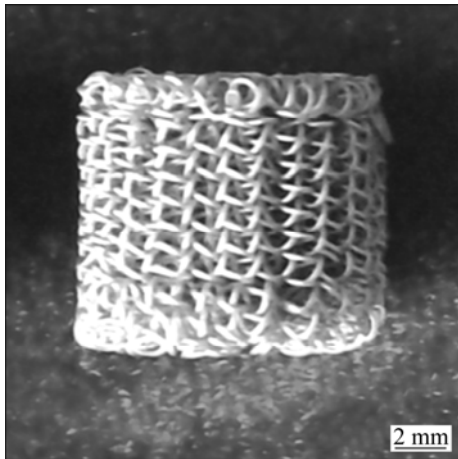


Fig. 1 Porous titanium sintered by titanium wires

Through decomposing and abstracting the structure of porous titanium sintered by titanium wire, a typical cell from one layer of porous titanium was selected. Pro-E was used for geometric modeling in order to improve the efficiency and quality of the numerical simulation, as shown in Fig. 2. There are three sintering necks in the cell, which are labeled as C1, C2 and C3 respectively. The benchmark dimensions of the cell in Fig. 2 are defined as follows. The diameter of titanium wire is d_{Ti} , the pitch diameter of spiral is D_2 , and the pitch of the spiral is t . The edge length of cell is defined as D_{st} , and $D_{st} = D_2 = t$. And the diameter of the sintering neck is d_s .

According to the tensile experimental result of titanium wire, parameters from true stress—strain curve of the titanium wire were selected for calculating and

setting the material parameters as shown in Table 1. On the plastic deformation stage, the “multi-linear” was chosen to define the material nonlinearity, as shown in Table 2.

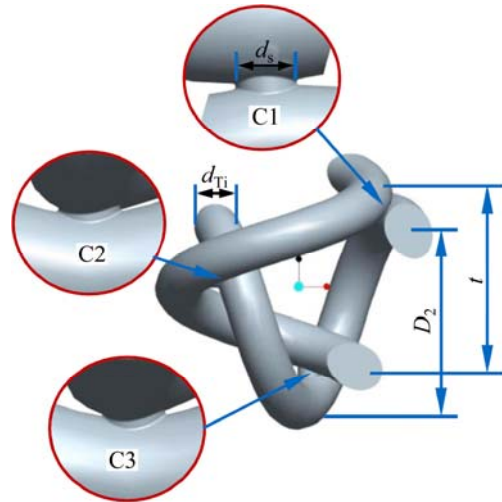


Fig. 2 Typical cell of porous titanium

Table 1 Mechanical properties of titanium wire

Parameter	Value
Elastic modulus, E/GPa	74.5
Poisson ratio, ν	0.33
Yield strength, σ_s/MPa	312
Ultimate strength, σ_b/MPa	473
Elongation, δ	22.1%

Table 2 Correspondence of plastic strain and stress of titanium wire

Plastic strain	Stress/MPa	Plastic strain	Stress/MPa
0	312.00	8	412.71
1	347.58	10	423.72
2	362.29	16	456.92
4	383.47	21.6	473.00
6	399.70		

The grid size of loading device was set at $80\text{ }\mu\text{m}$ and the grid of the cell was set at $20\text{ }\mu\text{m}$. The three sintering necks of the cell were further refined with the grid size of $5\text{ }\mu\text{m}$.

In order to approach to the real force status, the cell was placed in a loading device made by a single layer of porous titanium. Then compressive load was added to the loading device, as shown in Fig. 3.

3 Compressive performance

3.1 Compressive stress—strain curve

Figure 4 shows the stress—strain curves of porous titanium cell ($D_{st}=800\text{ }\mu\text{m}$, $d_{Ti}=200\text{ }\mu\text{m}$) under uniaxial

compressive load. It can be known that the compressive yield stress ($\sigma_{0.2}$) of the unit cell of the porous titanium is about 1.1 MPa, and the corresponding strain value is 1.3%.

