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The effect of heating schedule and the size of the powder compact on the pore structure, mechanical and fatigue properties of porous TiNi produced by a self-propagating high-temperature synthesis method

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Porous TiNi bodies with different porous microstructures were produced from (Ti + Ni) powder mixtures by self-propagating high-temperature synthesis (SHS). Three different heating schedules were used to change the microstructure. Tensile strength, compression strength and elastic modulus were measured. Fatigue tests were performed. The relationship between the porous microstructure and mechanical properties are discussed.

Key words: TiNi, porous body, SHS, ignition, heating schedule, compact size.

Introduction

Titanium-nickel intermetallic compound is a representative shape memory alloy (SMA) due to a unique thermal shape memory effect, superelasticity, good corrosion resistance, and high damping properties. These properties make it ideal as a biomaterial, especially in orthopaedic surgery and orthodontics. In fact TiNi SMA implants have been developed and are widely used in cardiovascular and gastrointestinal applications [1].

A SHS (Self-Propagating High-temperature Synthesis) process is a very suitable for the production of porous TiNi shape memory alloy. The synthesis reaction occurs generally through ignition, propagation of a combustion wave front and cooling. It has advantages such as process economics, simplicity and purification of the reaction product [2, 3].

In this study we produced cylindrical porous TiNi bodies with different microstructures by SHS varying the heating schedule prior to ignition and the size of the powder compact. Tensile, compression strength and elastic modulus were measured. Fatigue tests were also performed. The relationship between the porous microstructure and mechanical properties are discussed together with a comment on the biocompatibility.

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Experimental Procedure

A detailed production procedure has been already given in our previous paper [4]. Ti and Ni starting powders were mixed in an argon-gas sealed container using a ball mill to have a composition of Ti:Ni=50:50 at% and then vacuum-dried. The prepared powder mixture was loaded in a quartz tube with diameters of 20 and 40 mm, and loose-compacted by tapping. A compacted preform was loaded into a reaction furnace under flowing argon-gas with a heating rate of 20 K/minute and ignited electrically with the use of a tungsten filament. After the SHS reaction the sample was cooled to room temperature. Three different heating schedules prior to ignition were used: (1) A-type (heated to 450 °C and instantly ignited), (2) B-type (heated to 380 °C, soaked for 1hour and then ignited), and (3) C-type (heated to 400 °C, furnace-cooled for 30 minutes and then ignited). The samples were cross-sectioned by electric-discharge wire-cutting. X-ray diffractometry (XRD), scanning electron microscopy (SEM) and differential scanning calorimetry (DSC) were used for phase analysis, microstructural observation and image analysis, and the determination of transformation temperature.

Results and Discussion

Figure 1 shows SEM images of the starting Ti and Ni powders used in this study. Ti powder has a smooth surface and irregular shape while Ni has a spiky and round shape. Particle size analysis showed that the

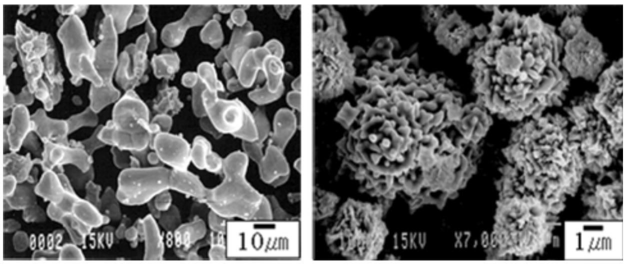

Fig. 1. SEM micrographs of the starting powders used in this stu (left: Ti, right: Ni).

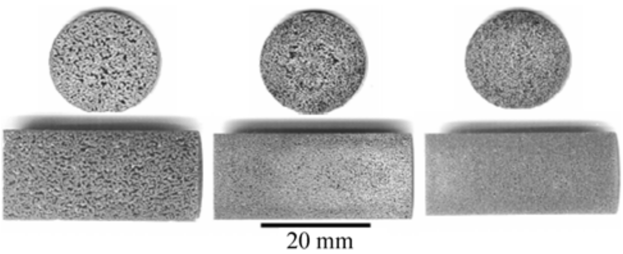

Fig. 2. Cross-sections of TiNi bodies produced in this study which are 20 mm diameter. (Upper row: section transverse to the combustion wave propagation, lower row: longitudinal section, from left to right: A-, B, and C-type)

average sizes of Ti and Ni were 29 and 12 μm, respectively, whose distributions were unimodal for both.

