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Shape memory characteristics of porous Ti-Ni-Mo alloys prepared by solid state sintering

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ABSTRACT

$Ti_{50}Ni_{49.7}Mo_{0.3}$, $Ti_{50}Ni_{49.6}Mo_{0.4}$ and $Ti_{50}Ni_{49.5}Mo_{0.5}$ alloy fibers were prepared by a melt overflow process. Two-step B2-R-B19′ transformation was observed in the rapidly solidified Ti-Ni-Mo fibers. Upon increasing the Mo-content from 0.3 to 0.5 at.%, the austenite transformation finish temperature (A_f) of R → B2 decreased from 43 to −42 °C. Porous shape memory alloy pellets with 75% porosity were fabricated by a vacuum sintering technology, using the alloy fibers. Mechanical properties and shape memory effect of the highly porous $Ti_{50}Ni_{49.6}Mo_{0.4}$ alloy, of which A_f is lower than room temperature, were investigated using a compressive test. The plateau of a stress-strain curve was observed at about 2 MPa and resulted in 5% elongation associated with stress-induced martensitic transformation. It was also found that a recovered strain was 1.8% on heating after the compressive deformation. Because of the high porosity of this specimen, an elastic modulus of 0.91 GPa could be obtained.

1. Introduction

Ti-Ni-based shape memory alloys have been widely used in a number of different fields, such as mechanical and electrical applications, as well as aeronautics and astronautic field [1]. In addition, it has also been found that TiNi intermetallic alloys exhibit good corrosion resistance and high wear resistance, making them a superior tribo-material [2]. In recent years, Ti-Ni alloys have been extensively applied as metallic biomaterials in making biomedical apparatus and implant devices, due to their good biocompatibility and biological properties as well as their unique shape memory effects and superelasticity [3]. Ti-Ni alloys in porous form have attracted an additional interest as biomaterials for implantation since the introduction of pores into the bulk material provides in-growth of living tissues and firm fixation in addition to reducing the alloy density [4].

Powder metallurgy is known to be a promising method for the production of porous near-net-shape components [5,6]. It was reported that the porosity produced by self-propagating high temperature synthesis (SHS) of elemental Ti and Ni powders as well as by powder metallurgical process of Ti-Ni alloy powders was less than 25% [5,7]. However, porosity levels would need to be relatively higher, in order to bring down the stiffness of the metallic materials sufficiently to match that of bone [8]. Such high porosity would also favor bone in-growth, and hence good adhesion. In this study, a new method to fabricate the highly porous shape memory alloys is proposed. Fine Ti-Ni-based alloy fibers were prepared by a melt overflow process. The porous bulk specimens, which have three-dimensional network structure, interconnected pores and high porosity, were produced using the solid-state sintering of the fibers and the effect of the high porosity on the mechanical performances was investigated using compressive tests. In order to use the excellent superelastic property of shape memory alloys in many medical applications, the austenite transformation finish temperature must be less than 37 °C, which is human body temperature. The transformation temperatures were also controlled by substitution of Mo for Ni in Ti-Ni alloy in this study.

2. Experimental procedures

$Ti_{50}Ni_{49.7}Mo_{0.3}$, $Ti_{50}Ni_{49.6}Mo_{0.4}$ and $Ti_{50}Ni_{49.5}Mo_{0.5}$ alloy ingots were prepared from high purity elements of nickel, molybdenum and sponge titanium using an arc melter in a water-cooled Cu hearth. The alloys were re-melted several times under the high purity of argon atmosphere to ensure the homogeneity. The experimental studies were performed using an arc melt overflow rapid solidification process. The mother alloys was placed in a

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water-cooled hearth, and then skull melted under an argon atmosphere using a plasma beam. The hearth was then tilted about the rotating quenching wheel. The molten metal overflowed over a relatively horizontal edge to make contact with a quenching wheel surface. The quenching wheel substrate served as a continuous permanent mold. The diameter of the molybdenum quenching wheel is 122 mm and the linear speed of the wheel was maintained at 7.7 m/sec to produce relatively thin fibers. Microstructural and compositional investigations were performed using scanning electron microscopy (SEM) and energy dispersive X-ray spectrum (EDS). Transformation temperatures were measured using differential scanning calorimetry (DSC) at a cooling and heating rate of 10 °C/min and crystal phases were identified using X-ray diffraction (XRD), with CuKα radiation.

The fibers were cut into segments, with the length ranging from 5 to 10 mm. The fibers were uniformly placed into the predetermined packing chamber of a mold-pressing equipment, and pressure was then applied by screwing the bolts. Sintering was carried out in a vacuum induction furnace. The sintering temperature and time were 1200 °C and 30 min, respectively. The porous pellets of $10 \times 10 \times 18\ mm^3$ tetragonal shape were then fabricated for the compressive tests. The mechanical behaviors of the porous shape memory alloys were investigated using uniaxial compression experiments with a speed of cross head of 0.24 mm/min.

