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Original Research Paper

Densification and microstructural evolution of spark plasma sintered NiTi shape memory alloy

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A B S T R A C T

The effect of particle size and sintering temperature on the densification and microstructural characteristics of nickel-titanium shape memory alloy (*NiTi*-SMA) has been investigated using spark plasma sintering (SPS) process. The *Ni* and *Ti* elements in different particle sizes were alloyed in the composition of $Ni_{50.6}Ti_{49.4}$. The milled *NiTi* powders were consolidated using SPS process in a temperature range of 700–900 °C. The densification was characterized by plotting temperature, current and relative displacement of punch as a function of holding time. The results showed that a maximum relative density of ~98% can be achieved for *NiTi*-SMA with an average particle size of 10 μm at a sintering temperature of 900 °C. The microstructure of the sintered *NiTi*-SMA was examined using scanning electron microscope (SEM) and composition of *NiTi* alloy was analyzed using energy dispersive spectroscopy (EDS) analysis. The effect of sintering temperature on the microstructural evolution and transformation was also studied.

1. Introduction

The nearly equiatomic *NiTi* alloys find application especially in biomedical fields as repairing and replacement of human hard tissue and bone staple with beneficial properties such as superelasticity (SE), shape memory effect (SME), biocompatibility and corrosion resistance [1,2]. The unique properties such as SE and SME were executed by thermo-elastic martensite transformation and its reversion, from high temperature austenite *(B2)* to low temperature martensite phases *(B19′)* and vice versa. This martensite transformation is affected by the *Ni* grain size and the content of *Ni* in *NiTi* composition [3,4]. Moreover, the grain size in the bulk material can be altered by the influence of temperature during the sintering process. Thus, the investigation of particle size and sintering temperature has a significant role during the sintering of *NiTi* alloys.

In the recent past, various powder metallurgy methods such as self-propagating high-temperature synthesis (SHS) [5], microwave sintering [6], Metal injection molding [7], conventional sintering [8] and selective laser melting [9] have been used to facilitate *NiTi* shape memory alloys. The final density obtained for the fabricated materials determines efficiency of the sintering process. SPS was used to sinter the materials to its full density in a short time at a lower temperature [10,11]. Higher densification of materials can be achieved during SPS process by making effective contact between the particle surfaces. Evaporation and melting with the application of load during SPS resulted in the attainment of effective contact between particles [12]. During SPS process, the heat is generated by the flow of pulsed current through graphite punch and dies and finally in to powder particles. Thus, the discharge can be created between the powder particles that causes the generation of higher temperature on the contact surfaces of particles which resulted in local melting. The necks were formed between the powder particles when the melted zones bonded and solidified due to rapid transfer of heat along the surfaces. As the sintering temperature is maintained for a specific period of time for a uniform external pressure, diffusion takes place in the surface and grain boundary. Hence, a highly densified product could be made by SPS process at a shorter time as compared with other conventional sintering techniques such as vacuum arc melting, vacuum induction melting and microwave sintering [13–16].

The SPS parameters such as pressure, temperature, time, pulsed current and voltage influence the mechanical and microstructural characteristics of the Sintered products. An earlier investigation carried out to evaluate the effect of particle size, pressure and sintering temperature on densification of *Cu* powder concluded that the densification was mainly influenced by the particle size and

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sintering temperature [17]. The deformation, specific surface area of particles, local pressure and current intensity in SPS technique were influenced by the size of powder particles [18]. Increasing the sintering temperature from 1000 to 1100 °C, the density was found to be increasing from 97 to 99% for *Ti* alloy produced by SPS technique [19]. Similarly, the relative density of tungsten powder processed by SPS was increased from 81% to 95% with increasing the sintering temperature from 1600 to 1800 °C [20]. The formation of desirable phase of austenite (*B2*) in *NiTi* can be increased with the increasing of sintering temperature along with the reduction of undesirable secondary phases such as Ni_3Ti and Ti_2Ni [21]. Thus, the density and microstructure of the product sintered by SPS process are significantly affected by the sintering temperature and particle size.

In the present work, *NiTi* shape memory alloy was sintered at different temperatures (700, 800 and 900 °C) and particle sizes (45 and 10 µm). This investigation aimed to study the influence of sintering temperature and the particle size on the densification and microstructural characteristics of the *NiTi* binary alloy using SPS process.

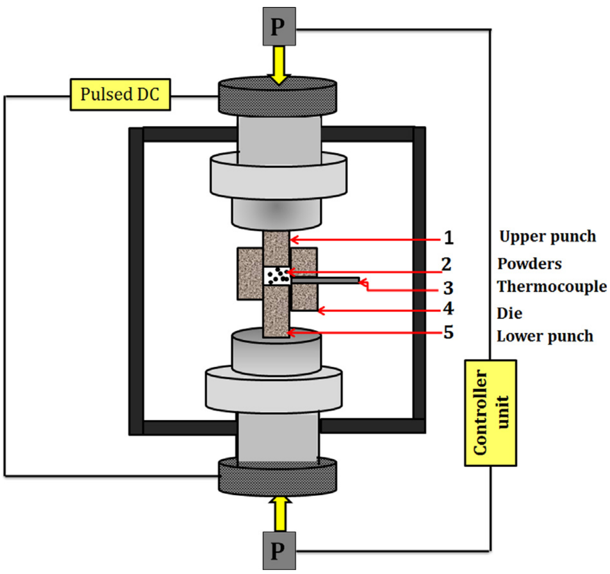

Fig. 1. Schematic view of SPS-setup.