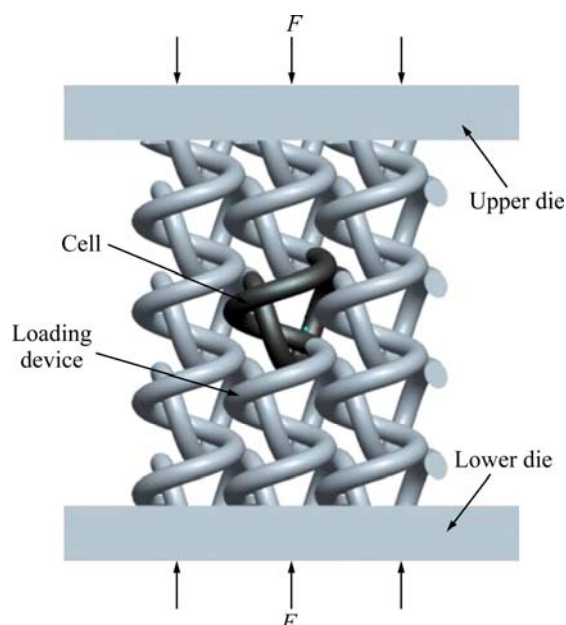


Fig. 3 Loading device of porous titanium cell

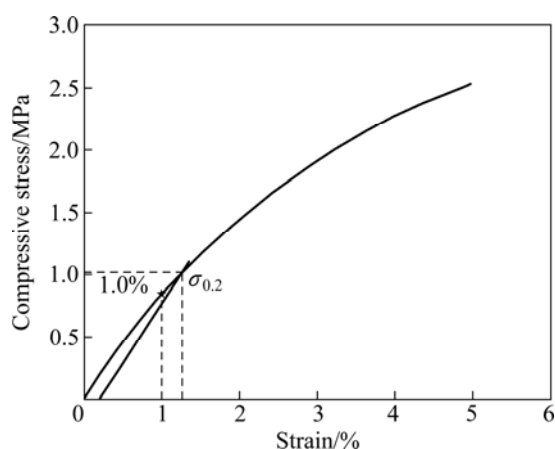


Fig. 4 Stress—strain curves of porous titanium cell under uniaxial compressive load

3.2 Stress distribution

Figure 5 shows the stress distribution of the cell when the strain value is 1.0%. It can be seen that the maximum equivalent stress is mainly concentrated in the three sintering necks marked C1, C2 and C3, especially in the sintering neck C1. The equivalent stress in the sintering neck C1 firstly exceeds the yield stress, but does not exceed the limit stress.

It can be expected that the sintering neck will be firstly destroyed when the deformation reaches a certain value. It shows that the strength of the sintering neck will play an important role in the mechanical properties of porous titanium and that enhancing the strength of the

sintering neck will greatly improve the strength of the porous titanium.

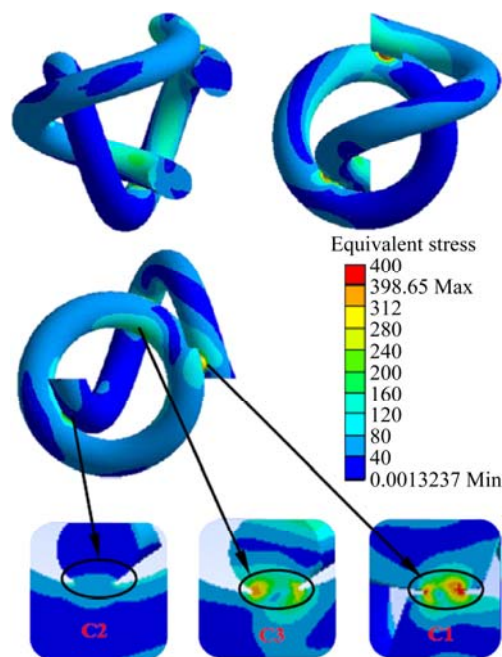


Fig. 5 Equivalent stress distribution of cell and different sintering neck section when strain value is 1.0%

4 Mechanical properties

Since the compressive properties of the porous titanium cell are determined by the relative structure, the concept of relative size is required for the impact of the sintering neck. Therefore, the relative sintering neck diameter is defined as d_{sr} . And its expression is shown as

$$d_{sr} = \frac{d_s}{d_{Ti}} \quad (1)$$

where d_{sr} is the relative diameter of the sintering neck, d_s is the actual diameter of the sintering neck and d_{Ti} is the diameter of titanium wire.

4.1 Effect of relative diameter of sintering neck

There are two ways to change the relative diameter of sintering neck and to remain the porosity of porous titanium cell as 74% at the same time. One way is to keep cell structure as the reference size ($D_{st}=800 \mu\text{m}$, $d_{Ti}=200 \mu\text{m}$). The diameter of sintering neck is set at 50 μm , 75 μm , 100 μm , 125 μm and 150 μm , respectively, thus the relative sintering neck diameter is 0.250, 0.375, 0.500, 0.625 and 0.750, accordingly. Figure 6 shows the compressive properties of the porous titanium cell with different diameters of sintering neck.

