

Research on elastic modulus of biomedical porous NiTi shape memory alloy

YONG-HUA LI*, BING- JIAN YAN, BING LI, QI ZHANG, JUN MU

School of Materials Science and Engineering, Shenyang Ligong University, Shenyang 110159, P.R. China

Biomedical porous NiTi shape memory alloy (SMA) with different porosity was fabricated by two approaches of powder sintering and combustion synthesis, respectively. It is characterized by biomimetic porous structure. The elastic modulus in compression of the porous NiTi SMA was measured. Furthermore, the effective elastic modulus of porous NiTi SMA was calculated using Voigt upper limit and homogenization theory, respectively. The predicted data agreed basically with the variation trend of the experimental data.

(Received November 17, 2014; accepted January 21, 2015)

Keywords: Porous, NiTi shape memory alloy, Elastic modulus, Implant

1. Introduction

Among the common biomedical metallic materials, titanium alloys are widely used in orthopedics fields because of excellent biocompatibility and good mechanical properties [1]. But the “stress shielding” effect which results from the significant difference of the elastic modulus between the implanted metal and bone should not be neglected. The metallic implant with much higher elastic modulus will carry most of loading. Consequently, the intact bone may cause osteoporosis, bone resorption or even loosening of the implant [2].

There are two common effective approaches to reduce the elastic modulus of biomedical metallic implant. One is to develop new near β type titanium alloy with low elastic modulus like Ti-15Mo alloy, Ti-12Mo-6Zr-2Fe alloy, etc. [1]. The other is to fabricate integral porous structure metals like porous titanium, porous NiTi shape memory alloy (SMA) and so on [3-15].

Porous NiTi SMA has drawn much attention due to the unique porous structure and superelasticity, appropriate mechanical property. The porous structure allows ingrowth of bone tissue and transportation of nutrition. The proper elastic modulus matches that of cancellous bone.

Recently, some experimental research efforts were devoted to fabricate porous NiTi SMA with desirable architecture and microstructure by different methods like combustion synthesis, or self-propagating high-temperature synthesis (SHS), powder sintering, capsule-free hot isostatic pressing, etc. [6-10]. Furthermore, some investigations focused on mechanical property, corrosion resistance and surface modification with hydroxyapatite coating of porous NiTi SMA [11, 12]. *In vitro* evaluation showed its good biocompatibility [13].

In vivo study indicated its excellent osteointegration and bone ingrowth of NiTi foam [14]. Intervertebral fusion device is one of the successful applications of porous NiTi SMA [15].

Additionally, some theoretical studies of porous NiTi SMA involved in numerical simulation of inelastic, superelastic-plastic or pseudoelastoplastic behavior [16-18]. Micromechanical averaging techniques were used to establish the effective elastic and inelastic behavior based on information about the mechanical response of the individual phases and shape and volume fraction of the inhomogeneities [16]. A new constitutive model for porous shape memory alloys based on the Gurson–Tvergaard–Needleman formulation was proposed to investigate the effect of micro-voids on the superelastic-plastic behavior of shape memory alloy [17]. A phenomenological two-phase constitutive model was proposed for pseudoelastoplastic behavior of porous shape memory alloy [18].

Microstructure-based stress for porous ceramics was analyzed by homogenization method with digital image-based modeling [19]. An analytical homogenization method was developed to estimate the effective mechanical properties of fluid-filled porous media [20].

In this paper, porous Ni-50Ti (at.%) SMA was prepared from powder sintering and combustion synthesis, respectively. Pore morphology and elastic modulus in compression were investigated by using scanning electron microscopy (SEM) and compression test, respectively. Effective elastic modulus of porous NiTi SMA was predicted using Voigt upper limit and homogenization theory, respectively.

2. Experiment details

2.1 Powder sintering process of porous NiTi SMA

Ni and TiH₂ powders were blended according to the nominal composition of Ni-50Ti (at.%), then NH₄HCO₃ powders as space-holder were blended with the mixed metal powders and cold pressed into green compacts. Under vacuum condition, the compacts placed in furnace were heated to 473K for 1h to decompose NH₄HCO₃ powders, and then heated to 1053K for 1h to dehydrogenate TiH₂ powders. At last, the compacts were heated to 1323K and sintered for 4 h and then cooled.