2. Experimental details

Ni powder (Particle size: 37–40 µm, Purity: 99.8 %) and *Ti* powder (Particle size: 44–47 µm, Purity: 99.5 %) were procured from the M/s Alfa Aesar – United States. The elemental powders were mixed to develop the alloy system with the composition of *Ni*-50.6, *Ti*-49.4. These *Ni* and *Ti* elements were blended and subsequently mechanical alloyed for 30 h in a two station high energy planetary ball mill (Insmart Systems MBM 07, twin bowl). The nanostructured alloyed elements were synthesized using a ball to powder weight ratio of 10:1 at a milling speed of 200 rpm. Thereafter, the powders were sintered in two variants such as (i) as mixed $Ni_{50.6}Ti_{49.4}$ and (ii) nanostructured $Ni_{50.6}Ti_{49.4}$. The granulated powders were placed into a graphite die of 15 mm diameter then consolidated by using the SPS equipment (Dr. Sinter 515S apparatus, SPS Syntex Inc., Kanagawa, Japan); the schematic diagram of SPS tooling setup is shown in Fig. 1. The sintering process was carried out at different temperatures (700, 800 and 900 °C) in a vacuum atmosphere with a heating rate of 50 °C/min. The pressure was maintained at 30 MPa throughout the sintering process.

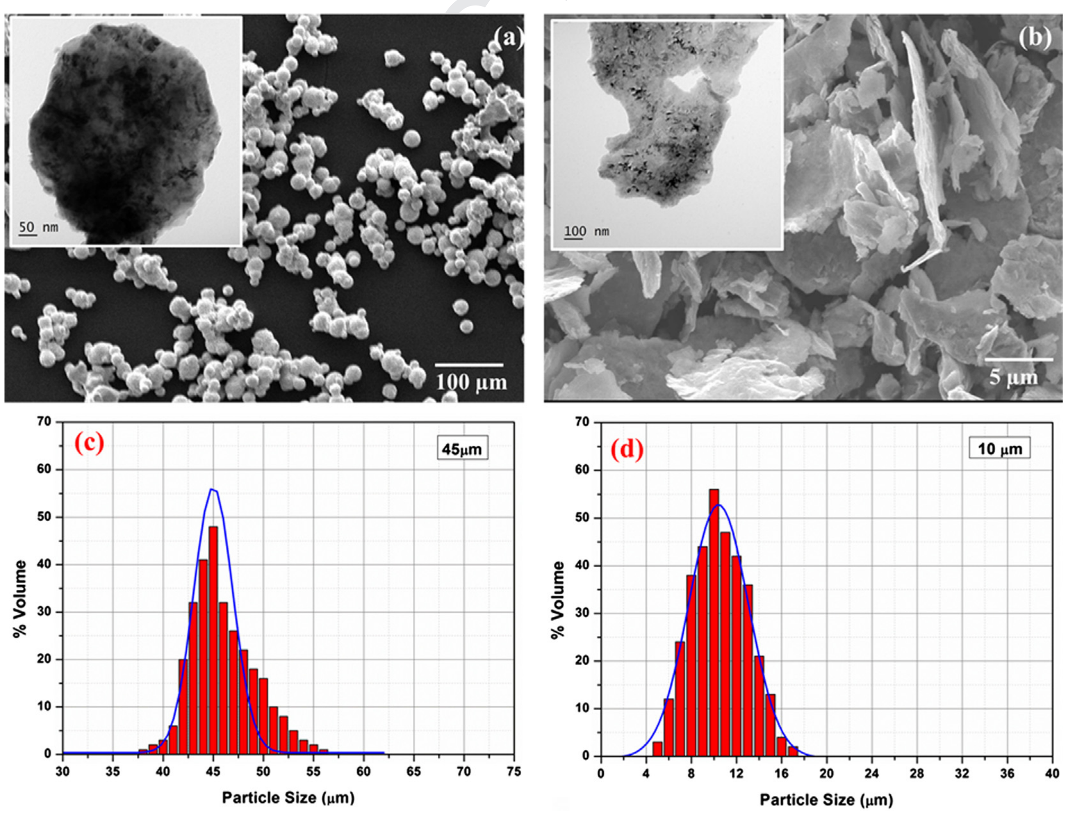

Fig. 2. SEM and PSA of NiTi powder elements with (a, c) 45 µm (b, d) 10 µm in size.

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