3. Results and discussion

Rapidly solidified fibers were prepared by the melt overflow processing and the continuous fibers of about 50 μm diameter were shown in Fig. 1(a). The columnar microstructure was observed in a typical microstructure of etched longitudinal section in Fig. 1(b). Because the melt came into contact with the quenching wheel during the high speed casting of the melt overflow system, the heat would be extracted to the direction of the wheel during solidification, so that the fibers consisted of long grains, crystallographically oriented with their columnar directions normal to the surface, which was the reverse direction of the heat-extraction. Fig. 1(c) shows a typical shape of round cross-section of the fiber.

Fig. 2 shows DSC curves of $Ti_{50}Ni_{49.7}Mo_{0.3}$, $Ti_{50}Ni_{49.6}Mo_{0.4}$ and $Ti_{50}Ni_{49.5}Mo_{0.5}$ alloys. It is seen that all DSC curves show two exothermic peaks on cooling and one endothermic peak on heating. In order to explain the transformation behaviors of DSC peaks in Fig. 2(a)–(c), XRD experiments were carried out as $Ti_{50}Ni_{49.7}M_{0.3}$ alloy fibers being cooled. Fig. 3 shows the analytical results of XRD patterns as a function of the temperature. Only the diffraction peaks corresponding to B2 (cubic) parent phase are found in an XRD pattern at 50 °C.The diffraction peaks of R (trigonal) martensite start to appear at lower temperature (20 °C). The diffraction peaks of the B2 phase disappears and the diffraction

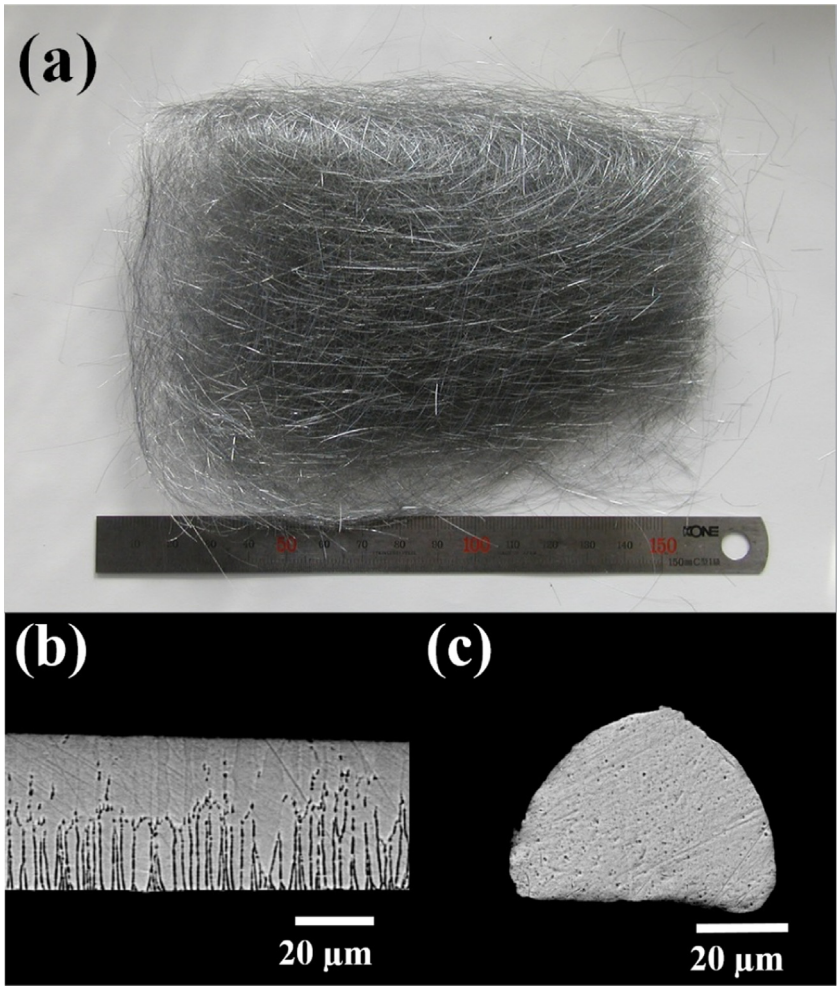

Fig. 1. (a) Photograph and SEM micrographs of (b) etched longitudinal section and (c) cross-section of Ti-Ni-Mo alloy fibers.