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The effect of solution treatment under loading on the microstructure and phase transformation behavior of porous NiTi shape memory alloy fabricated by SHS

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ABSTRACT

Porous NiTi shape memory alloy (SMA) was fabricated by self-propagating high-temperature synthesis (SHS). With this study, a new solution treatment "solution treatment under loading" was applied to porous NiTi SMA fabricated by SHS to determine microstructural improvement regarding single phase NiTi. The effect of solution treatment under load on chemical composition, constituent phases and phase transformation behaviors of the specimens was investigated and discussed. The chemical composition of the specimens considerably changed with solution treatment under loading. Intermetallic phases such as Ti₂Ni and Ni₄Ti₃ disappeared, the density of B2(NiTi) phase increased and phase transformation temperatures sharply decreased. Porous single phase B2(NiTi) SMA with high chemical homogeneity could be obtained by the load applied during solution treatment at 1050 °C.

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1. Introduction

Recently, the production of porous NiTi shape memory alloys (SMAs) has been received considerable interest due to their extraordinary mechanical characteristics similar to those of some natural biomaterials for hard tissue implants, quite high damping capacity and low relative density [1,2]. In addition, they have some unique properties like shape memory effect, superelasticity, excellent corrosion resistance, and good biocompatibility. Moreover, the porous structure of the alloys would help tissue cell in growth, nutrition exchange and medicament transportation [3,4].

So far, porous NiTi SMAs have been fabricated with powder metallurgy (PM) processes such as combustion synthesis with selfpropagating wave [5-7], metal injection molding (MIP) [8], hot isostatic pressing (HIP) [8], and spark plasma sintering (SPS) [9]. These processes can avoid the problems associated with casting, like segregation or extensive grain growth and have the added advantages of precise control of composition and easy realization of complex part shapes. However, they can cause oxidation and tendency to form other Ti-Ni phases, such as Ni₃Ti, Ti₂Ni and Ni₄Ti₃

due to the composition fluctuation in the specimen since the raw powders are not mixed sufficiently and the particle size of the reactants is not small enough [2,9,10]. In some cases even elemental Ni in varying proportion occurs, which is toxic in living tissue [10].

None of the above PM processes has allowed obtaining fully dense high purity single phase NiTi components [11]. The mechanical properties of NiTi depend on its phase state at a certain temperature. Fully austenitic NiTi material generally has suitable properties for surgical implantation and superelasticity [12]. Ti₂Ni, Ni₃Ti and Ni₄Ti₃ phases existing in SHS-synthesized porous NiTi SMA may increase the brittleness of the products. Moreover, they can lead to the cavitation corrosion and deteriorate the biocompatibility of porous NiTi SMAs in the physiological environments [1.13].

Different procedures are required to remove undesired phases formed during fabrication. The amount of Ni₄Ti₃ phase in porous NiTi SMA decreases slightly after solution treatment. However, Ti₂Ni and Ni₃Ti phases are extremely difficult to remove by subsequent thermal treatments since they are thermodynamically stable [2,13,14]. Therefore, it is necessary to implement different procedures for removing the undesired Ti₂Ni and Ni₃Ti phases in porous NiTi SMAs.

Bertheville and Bidaux [11] studied an alternative powder metallurgical process called VPCR (vapor phase calciothermic reduction) for the fabrication of single phase NiTi shape memory alloys. They reported that the XRD peaks of austenite B2(NiTi) and



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of CaO were visible when using the VPCR process. In another study [14], they reported a thin calcium oxide film over the surface of the compact which can easily be removed by polishing or leaching. In addition, it was determined by them that the vacuum sintered compact was predominantly B2(NiTi) phase and secondly Ti_2Ni and Ni_4Ti_3 at room temperature while the sintered compact under reducing Ca vapor was the major stable B19'(NiTi) phase instead of the B2(NiTi) phase.

It is well accepted that the shape memory effect (SME) and superelasticity effect (SE) of NiTi SMAs depend critically on the reversibility of the martensitic transformation [15]. Various researchers [4,16–18] have studied the effects of porosity, aging and solution heat treatment on the martensitic transformation behavior of porous NiTi SMAs. However, the effects of solution treatment under loading on characteristic of porous NiTi SMAs fabricated by SHS have not been investigated in the literature.