The other way is to fix the diameter of sintering neck as 100 μm while scaling the edge length of cell and the diameter of the titanium wire at the same time. Five cases are set as follows: $D_{st}=600 \mu\text{m}$, $d_{Ti}=150 \mu\text{m}$;

$D_{st}=700\text{ }\mu\text{m}$, $d_{Ti}=175\text{ }\mu\text{m}$; $D_{st}=800\text{ }\mu\text{m}$, $d_{Ti}=200\text{ }\mu\text{m}$; $D_{st}=900\text{ }\mu\text{m}$, $d_{Ti}=225\text{ }\mu\text{m}$ and $D_{st}=1000\text{ }\mu\text{m}$, $d_{Ti}=250\text{ }\mu\text{m}$. And the relative diameters of the sintering neck are 0.667, 0.571, 0.500, 0.444 and 0.4, accordingly. Figure 7 shows the compressive mechanical properties of the porous titanium cell while scaling the size of cell.

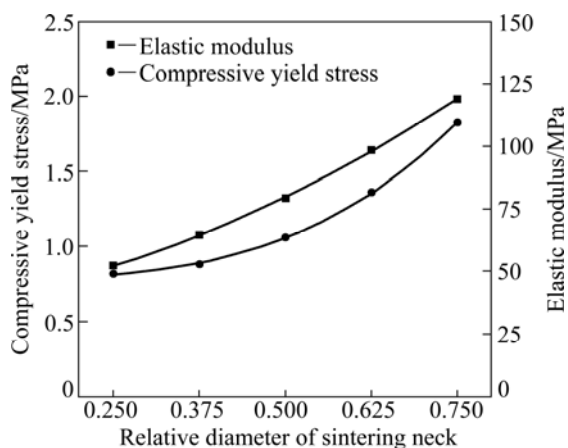


Fig. 6 Compressive mechanical properties of porous titanium cell with different diameters of sintering neck

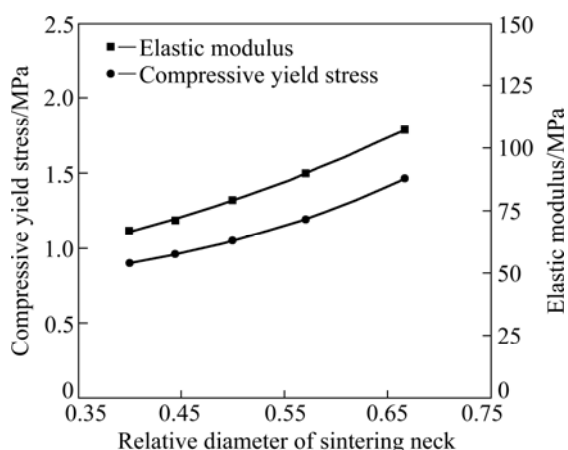


Fig. 7 Compressive mechanical properties of porous titanium cell with different diameters of sintering neck by scaling the size of cell

It can be seen that the relative diameter of the sintering neck has a huge impact on the compressive mechanical properties of porous titanium cell. Specifically, with the increase of the relative diameter, compressive yield stress and elastic modulus of the cell both constantly increase.

Since the influence of the sintering neck mainly concentrates on the relative diameter of the sintering neck, the two situations in Figs. 6 and 7 are combined to find the relationship between the compressive mechanical properties of porous titanium cell and the relative diameter of sintering neck. Figure 8 shows the fitting of compressive yield stress of porous titanium cell with different relative diameter of sintering neck. It is

obvious that the results of those two situations just fit the same curve, which means the effect of the relative diameter of the sintering neck on compressive yield strength obeys the same law. Specifically, with the increase of the relative diameter of sintering neck, the compressive yield strength increases significantly.

