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# A recent development in producing porous Ni–Ti shape memory alloys

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#### Abstract

Porous Ni–Ti shape memory alloys (SMAs) are promising candidates for biomedical materials. In the present study, a bulk porous Ni–Ti SMA with a banded structure of channels and 54 vol.% porosity has been successfully prepared by self-propagating high-temperature synthesis (SHS). The Ni–Ti SMA synthesized has an open porous structure, also its pores and channels with various size and shape are three-dimensionally interconnected. Compared to the conventional powder sintering method, the present method extends the porosity range of porous Ni–Ti SMAs. A schematic representation of SHS synthesized Ni–Ti SMAs has been suggested. © 2000 Elsevier Science Ltd. All rights reserved.

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# 1. Introduction

Kazuhiro Otsuka and Xiaobing Ren have reviewed the recent developments in the research and application of SMAs [1], especially with Ni-Ti SMAs such as Ni-Ti film and high-temperature Ni-Ti SMAs. However, another hopeful candidate, porous Ni-Ti SMA, for biomedical material has not been discussed. Porous Ni-Ti SMA has been recently acknowledged as a very important biomaterial for use as bone implants due to its excellent mechanical properties, good corrosion resistance, high biocompatibility, special pseudoelasticity and shape as well as volume memory effect [2]. And its porous structure permits the ingrowth of new-bone tissue along with the transport of body fluids [3]. Moreover, by obtaining different porosity through controlling the production process, the elastic modulus of the final porous Ni-Ti SMA is adjustable and can match that of human bone. Additionally, load-bearing materials in nature (for example, wood, coral and animal bone) are porous in structure, thus porous Ni-Ti SMAs have been considered for many applications such

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as load-bearing artificial bones, for example, spine surgery, lumber osteochondrosis, dental implant, and cranial-facial endoprosthesis [4].

Several powder metallurgical processes, which focused on compact Ni–Ti SMAs, have been developed for manufacturing Ni–Ti SMAs [5–8]. But the difficulty in preparation of porous Ni–Ti SMAs has restricted the research and application of porous Ni–Ti SMAs. Therefore, development of new processes for producing porous Ni–Ti SMAs is now an interesting research topic. In our previous study [9], a conventional powder sintering method was developed for the synthesis of porous Ni–Ti SMAs with porosity in the range of 30–40 vol.%. However, to produce bulk products with higher desired porosity, new methods are needed. In the present study, SHS was used and an attempt was made to control the green porosity using a newly designed cold press die.

# 2. Experimental details

Fig. 1 shows the characteristics of the Ni (38  $\mu$ m) and Ti (21  $\mu$ m) powders. These two kinds of powders were weighed and mixed to 50 at.%Ni. The mixed powder was cold pressed in a newly designed die (Fig. 2). By

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using different compaction pressure with or without a binder, green compacts with different porosity can be obtained, and then it is possible to obtain porous Ni–Ti SMAs with desired porosity. In the present study, the green compact (cylindrical, 80 mm in length and 33 mm in diameter), whose porosity is 40 vol.% after being cold pressed with a pressure of 75 MPa, was placed in a reaction chamber and ignited at a preheating temperature of 550°C.

The phase constituent of the product was determined by X-ray diffraction (XRD) analysis. The pore distribution and pore shape of the product were analyzed with SEM and optical micrography.

## 3. Results

It is found that the combustion synthesis reaction can be ignited at a preheating temperature of 550°C. A dazzling light and evolution of some smoke from the top surface of the compact is observed after the heating power is turned on for several seconds. The light and the evolution of smoke, which appear to be similar to the propagation of a combustion wave, propagate along the compact for tens of seconds.



Fig. 1. The characteristics of the elemental Ti and Ni powders.

After removal of the reaction compact out of the reaction chamber, it is found that the compact almost retains its shape and is highly porous with a porosity of 54 vol.%. The macrograph of the longitudinal section of the product as synthesized and the shape as well as distribution of pores in the porous Ni–Ti SMA are shown in Figs. 3–5, respectively. As can be seen, there are numerous pores with various sizes and shapes, and a banded structure of channels and some shallow pores can be easily observed. The channels distribute along the propagating direction of the combustion wave. Moreover, the channels and pores, which tend to further



Fig. 2. The cold press die used in the present study.



Fig. 3. The macrograph of the longitudinal section of the synthesized porous Ni–Ti SMA.