Porous NiTi SMAs fabricated by SHS have more porosity than porous NiTi SMAs fabricated with the other PM processes according to literature [2,19]. So far, various ignitors such as tungsten coil [20], ignition reagent composed of Ti and C powder [2], laser [21] or other means have been employed for the fabrication of porous NiTi SMAs by SHS.

In this study, high-voltage electric arc was used to ignite the specimens in the fabrication of porous NiTi SMA by SHS. Also, a new solution treatment, "solution treatment under loading" was applied to porous NiTi SMA fabricated by SHS to determine microstructural improvement regarding single phase NiTi. In addition, the phase transformation behavior was also investigated.

2. Experimental procedures

The raw materials used were Ni (99.8 wt.%) and Ti (99.5 wt.%) powders with an average size of 44 µm (Alfa Aesar). Firstly, the powders of Ni and Ti with 50.5 at.%Ni were blended in a rotating container for 24 h for a homogenous mixture, and then the blended powder was pressed into cylindrical compacts of 10 mm in diameter and 15 mm in height using a hydraulic press at a cold compaction pressure of 100 MPa. The green samples after compacting were preheated up to 200 °C with a heating rate of 15 °C/min in a furnace under the protection of high purity argon gas, then were subjected to electrical discharge pulse (14 kV and 30 mA) for about 2 s [22,23]. The temperature of the green samples was increased in a short time at the beginning of current application and ignition started. Once ignited, combustion wave selfpropagated along the axis of the specimen to the other end in a very short time, thus porous NiTi SMA was synthesized. To investigate the effects of solution treatment under loading on microstructure and phase transformation behavior, synthesized specimens were separately solution treated under a load of 25 kg (3.2 MPa) and 50 kg (6.41 MPa) at 1050 °C for 1 h in a furnace under the protection of high purity argon gas. Finally, the specimens were quenched into water at room temperature.

To investigate the chemical composition, the surfaces of specimens were etched by a mixture of 10% HF, 5% HNO₃ in water [13]. An energy-dispersive X-ray spectrometer (EDS) coupled with the scanning electron microscopy (SEM, LEO Evo-40VP) was used to locally measure the chemical composition of the specimens. The phase constituents were determined by X-ray diffraction (XRD, Rigako Rad-B D-Max 2000 XRD) analysis using CuK\alpha radiation with 1.54046 Å. The phase transformation temperatures and energies were measured by differential scanning calorimeter (DSC, PerkinElmer Pyris 6). The samples, typically 14–17 mg in weight, were placed in aluminium pans. The measurements were carried out under nitrogen gas flow. In the DSC thermal analysis, specimens were heated from –50 to 140°C and kept isothermally for 2 min to establish thermal equilibrium, then cooled down to –50°C, and also kept isothermally for 2 min and then ramped back to 140°C again. Finally, they were cooled to –50°C to finish the thermal cycle. Heating and cooling rates were kept at \pm 5 and \pm 10°C/min for the first and second thermal cycles respectively.

3. Results and discussions

Fig. 1 shows the general morphology of porous NiTi fabricated by SHS which consists of the combustion channels and pores. The distribution of the pores is uniform and most of the pores are isolated and rarely interconnected. The pores have fairly low size in μ m and various shapes. The specimens fabricated by SHS has a general porosity of 55.5 vol.% and the porosity decreases to 40.6 vol.% with



Fig. 1. Optic micrograph of porous NiTi synthesized at 200°C.

solution treatment under loading [23]. The pore characteristics and porosity ratios of the specimens produced are suitable for hard tissue implants because the ideal implant material should have the porosity in the range of 30-90%, and the optimal pore size for bone tissue ingress is $100-500 \mu m$ [24].

Fig. 2 shows the microstructure of the porous NiTi fabricated by SHS. The EDS quantitative analysis result of atomic composition was Ni–41.01 at.%Ti for the grain boundary pointed out as Ellipse1 in Fig. 2, corresponding to Ni₃Ti₂ or Ni₄Ti₃ phase. The same composition was seen for the formations like mushrooms or water granules in shape, too. The atomic composition of elliptic area showed as E0 in Fig. 2 was Ni–59.29 at.%Ti, which is possibly a pore. EDS analysis shows that the composition of platelike formations in Fig. 2 is Ni–43.81 at.%Ti, which can be identified as Ni₄Ti₃ or Ni-rich B19'(NiTi) martensite phase by combining EDS and XRD results. NiTi matrix is seen grey; Ni₃Ti₂ or Ni₄Ti₃ phases are seen as light grey in Fig. 2, and NiTi₂ phase in Fig. 3a is seen as cornered shapes in dark grey.