Photographs of cross-sections of typical porous TiNi bodies 20 mm in diameter are given in Fig. 2. In this figure the upper row shows the transverse section (normal to the direction of combustion wave propagation), while the lower row the longitudinal section (parallel to that).

The A-type specimen seems to have the largest pore size. Irrespective of heating schedule, all specimens show a homogeneous porous structure. In order to observe the porous microstructure more clearly, SEM images of cross-sections of the specimens in Fig. 2 are given in Fig. 3. All pores are interconnected. The A-type specimen shows the largest pore size and the C-type the smallest. It seems that there is almost no

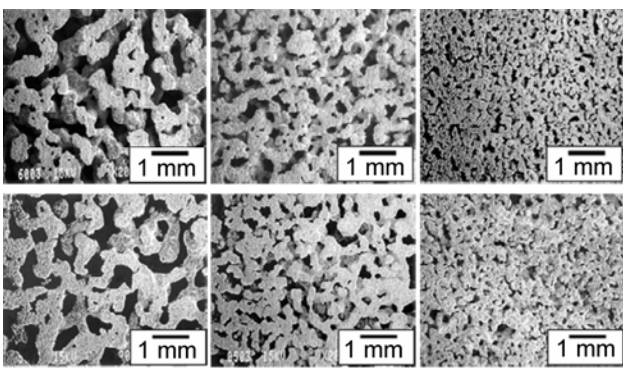

Fig. 3. SEM photos of cross-sections of TiNi bodies given in Fig. 2. (Upper row: section transverse to the combustion wave propagation, lower row: longitudinal section, from left to right: A-, B, and C-type)

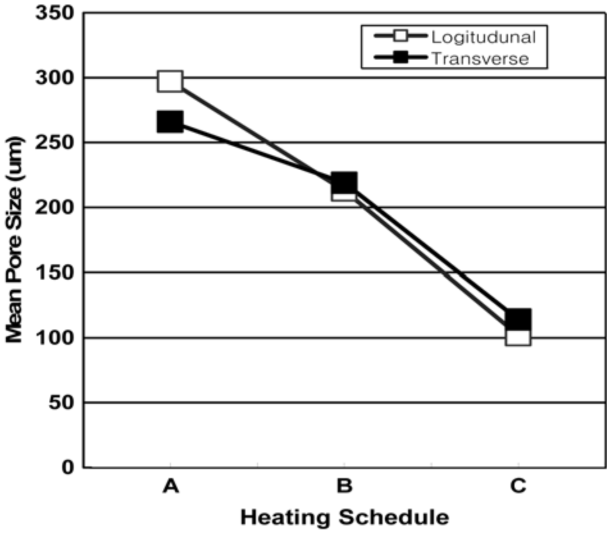

Fig. 4. Mean pore size of the specimens in Fig. 3.

Table 1. Ignition Temperature and Maximum Combustion Temperature during SHS Reaction of (Ti+Ni) Powder Compact with Diameters of 20 and 40 mm, heated with Various Schedules of A, B and C Types.

			A-type	B-type	C-type
Ignition Temp. (°C)	20Φ	Surface	450	330	340
		Inside	311	328	337
	40Φ	Surface	450	340	350
		Inside	150	305	334
Max.Comb. Temp. (°C)	20Φ		959	946	943
	40Φ		1060	1127	1156

difference in pore size between the sectioning directions which confirms also that the pores are three-dimensionally interconnected. For these samples the summarized results of mean pore size determined by image-analysis are given in Fig. 4. In spite of the difference in pore size, all specimens had almost the same porosity of 64%.