(b)

Fig. 4. Typical optical and SEM micrographs of the porous Ni–Ti SMA.

coagulate, are almost three-dimensionally interconnected and form an open porous structure.

Fig. 6 shows the XRD patterns of the mixed powder and the synthesized porous Ni–Ti SMA. It can be found that the Ni and Ti powders have been just mechanically blended, and the combustion reaction results in the formation of 100% Ni–Ti intermetallic compounds, which are mainly Ni–Ti phases (i.e. B2(NiTi) and B19'(NiTi)).

## 4. Discussion

Fig. 7 is suggested here as the schematic representation of the SHS synthesized Ni–Ti process. With high heating power passing through the tungsten coil, the top surface temperature of the compact reaches the ignition temperature and the combustion reaction occurs, with a dazzling light and the evolution of smoke as a result of the volatilization of impurity and the escape of adsorbed gases. During combustion, the following reaction mainly takes place:



Fig. 5. Optical and SEM micrographs of the longitudinal section of the porous Ni–Ti SMA with a banded structure showing channels along the propagating direction of combustion wave.

 $Ni + Ti \rightarrow NiTi + 67 \text{ kJmol}^{-1}$ 

As a result, the dominant phases of the synthesized product are NiTi phases (Fig. 6). After ignition, the heat released from the above reaction is transferred along the compact from the top surface to the end and makes the combustion self-sustain. The combustion, and the following dazzling light as well as the evolution of smoke all propagate in the same direction, i.e. the propagating direction, throughout the compact as the heat transfers. It has been found that the combustion temperature is high and can reach 1279°C [10], and a liquid phase appears transiently during combustion. The pores in the green compact (40 vol.%), the transient liquid phase, the volatilization of impurity, and the escape of the adsorbed gases leave numerous pores inside the compact. Therefore, the product as synthesized becomes highly porous with 54 vol.% porosity, and the pores and channels are almost three-dimensionally interconnected. Meanwhile, many small pores coagulate and



Fig. 6. XRD profiles of the mixed powder and the combustion synthesized porous Ni–Ti SMA.



Fig. 7. Schematic representation of the SHS process.

form channels during combustion due to the transient liquid phase. Because the compact is ignited in a limited central area and there exists thermal migration in temperature gradients, the combustion wave propagates like ripples on a pool. And the channels, like the trails of the combustion wave leaving behind the combustion, are formed along the propagating direction of combustion wave, forming a banded structure of channels as can be seen in Figs. 3 and 5. However, the liquid phase is limited and its appearance is transient. Thus, the compact is mainly homogeneously porous with various size and shape pores (see Fig. 4).

## 5. Conclusions

A bulk porous Ni–Ti SMA has been achieved in the SHS of a Ti+Ni powder compact prepared with a newly designed cold press die. Pores with various size and shape are three-dimensionally interconnected, and 100% Ni–Ti intermetallic compounds are formed in the SHS synthesized Ni–Ti SMA. Moreover, it is found that the present method makes it possible to produce porous Ni–Ti SMAs with high porosity. A schematic representation of SHS synthesized Ni–Ti SMAs is suggested to illustrate the banded structure of channels distributing in the bulk product along the propagating direction of combustion wave.

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# References

- [1] Otsuka K, Ren XB. Intermetallics 1999;7:511.
- [2] Itin VI, Gjunter VE, Shabalovskaya SA, Sachdeva RLC. Mater Charact 1994;32:179.
- [3] Pilliar RMJ. Biomed Mater Res 1998;21:1.
- [4] Pakhomenko JS, Syrkashev VA, Gjunter VE. Superelastic shape memory materials and implants in medicine, proceeding of the international conference, June 1996. Tomsk, Russia. Research Institute of Medical Materials and Shape Memory Implants, Tomsk, Russia, 1996, p. 20.
- [5] Yi HC, Moore JJ. Scripta Metall 1988;22:1889.
- [6] Funami K, Sekignchi Y, Funakubo H. J Japan Inst Metals 1984;48:1113.
- [7] Jardine AP, Field Y, Hermen H, Marantz DR, Kowalsky KA. Scripta Metall 1990;24:2391.
- [8] Otaguchi M, Kaieda Y, Oguro N, Shite S, Oie TJ. Japan Inst Metals 1990;54(2):21.
- [9] Li BY, Rong LJ, Li YY. J Mater Res 1998;13:2847.
- [10] Munir ZA, Anselmi-Tamburini U. Mater Sci Rep 1989;3:277.