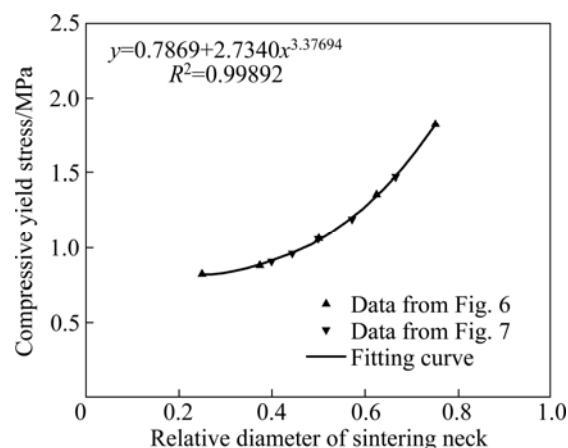


Fig. 8 Fitting of compressive yield stress of porous titanium cell with different relative diameter of sintering neck

Figure 9 shows the fitting of elastic modulus of porous titanium cell with different relative diameter of sintering neck. The different relative diameter of sintering neck from the two situations also obeys the same law.

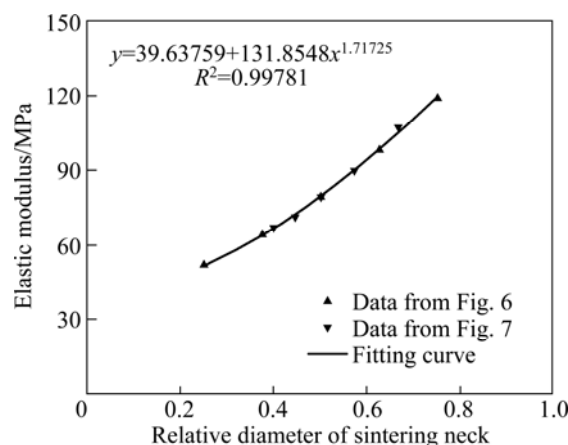


Fig. 9 Fitting of elastic modulus of porous titanium cell with different relative diameter of sintering neck

NANJANGUD and GREEN [20] gave an example which showed an approximate linear relationship between the elastic modulus values and the neck ratio/particle radius ratio of porous glass specimens produced by sintering of spherical particles. This model was only derived for small neck ratio/particle radius ratio values. And it seems to have a similar law with that of porous titanium sintered by titanium wire in this work.

For the relationship between the relative diameter

and compressive mechanical properties, the best fitting results of the numerical fitting will be acquired when we use the formula style: $y=a+bx^c$, where a means the contribution coefficient of helical pore structure, b means the contribution coefficient of sintering neck, and c is the contribution exponent of the sintering neck to the mechanical properties of porous titanium cell. Thus, we can get the fitting results as

$$\sigma_{d_{sr}} = 0.7869 + 2.7340d_{sr}^{3.3769} \quad (2)$$

$$E_{d_{sr}} = 36.63759 + 131.8548d_{sr}^{1.71725} \quad (3)$$

where $\sigma_{d_{sr}}$, d_{sr} and $E_{d_{sr}}$ denote the compressive yield stress, relative diameter of sintering neck and elastic modulus, respectively.

Suppose the value of d_{sr} is zero, then the value of a in the formula $y=a+bx^c$ will express the compressive mechanical properties of porous titanium cell without any sintering neck (for the compressive yield stress, the value of a is 0.7869 MPa, and for elastic modulus the value of a is 36.63759 MPa). These properties are caused by the special helical structure of the porous titanium. When the value of d_{sr} is 1.0, the diameter of sintering neck is as large as that of titanium wire. So the value of b means the basic part of compressive mechanical properties which is contributed by the sintering neck (for the compressive yield stress, the value of b is 2.7340 MPa, and for elastic modulus the value of b is 131.8548 MPa). From Eqs. (2) and (3), it can be found that b is approximately 3.5 larger than a , which indicates that the sintering neck has a huge impact on the compressive mechanical properties of the porous titanium cell. The value of c can be considered the contribution exponent of the sintering neck to the mechanical properties of porous titanium cell. When d_{sr} is larger than 1.0, the contribution of b is amplified. While d_{sr} is less than 1.0, the contribution of b is reduced.