2.2 Combustion synthesis process of porous NiTi SMA

Ni and Ti powders were mixed according to equiatomic ratio and made into green compact and then placed into combustion chamber. Under protection of pure argon, the compact preheated at 673K was ignited at one end by external heating source. Then the combustion wave would propagate instantaneously and cooled, porous NiTi SMA could be synthesized.

2.3 Characterization of porous NiTi SMA

The porous NiTi SMAs prepared by powder sintering and combustion synthesis, respectively were cut into specimens with dimension of $\Phi 9\text{mm} \times 15\text{mm}$ and cleaned for compression test.

Samples of $\Phi 2.5\text{mm} \times 1\text{mm}$ was cut and cleaned for differential scanning calorimetry (DSC) test. The results indicate that the sintered and synthesized porous NiTi SMAs are in martensitic state at ambient temperature.

3. Results and discussion

Fig. 1 shows the pore morphology of porous NiTi SMA fabricated by powder sintering. It can be seen clearly that it is featured by some large open and permeable pores connected with walls on which some small and close pores located. Fig. 2 presents the pore morphology of porous NiTi SMA prepared from combustion synthesis. It is characterized by three-dimensionally interconnected reticular structure. The open porous structure favors transportation of human body fluid and nutrients, ingrowth of bone tissue.

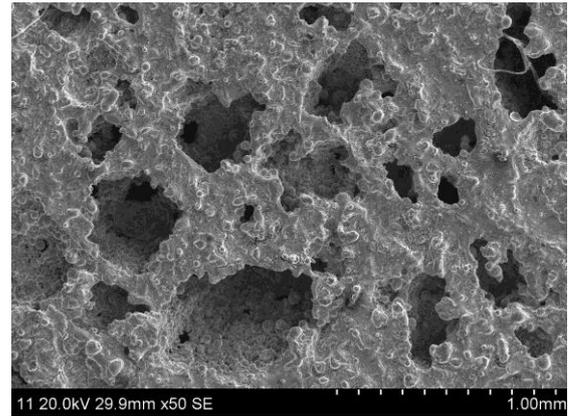


Fig. 1. SEM image of pore morphology for porous NiTi SMA prepared from powder sintering.

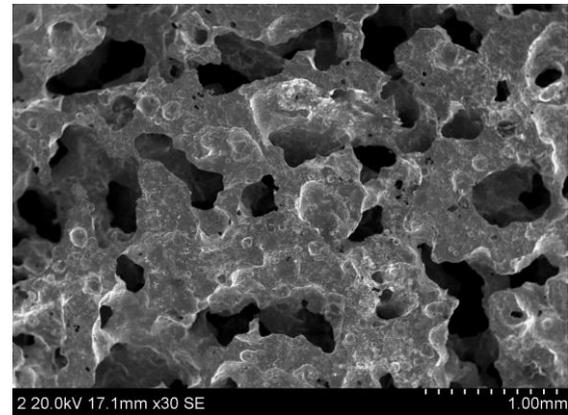


Fig. 2. SEM image of pore morphology for porous NiTi SMA prepared by combustion synthesis.

Elastic moduli of the sintered and synthesized porous NiTi SMAs are plotted against porosity, as shown in Fig. 3.

Apparently, elastic modulus decreases significantly with the increase of porosity.

Voigt upper limit for effective elastic modulus (K_V) is a simple approach to calculate the effective elastic modulus of composite [20]. It can be expressed as Eq. (1)

$$K_V = \sum C_i K_i \quad (1)$$

where C_i and K_i are the volume fraction and elastic modulus of the components, respectively.

Porous NiTi SMA can be considered to be composed of components of pore and NiTi SMA. The elastic moduli of pore and martensitic NiTi SMA are 0 and 40GPa, respectively. Volume fractions of the pore and NiTi component correspond to P (porosity) and $(1-P)$, respectively. The variation of the predicted Voigt upper limit for effective elastic modulus with porosity of porous NiTi SMA can be seen in Fig. 3.