Although the phases such as Ni₄Ti₃ and NiTi₂ usually form in porous NiTi alloys fabricated by SHS [2], the phases such as Ni₃Ti, pure Ni and pure Ti rarely occur [10]. Ni₃Ti, pure Ni and pure Ti were not observed in this study. The presence of Ni₄Ti₃ and Ni₃Ti₂ in the porous NiTi SMA synthesized by SHS is due to the overall stoichiometry of the Ni-rich (50.5 at.%Ni) specimen. Moreover, the presence of Ti₂Ni phase, since Ti powder is more flammable than Ni powder, could make the parent NiTi phase richer in Ni. If the mixing is not homogeneous, the amount of undesired phases becomes larger [2,9,10].



Fig. 2. SEM micrograph and EDX regions of the porous NiTi SMA fabricated by SHS.



Fig. 3. SEM micrographs and EDX regions of the specimens. (a) Solution treatment under 3.2 MPa and (b) solution treatment under 6.41 MPa.

Fig. 3 shows the microstructures of solution treated specimens under different loads at 1050 °C for 1 h. where the microstructure is predominantly NiTi phase as desired. The result of the atomic composition from the EDS quantitative analysis in Fig. 3a was Ni–64.19 at.%Ti for the cornered dark grey area (Ellipse0, E0), possibly corresponding to NiTi₂. Ni₄Ti₃ and Ni₃Ti₂ phases were not observed in Fig. 3. The load applied accelerated the dissolution of these phases by increasing local stress and thus diffusion [25]. There is some amount of NiTi₂ phase in the solution treated specimen under 3.2 MPa (Fig. 3a) but it disappeared completely after the application of a load of 6.41 MPa during the solution treatment (Fig. 3b). The EDS quantitative analyses results of atomic composition were Ni–46.41 at.%Ti and Ni–46.23 at.%Ti for the areas pointed out as Ellipse0 and Ellipse1 in Fig. 3b respectively, corresponding to NiTi phase richer in Ni.

Fig. 4 shows XRD patterns of the synthesized (curve-a) and solution treated specimens under loading. It is seen that the desired products such as B2(NiTi) and B19'(NiTi) are the predominant phases in the Fig. 4 (curve-a). In addition, the SHS process resulted in the formation of several second phases such as Ti_3Ni_4 and Ti_2Ni . Chu et al. [13] also determined the same phases. In addition, they reported that the amount of metastable Ni_4Ti_3 phase decreased sharply after solution heat treatment at 1050 °C for 4 h, and Ti_2Ni phase could not be removed by solution treatment [13]. The results in our study where load and solution treatment were implemented simultaneously showed that when the load on the specimen was 3.2 MPa during solution treatment, it can be seen in Fig. 4 (curve-b) that the amount of B2(NiTi) phase increased, B19'(NiTi) decreased, Ni_4Ti_3 was not observed but still there was little NiTi₂. When the



Fig. 4. XRD patterns of porous NiTi SMAs, (a) the specimen synthesized, (b) the specimen applied load of 3.2 MPa at $1050 \degree C$ for 1 h and (c) the specimen applied load of 6.41 MPa at $1050 \degree C$ for 1 h.

load on the specimen was raised up to 6.41 MPa during solution treatment, Ni₄Ti₃, Ti₂Ni and B19'(NiTi) phases disappeared and the microstructure transformed to B2(NiTi) phase (Fig. 4 (curve-c)).

Zhu et al. [26] reported free nickel beside TiNi and intermetallics such as Ti_2Ni and Ni_3Ti from XRD results when sintering time was short. They also reported that free Ni disappeared, and the specimen was composed mainly of NiTi phase a small amount of other intermetallics when the sintering time exceeded 8 h.