The size of the powder compact affected the ignition

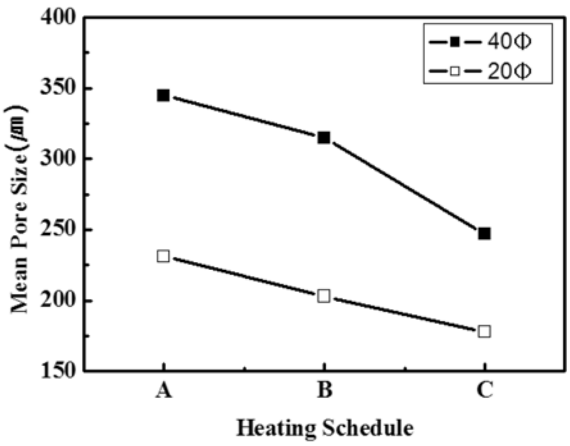

Fig. 5. Mean pore size of TiNi bodies with diameters of 20 and 40 mm.

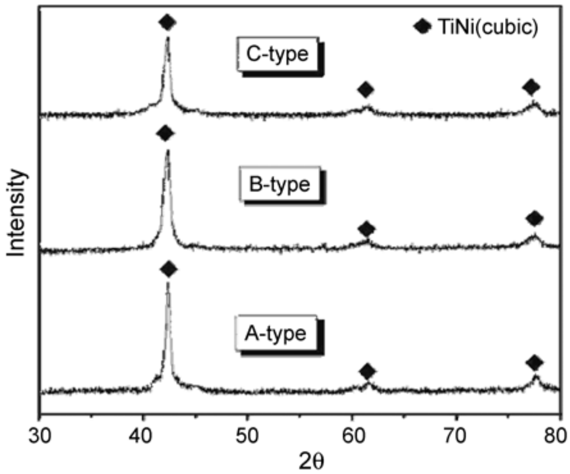

Fig. 6. XRD patterns of the TiNi bodies produced.

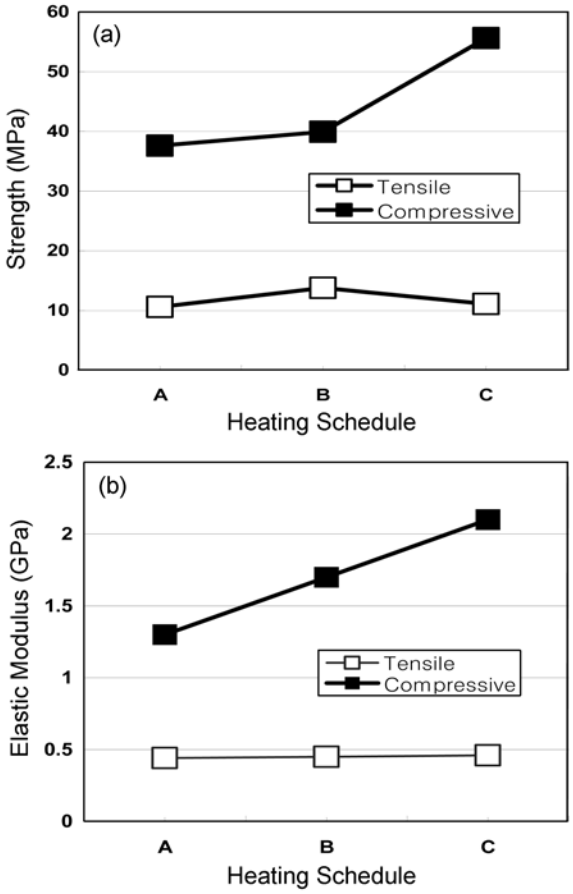

Fig. 7. Mechanical properties of TiNi porous bodies produced in this study which had a 40 mm diameter: (a) Tensile and compressive strength and (b) elastic modulus. (Tensile test specimens were prepared from the 40 mm diameter TiNi bodies while the compression test specimens were 20 mm in diameter.)

temperature, the maximum combustion temperature and the mean pore size, as shown in Table 1 and Fig. 5. The difference in the maximum combustion temperature is more evident than that in the ignition temperature. A higher maximum combustion temperature seems to be