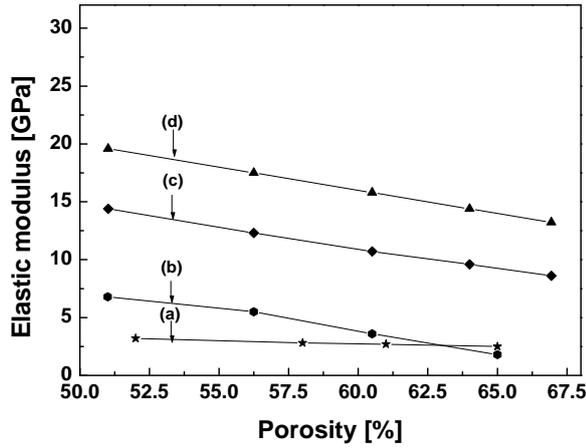


Fig. 3. Variation of elastic modulus with porosity of porous NiTi SMA. (a) experimental data of the sintered samples; (b) experimental data of the synthesized samples; (c) theoretical data calculated by homogenization method; (d) theoretical Voigt upper limit.

Homogenization theory is a common method to calculate the effective elastic modulus of a composite with periodic structure [21-24]. In order to simplify the inhomogeneous porous structure of actual metal foam, a periodic porous structure model and representative base cell is proposed and shown in Fig. 4 [22]. The interconnected walls and blank space correspond to the solid martensitic NiTi SMAs and pores, respectively.

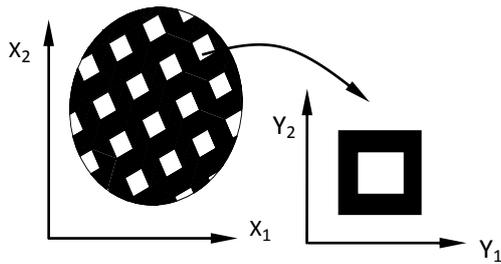


Fig. 4. Periodic porous material and representative base cell.

Suppose that Y is the period of field functions of strain or stress. The mesoscopic equilibrium equation [21, 22] is

$$\frac{\partial}{\partial y_j} [C_{ijkl} e_{ykl}(\chi_i^{kl})] + \frac{\partial C_{ijkl}}{\partial y_j} = 0 \quad (2)$$

the effective elastic modulus can be expressed as

$$C_{ijkl}^H = \frac{1}{|Y|} \int_Y C_{ijmn} [T_{mn}^{kl} + e_{ymn}(\chi^{kl})] dY \quad (3)$$

where C_{ijkl} is the elastic constant of the matrix in the unit, χ^{kl} is the generalized displacement, e_{ykl} is the strain.

$$T_{ij}^{kl} = \frac{1}{2} (\delta_{ik} \delta_{jl} + \delta_{il} \delta_{jk}) \quad (4)$$

Where δ_{ij} is Kronecker Delta function the discrete equation of Eq.(4) is

$$[K][\chi^{kl}] = [P^{kl}] \quad (5)$$

where K is the stiffness matrix of the unit, P is the loading matrix.

$$[K] = \int_{-1}^1 \int_{-1}^1 [B]^T [D] [B] t |J| d\xi d\eta \quad (6)$$

$$[P^{kl}] = - \int_{-1}^1 \int_{-1}^1 [B]^T [D] t |J| d\xi d\eta \quad (7)$$

where B , D and J are the strain, stress-strain relationship and Jacobi matrix, respectively, t is the thickness of the unit.

Considering the periodic boundary condition, the effective elastic modulus can be obtained based on the calculated generalized displacement χ^{kl} .

The effective elastic modulus of porous NiTi SMA can be predicted using finite element method based on Eqs. (3) and (5) [23, 24]. As presented in Fig. 3, variation trend of the predicted effective elastic modulus is similar to that of experimental data of porous NiTi SMA.

The elastic modulus range of compact and cancellous bones is 17~18.9 and 0.05~2 GPa, respectively [25, 26]. The experimental elastic modulus data of porous NiTi SMAs are comparable to those of cancellous bones.

4. Conclusions

The investigations have demonstrated that porous NiTi SMA with controlled porosity can be fabricated by powder sintering and combustion synthesis, respectively. The effective elastic moduli predicted by Voigt upper limit

and homogenization method coincide basically with the variation tendency of experimental data. Matching of elastic moduli between the porous NiTi SMA and bone indicates that NiTi foam is expected to be an implant candidate used for replacement or repair of hard tissue.