Recently, Biswas [27] has proposed that the second phase (Ni₃Ti, NiTi₂) can be eliminated completely by a post-reaction heat treatment. While unreacted Ni disappeared the Ni₁₄Ti₁₁ and Ni₃Ti are found distributed within NiTi after post-reaction heat treatment at 1050 °C, the specimen post-heat treated at 1150 °C is clearly single phase. This is because of the eutectic temperature of NiTi₂ and N₃Ti is 984 and 1118 °C, respectively. The higher post-treatment temperature of 1150 °C induced the dissolution of undesired phases [28]. During the formation of NiTi, NiTi₂ and Ni₃Ti phases exothermic reactions occur as given below [26]:

Ni + Ti -	\rightarrow NiTi + 67 kJ/mol	(1)

$$Ni + Ti \rightarrow NiTi_2 + 83 \text{ kJ/mol}$$
 (2)

$$Ni + Ti \rightarrow Ni_3Ti + 140 \text{ kJ/mol}$$
 (3)

In the binary Ni–Ti phase diagram, NiTi, NiTi₂ and Ni₃Ti phases are stable phases. Moreover, according to the amount of energy exerted during the formation of phases, Ni₃Ti and NiTi₂ phases are more thermodynamically favoured than NiTi. Consequently, it is difficult to completely remove Ni₃Ti and NiTi₂ phases from the synthesized sample only by altering heat treatment [26]. In our study, a single phase porous B2(NiTi) was obtained by solution treatment under loading at 1050 °C. This situation is harmonious with EDS and XRD results.

Fig. 5 shows the result of DSC measurement for the non-solution treated specimen. The broad peak in the heating curve corresponds to the martensite (monoclinic) to austenite (cubic) transformation, and the broad peak on the cooling curve corresponds to the transformations from austenite to martensite. There are two peaks in the first heating curve corresponding to B19' \rightarrow R (intermediate trigonal martensite) \rightarrow B2 transformation. The first peak disappeared in the second heating curve. A similar transformation, B19' \rightarrow R \rightarrow B2 transformation of Ni₄Ti₃ after aged at 400 and 450 °C and B19' \rightarrow B2 transformation after aged at 475, 500 and 550 °C, was determined by Chu et al. [18]. It is thought that the incoherent stress fields in the structure disappeared after the first heating-cooling cycle, thus one-step (B19' \rightarrow B2) transformation occured.

A one-step transformation from the high-temperature parent phase B2 to the low-temperature monoclinic martensite phase B19' in the cooling curves is seen in Fig. 5. For equiatomic dense NiTi

Table 1									
Measured DSC transformation temperatures and associated energies for porous NiTi SMA synthesized.									
U a atim m mata	A (0C)	A (0C)	A (8C)	M (C)	M (°C)	M (C)	A $TTM \rightarrow A$		

Heating rate (°C/min)	<i>A</i> _s (°C)	<i>A</i> _p (°C)	$A_{\mathrm{f}}(^{\circ}\mathrm{C})$	<i>M</i> _s (°C)	<i>M</i> _p (°C)	$M_{\rm f}(^{\circ}{ m C})$	$\Delta H^{M \rightarrow A}$ (J/g) heating	$\Delta H^{A \rightarrow M}$ (J/g) cooling	$\begin{array}{l} \Delta S^{\mathrm{M}\rightarrow\mathrm{A}} \\ (\times10^{-3}\mathrm{J/gK}) \end{array}$	$\begin{array}{l} \Delta S^{\mathrm{A}\rightarrow\mathrm{M}} \\ (\times10^{-3}\mathrm{J/gK}) \end{array}$
5	91.86	99.63	107.26	67.64	65.10	60.58	-0.744	0.782	-2.064	2.169
10	85.81	96.11	103.90	67.13	62.54	53.97	-0.985	1.053	-2.747	2.937

alloys, the similar martensitic transformation usually is seen in the cooling curve. Two-steps or multi-stage martensitic transformation (MST) can also occur in dense NiTi alloys after certain thermal or mechanical treatments, which can be described as $B2 \rightarrow R, R \rightarrow B19'$, or $B2 \rightarrow B19'$ [15].