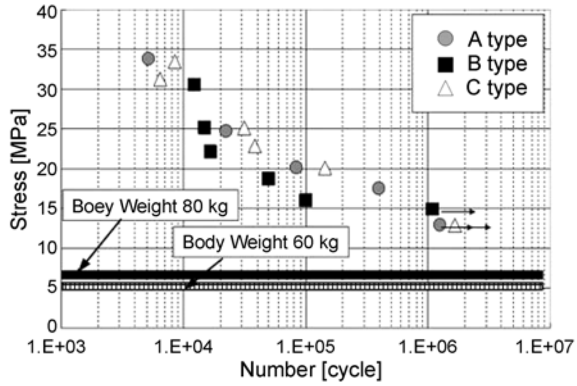

Fig. 8. Fatigue test results of 40 mm diameter TiNi bodies (all specimens showed no failure even above 10^6 cycles).

related with an increase in the mean pore size. The sample with a diameter of 40 mm showed a higher maximum combustion temperature and a larger pore size compared to the sample with a 20 mm diameter.

From the XRD analysis, it was confirmed that the major phase formed in the TiNi porous bodies produced is mainly the B_2-TiNi (cubic) phase (Fig. 6). This result is somewhat different to that in our previous study in which a trace of Ti_2Ni had also been detected.

Results of mechanical properties are summarized in Fig. 7. It should be noted that the tensile test specimens were prepared from the 40 mm diameter TiNi porous bodies, not 20 mm as in the case of the compression test specimens. As can be seen in the figure, the C-type specimen shows the highest compressive strength of 55.6 ± 11.0 MPa, while the strength of A- and B-type are 37.6 ± 2.3 and 39.9 ± 7.9 MPa, respectively. The tensile strengths were in the range of 10.6-13.8 MPa. This value is much lower than that of our previous study (15-25 MPa) [6]. This can be explained by the size effect. That is, in [6] the tensile test specimens had been prepared from 25 mm diameter TiNi porous bodies.

The mechanical properties given in Fig. 7 are in a good range similar to those of human bones which have been given in reference [7]. Fatigue test results showed no failure even above 10^6 cycles under the condition of body weights of 60 and 80 kg (Fig. 8). *In vivo* biocompatibility tests using rabbits also showed an excellent result [8].

Conclusions

TiNi porous bodies with different porous microstructures and sizes were produced from a (Ti + Ni) powder mixture by a self-propagating high-temperature synthesis (SHS) method, varying the heating schedule before ignition. The average pore size was in the range of 108-280 μm depending on the heating schedule. All pores are three-dimensionally well interconnected. The tensile, compressive strength and elastic modulus are similar to those of human bones. Fatigue tests showed an excellent result for the biomedical application of this material.

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References

1. T. Duerig, A. Pelton and Stockel, Material Science and Engineering 15 (1999) 273-275.
2. A.G. Merzhanov, Experimental Heat Transfer, Fluid Mechanics and Thermodynamics, Edizioni ETS (1997) 1869.
3. J.J. Moore and H.J. Feng, Progress in Materials Science 39 (1995) 243-273.
4. J.S. Kim, S.H. Lee, J.H. Kang, V.E. Gjunter, S.B. Kang, T.H. Nam, and Y.S. Kwon, SMST-2000: Proceedings of SMST 2000, California, USA (2000) 77.
5. S.B. Kang, K.S. Yoon, J.S. Kim, T.H. Nam, and V.E. Gjunter, Materials Transactions 43[5] (2002) 1045-1048.
6. J.S. Kim, J.H. Kang, S.B. Kang, K.S. Yoon, and Y.S. Kwon, Advanced Engineering Materials 6 (2004) 403-406.
7. J. Black and G. Hastings (Ed.), Handbook of Biomaterial Properties, Chapman & Hall (1998).
8. J.S. Kim, S.G. Yang, J.H. Kang, S.B. Kang, K.S. Yoon, and Y.S. Kwon, Materials Science Forum 449-452 (2004) 1097-1100.