Acknowledgements

The authors are grateful for the Science Public Welfare Research Funds of Liaoning Province in China under Grant No.2012002008.

References

- [1] K. Wang, *Mater. Sci. Eng. A* **213**, 134 (1996).
- [2] G. Ryan, A. Pandit, D.P. Apatsidis, *Biomaterials*, **27**, 2651 (2006).
- [3] Ik-Hyun Oh, N. Nomura, N. Masahashi, S. Hanada, *Scripta Mater.*, **49**, 1197 (2003).
- [4] Y. H. Li, Z. Q. Sun, X. L. Li, P. P. Ding, L. K. Gong, *J. Optoelectron. Adv. Mater.*, **16**, 513(2014).
- [5] G. Chen, P. Cao, G. A. Wen, N. Edmonds, Y. M. Li, *Intermetallics*, **37**, 92 (2013).
- [6] V. I. Itin, V. E. Gyunter, S. A. Shabalovskaya, R. L. C. Sachdeva, *Mater. Charact.*, **32**, 179 (1994).
- [7] Y. H. Li, H. Liu, J. Zhang, Z. Q. Sun, Z. Y. Deng, *Optoelectron. Adv. Mater. – Rapid Comm.*, **7**, 414 (2013).
- [8] A. Bansiddhi, T. D. Sargeant, S. I. Stupp, D. C. Dunand, *Acta Biomater.*, **4**, 773 (2008).
- [9] B. Yuan, X. P. Zhang, C. Y. Chung, M. Zhu, *Mater. Sci. Eng. A*, **438**, 585 (2006).
- [10] S. L. Zhu, X. J. Yang, D. H. Fu, L. Y. Zhang, C. Y. Li, Z. D. Cui, *Mater. Sci. Eng. A*, **408**, 264 (2005).
- [11] Y. H. Li, G. B. Rao, L. J. Rong, Y. Y. Li, *Mater. Lett.*, **57**, 448 (2002).
- [12] H. C. Jiang, L. J. Rong, *Surf. Coat. Technol.*, **201**, 1017 (2006).
- [13] M. Assad, A. Chernyshov, M. A. Leroux, C. H. Rivard, *Bio-Med. Mater. Eng.*, **12**, 225 (2002).
- [14] S. Kujala, J. Ryhanen, A. Danilov, J. Tuukkanen, *Biomaterials*, **24**, 4691 (2003).
- [15] M. Assad, P. Jarzem, M.A. Leroux, C. Coillard, A. V. Chernyshov, S. Charette, C. H. Rivard, *J. Biomed. Mater. Res. B*, **64**, 107 (2003).
- [16] P. B. Entchev, D. C. Lagoudas, *Mech. Mater.*, **34**, 1 (2002).
- [17] J. S. Olsen, Z. L. Zhang, *Inter. J. Solids Struct.*, **49**, 1947 (2012).
- [18] T. El Sayed, E. Gürses, A. Siddiq, *Comput. Mater. Sci.*, **60**, 44 (2012).
- [19] N. Takano, M. Zako, F. Kubo, K. Kimura, *Inter. J. Solids Struct.*, **40**, 1225 (2003).
- [20] A. Chakraborty, *Inter. J. Solids Struct.*, **48**, 3395 (2011).
- [21] S. Y. Du, B. Wang, *Mesomechanics for composites (In Chinese)*, Beijing: Science Press, 1998
- [22] S. B. Zhuang, C. C. Wu, M. L. Feng, Z. Yuan, *Mater. Sci. Eng. (In Chinese)*, **19**, (4) 9 (2001)
- [23] B. Hassani, E. Hinton, *Comput. Struct.*, **69**, 707 (1998).
- [24] B. Hassani, E. Hinton, *Comput. Struct.*, **69**, 719 (1998).
- [25] W. Suchanek, M. Yoshimura, *J. Mater. Res.*, **13**, 94 (1998).
- [26] L. L. Hench, E. C. Ethridge, in: *Biomaterials: An Interfacial Approach*, Academic Press, New York, 1982.

*Corresponding author: yhlien@163.com