

Martensitic transformation and microstructure of Ti-rich Ti–Ni gas-atomized powders

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Abstract

Martensitic transformation was investigated for Ti-rich Ti–Ni gas-atomized powders as a function of powder size. The large powders clearly exhibit multiple martensitic transformations on both cooling from austenite to martensite and vice versa. With increasing powder size, the minor martensitic transformation temperatures increase and the amount of heat accompanying the minor transformation also increases. However, the main martensitic transformation temperature is almost the same, irrespective of the powder size. Microstructures of the as-atomized powders were examined to clarify why the powders exhibit the multiple martensitic transformations.

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1. Introduction

Shape memory alloys (SMAs) are one of the most successful functional materials. They have been studied for several decades and are considered to be candidates for actuator materials, because they can produce large recovery strain and force via the martensitic phase transformation [1–4]. Among the various kinds of SMAs, TiNi is one of the most attractive, and has been intensively investigated as it exhibits superior shape memory properties at around room temperature. Thus not only bulk specimens but thin films and powders of TiNi have been prepared and studied as practical materials. The mechanical properties and microstructures of TiNi thin films have been examined in order to apply TiNi to microelectromechanical systems (MEMS) [5–15]. For powders, consolidation methods, such as spark plasma sintering (SPS) and hot isostatic

pressing (HIP), have been to prepare bulk near-net-shape TiNi [16–26]. However, consolidation of elemental powders of Ti and Ni often results in substantial porosity [27] in synthesized bulk TiNi. Recently, SMAs have been also studied as damping materials or high energy absorption structural materials [28–30], and it is considered that porous TiNi bulk specimens are especially suitable for damping materials [31]. Hence, consolidation is appropriate for preparing high damping materials or high energy absorption materials. However, consolidation of Ti and Ni elemental powders often yields TiNi₃, whose effect on shape memory properties is not clear [21,27,32,33]. Consolidation of TiNi alloy powders prevents such secondary phases from precipitating in the synthesized TiNi bulk porous specimens and improves their shape memory properties. It is therefore necessary to examine the martensitic transformation behavior and microstructures of TiNi alloy powders before consolidation.

In the present study, Ti-rich Ti–Ni powders were prepared by gas-atomization of arc-melted Ti–Ni ingots, and

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martensitic transformation temperatures and microstructures of those powders were examined as a function of powder size.

2. Materials and methods

Ti–Ni binary shape memory alloy powders were prepared by gas-atomizing arc-melted Ti–49.0Ni (at.%) ingots in an Ar atmosphere. Martensitic phase transformation temperatures of the powders were measured by differential scanning calorimetry (DSC) at a cooling and heating rate of 0.167 K s^{-1} . The as-atomized powders were kept at 423 K for 180 s in advance of DSC measurement. Then they were first cooled to 223 K, followed by heating to 423 K. The crystallization temperature, T_x , was also evaluated by DSC at a heating rate of 0.667 K s^{-1} . DSC measurement was performed several times on each specimen, but the error in the martensitic transformation temperatures was less than 2 K at most. Phases in the powders were identified by X-ray diffractometry (XRD). The powders were also examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Composition of the phases in the powders was measured with an electron probe microanalyzer (EPMA) equipped with several wavelength dispersive spectrometers. XRD measurement, SEM and TEM observation, and EPMA analysis were performed at room temperature, about 290–297 K. The powders were kept at room temperature, lower than 303 K, during the present study.

A portion of the atomized powders (approximately 10 g) was put through sieves with fine meshes ranging from 20 to 300 μm . The weight of the sifted powders is given in Table 1. In this study, the sieve mesh size is used in place of the actual size of the powders. Most of the powders are smaller than 100 μm . Powders smaller than 150 μm were examined in this study because the sifted powders larger than 150 μm contained a large number of flakes or needles, and because the weight of the larger powders was not enough for the experiments. Those flakes and needles were removed and were not used in this study.

Small amounts of the powders were manually ground using a mortar for XRD measurement to confirm the phases present inside the powders. Some as-atomized powders were embedded in epoxy resin and were polished for observation of cross-sections by means of SEM and

EPMA. Other as-atomized powders were embedded in Ni prepared by electroless plating [34], and their microstructure was examined by TEM after thinning by ion-milling.

3. Results

3.1. Behavior of martensitic transformation

Fig. 1 shows DSC traces of the Ti–Ni as-atomized powders as a function of sieve mesh size, i.e. powder size. The powders smaller than 125 μm exhibit almost one sharp and narrow peak (denoted as *M1* in Fig. 1) caused by martensitic transformation on both cooling and heating. Another exothermic heat peak (*M2*) is clearly observed for the powders ranging between 45 and 125 μm . This exothermic heat peak is also found in the powders smaller than 45 μm but is faint. The 90–150 μm powders exhibited two additional exothermic heat peaks (*M2* and *M3*) in addition to the *M1* major peak. The major peak of the 125–150 μm powders is considerably broader than others.

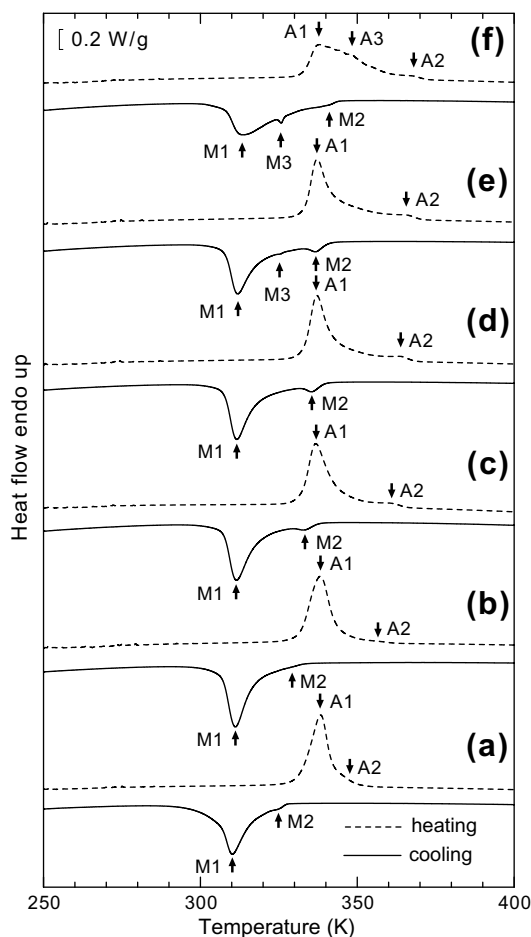


Fig. 1. DSC traces of the as-atomized powders as a function of powder size. (a) 0–20 μm , (b) 20–45 μm , (c) 45–75 μm , (d) 75–90 μm , (e) 90–125 μm , (f) 125–150 μm .

Table 1
Weight of Ti–49.0Ni as-atomized powders sifted by sieves

Mesh size (μm)	Weight (g)
0–20	0.938
20–45	3.819
45–75	3.669
75–90	0.654
90–125	0.559
125–150	0.027
150–300	0.092
~300	0.041

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