4.2 Effect of sintering neck position

Besides the diameter of the sintering neck, the position of the sintering point also plays an important role. In three-dimensional cell of the porous titanium, there are three different sintering points called C1, C2 and C3 (see Figs. 2 and 5). These three sintering points play different roles in the compressing process (see Fig. 5). In order to express the different combinations of the diameter of sintering neck at the three points, an expression is defined as (d_{C1}, d_{C2}, d_{C3}) standing for the sintering necks at C1, C2 and C3. Figure 10 shows the compressive mechanical properties of porous titanium cell with different combinations of sintering neck. The increase of diameter of sintering neck at C1, C2 and C3 will help to improve the compressive mechanical properties. Among them, the effect of increasing the

diameter of sintering neck at C2 position is very small while the effect of increasing the diameter of sintering neck at C1 position is huge. From the results in Fig. 10, the compressive yield stress and elastic modulus of (150, 100, 100) are 30% higher than those of (100, 150, 100), which indicates that under the same condition, increasing the diameter of sintering neck at C1 position is much effective on the increasing of compressive properties. So the diameter of sintering neck at C1 should be increased in the sintering process as possible as we can.

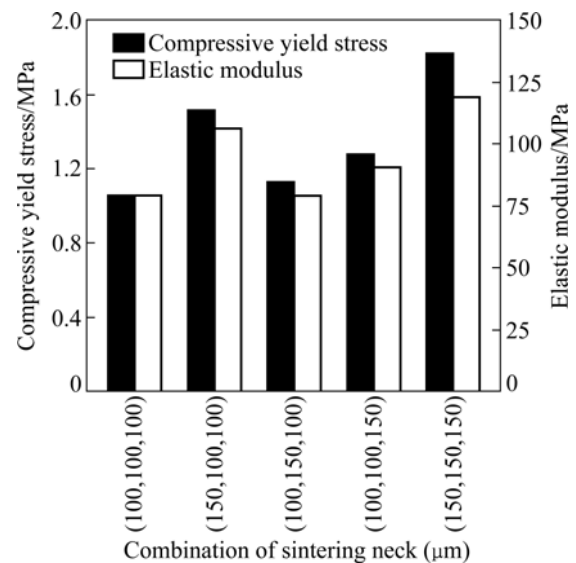


Fig. 10 Compressive mechanical properties of porous titanium cell with different combination of sintering neck

5 Conclusions

The compressive mechanical properties of porous titanium sintered by titanium wire have a close connection with the sintering neck.

1) Stress concentration will first appear at the sintering neck when the porous titanium cell is under uniaxial compressive loading.

2) Contribution coefficient of sintering neck is approximately 3.5 times larger than that of helical pore structure.

3) The larger the relative diameter of sintering neck is, the higher the compressive yield strength and elastic modulus will be.

4) The position of the sintering point also plays an important role. The sintering point of C1 position is the most important sintering position.

References

- [1] LONG M, RACK H J. Titanium alloys in total joint replacement—A materials science perspective [J]. *Biomaterials*, 1998, 19: 1621–1639.
- [2] NISHIGUCHI S, KATO H, FUJITA H, OKA M, KIM H M,