After solution treatment under loading no evident peak was seen in the temperature range (-50 and 140 °C) in DSC analysis. This situation shows that the parent NiTi phase becomes richer in Ni due to the disappearance of Ni₄Ti₃ with solution treatment under loading decreased the transformation temperatures. If Ni ratio decreases in parent phase due to the formation of Ni-rich Ni₄Ti₃ phase with aging at lower temperature, the transformation temperature again increases.

Transformation temperatures and associated energies measured by DSC for porous NiTi SMA synthesized are given in Table 1. The transformation temperatures and transformation enthalpies were evaluated with DSC. Transformation entropies were calculated with Eqs. (4) and (5) by taking into consideration the equilibrium condition in a system at constant temperature and pressure [28].

$$\Delta G = \Delta H - T_0 \,\Delta S = 0 \Rightarrow \Delta H = T_0 \,\Delta S \tag{4}$$

$$T_0 = \frac{M_{\rm s} + A_{\rm f}}{2} \tag{5}$$

 ΔG , ΔH , ΔS and T_0 are Gibbs energy change, enthalpy change, entropy change and equilibrium temperature, respectively. A_s and $A_{\rm f}$ are austenite start and finish, $M_{\rm s}$ and $M_{\rm f}$ are martensite start and finish temperatures, respectively. The transformation temperatures of porous NiTi fabricated by SHS are higher for the implants. The phase transformation temperatures decrease with the second thermal cycling, but the enthalpy and entropy of the transformation increase in absolute value. The changes in enthalpy and entropy for $B19' \rightarrow B2$ transformation are lower than those for $B2 \rightarrow B19'$ transformation. This result reveals that the heat required for $B2 \rightarrow B19'$ transformation is greater than that of $B19' \rightarrow B2$ transformation. Jiang and Rong [5] determined that the transformation temperatures decreased very little with thermal cycling up to 10 times.

(mV) 10 °C/min 4.00 Flow Heat 5 °C/mi M Cooling 2.00 0.00 Heating 5 °C/min -2.00 10 °C/min Temperature (°C) 0.00 40.00 80.00 120.00

Fig. 5. DSC curves of porous NiTi SMA synthesized at 200 °C after green pressure of 100 MPa

Therefore, we did not attempt more than two cycles in this study. The transformation temperatures of the specimens solution treated under loading could not be determined in the working range of DSC device since they are lower than -50 °C.

Prymak et al. [29] determined that A_p and M_p (peak temperatures in heating and cooling curves, respectively) for porous NiTi fabricated by SHS were 62.5 and 22.8 °C, respectively, which are lower than those of our study, but the width of the peaks was low (nearly 0.05 mW/mg). That is, the shape memory effect of specimens fabricated by them was little. In addition, it was determined by Jiang and Rong [5] that A_s and A_f points for austenitic transformation were 92 and 103 °C, respectively, and also M_p was 67 °C for porous NiTi fabricated by SHS. The transformation temperatures determined by them are closer to the transformation temperatures determined in our study.

Barrabes et al. [30] fabricated porous NiTi by SHS and determined that A_s and A_f points for austenitic transformation with a broad peak (30 °C) were 73 and 103 °C, respectively and the enthalpy of austenitic transformation was 2.28 J/g for the untreated NiTi. At the same time, they determined that $M_{\rm s}$ and $M_{\rm f}$ for martensitic transformation were 69 and 49.6 °C, respectively, and transformation enthalpy was 2.26 J/g. The transformation temperatures determined by them are closer to the transformation temperatures in our study and it is seen that the transformation hysteresis (between difference A_p and M_p) and enthalpies explained by them are higher.

4. Conclusions

- 1. Porus NiTi SMAs free from pure Ni, pure Ti and Ni₃Ti phases were fabricated by SHS. The distribution of the pores was uniform and most of the pores were isolated and rarely interconnected.
- 2. In case of loading during the solution treatment, undesired intermetallics disappear nearly completely. Porous single phase B2(NiTi) SMA with high chemical homogeneity was obtained by the load applied during solution treatment, but still more studies are needed to determine the effects of the load applied during solution treatment on dissolution of intermetallics.
- 3. The phase transformation temperature of synthesized specimen is higher for implant material. The phase transformation temperatures of specimen solution treated under loading decrease due to richer parent phase in Ni.

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