- KOKUBO T, NAKAMURA T. Titanium metals form direct bonding to bone after alkali and heat treatments [J]. *Biomaterials*, 2001, 22: 2525–2533.
- [3] GEETHA M, SINGH A K, ASOKAMANI R, GOGIA A K. Ti based biomaterials, the ultimate choice for orthopaedic implants—A review [J]. *Progress in Materials Science*, 2009, 54: 397–425.
- [4] RYAN G, PANDIT A, APATSIDIS D P. Fabrication methods of porous metals for use in orthopaedic applications [J]. *Biomaterials*, 2006, 27: 2651–2670.
- [5] KIENAPFEL H, SPREY C, WILKE A, GRISS R. Implant fixation by bone ingrowth [J]. *The Journal of Arthroplasty*, 1999, 14(3): 355–367.
- [6] XIANG C S, ZHANG Y, LI Z F, ZHANG H L, HUANG Y P, TANG H P. Preparation and compressive behavior of porous titanium prepared by space holder sintering process [J]. *Procedia Engineering*, 2011, 27: 768–774.
- [7] BARBAS A, BONNET A S, LIPINSKI P, PESCI R, DUBOIS G. Development and mechanical characterization of porous titanium bone substitutes [J]. *Journal of the Mechanical Behavior of Biomedical Materials*, 2012, 9: 34–44.
- [8] TEIXEIRA L N, CRIPPA G E, LEFEBVRE L P, de OLIVEIRA P T, ROSAA L, BELOTI M M. The influence of pore size on osteoblast phenotype expression in cultures grown on porous titanium [J]. *International Journal of Oral and Maxillofacial Surgery*, 2012, 41(9): 1097–1101.
- [9] ZOU C M, ZHANG E, LI M W. Preparation, microstructure and mechanical properties of porous titanium sintered by Ti fibres [J]. *Journal of Materials Science: Material in Medicine*, 2008, 19: 401–405.
- [10] ZHANG E, ZOU C M. Porous titanium and silicon-substituted hydroxyapatite biomodification prepared by a biomimetic process: Characterization and in vivo evaluation [J]. *Acta Biomaterialia*, 2009, 5: 1732–1741.
- [11] ZHANG E, ZOU C M, YU G N. Surface microstructure and cell biocompatibility of silicon-substituted hydroxyapatite coating on titanium substrate prepared by a biomimetic process [J]. *Materials Science and Engineering C*, 2009, 29: 298–305.
- [12] ZHANG E, ZOU C M, ZENG S Y. Preparation and characterization of silicon-substituted hydroxyapatite coating by a biomimetic process on titanium substrate [J]. *Surface & Coatings Technology*, 2009, 203: 1075–1080.
- [13] CHAWLA N, DENG X. Microstructure and mechanical behavior of porous sintered steels [J]. *Materials Science and Engineering A*, 2005, 390: 98–112.
- [14] OH I H, NOMURA N, MASAHASHI N, HANADA S. Mechanical properties of porous titanium compacts prepared by powder sintering [J]. *Scripta Materialia*, 2003, 49: 1197–1202.
- [15] GIBSON J L, ASHBY F M. *Cellular solids: Structure and properties* [M]. 2nd edition. Cambridge: Cambridge University Press, 1997: 152–200.
- [16] LIU P S. A new analytical model about the relationship between nominal failure stresses and porosity for foamed metals under biaxial tension [J]. *Materials and Design*, 2007, 28: 2678–2683.
- [17] HE G, LIU P, TAN Q B. Porous titanium materials with entangled wire structure for load-bearing biomedical applications [J]. *Journal of the Mechanical Behavior of Biomedical Materials*, 2012, 5(1): 16–31.
- [18] HARDY D, GREEN J D. Mechanical properties of a partially sintered alumina [J]. *Journal of the European Ceramic Society*, 1995, 15: 769–715.
- [19] ZHU S L, YANG X J, FU D H, ZHANG L Y, LI C Y, CUI Z D. Stress–strain behavior of porous NiTi alloys prepared by powders sintering [J]. *Materials Science and Engineering A*, 2005, 408: 264–268.
- [20] NANJANGUD C S, GREEN J D. Mechanical behavior of porous glasses produced by sintering of spherical particles [J]. *Journal of the European Ceramic Society*, 1995, 15: 655–660.
- [21] DEMIRSKYI D, AGRAWAL D, RAGULYA A. Neck growth kinetics during microwave sintering of copper [J]. *Scripta Materialia*, 2010, 62: 552–555.
- [22] DEMIRSKYI D, AGRAWAL D, RAGULYA A. Neck growth kinetics during microwave sintering of nickel powder [J]. *Journal of Alloys and Compounds*, 2011, 509: 1790–1795.
- [23] TANG B, TANG Y, ZHOU R, LU L S, LIU B, QU X M. Low temperature solid-phase sintering of sintered metal fibrous media with high specific surface area [J]. *Transactions of Nonferrous Metals Society of China*, 2011, 21: 1755–1760.

烧结颈对多孔钛压缩力学性能的影响

邹鹤鸣¹, 刘燕², 杨 铎¹, 王宏伟¹, 魏尊杰¹

1. 哈尔滨工业大学 材料科学与工程学院, 哈尔滨 150001;

2. 吉林大学 工程仿生教育部重点实验室, 长春 130022

摘 要: 为了研究烧结颈在螺旋孔隙多孔钛中所起的作用, 利用数值模拟的方法对烧结颈尺寸和烧结颈位置对多孔钛单胞压缩力学性能的影响进行研究。结果表明: 多孔钛单胞的压缩力学性能由单胞的螺旋孔隙结构和烧结颈决定, 烧结颈的贡献系数约是螺旋孔结构贡献系数的 3.5 倍; 随着相对烧结颈尺寸的增加, 多孔钛单胞的压缩屈服强度和弹性模量增加; 单胞中 C1 烧结点是最重要的烧结位置; 在相同条件下, 增加 C1 处的烧结颈尺寸对压缩性能的提高更有效。

关键词: 多孔钛; 螺旋孔结构; 单胞; 烧结颈; 压缩力学性能; 贡献系数

(Edited by YUAN Sai-qian)