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Studying Wear Behavior of Ni-Ti- Ag Shape Memory Alloy Synthesized by P/T

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K E Y W O R D S

Shape memory alloy. Ni-Ti matrix, nano-Ag, microstructure, hardness, wear.

A B S T R A C T

Because of the unique properties, Ni-Ti based shape memory alloys (SMAs) are increasingly attractive for a wide variety of engineering applications such as actuators, biomedical, or robot coupling. In this work, a third alloying element, namely nanoparticles of Ag (which is insoluble in Ni-Ti matrix), is added by powder technology to the Ni-Ti alloy to produce a Ni-Ti-Ag alloy. The Nanoparticles of the Ag element are added at 3, 5, 7, and 10 wt. % to produce four alloy specimens with different mixtures .The mixing process was done by a horizontal mixer for 120 min with a speed of 350 rpm, and then the mixture was compacted by using a compacting pressure of 600 MPa. Afterward, the compacted specimens were sintered at 600℃/min for 6 hrs. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to evaluate the microstructure and phases of the products. DSC examination was used to characterize the phase transformation temperatures in heating and cooling. Wear behavior was defined by using the pin-on-disc technique, and the hardness of the samples was calculated using Vickers's hardness apparatus. The results of this work showed that the nano-Ag added at 7 and 10 wt. % were distributed homogeneously in the Ni-Ti matrix, and that Ag slightly decreased hardness and increased the wear rate. The value of shape memory effect (SME) for the produced alloy was about 89.9% and the phase transformation in heating was at a temperature of about 186.48 ℃ and in cooling of about 140.3 ℃ for the specimen that contains 10 wt.% Ag nanoparticles.

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1. Introduction

For many decades, nickel-titanium alloy is considered as an important alloy in many industrial applications because of its shape memory effect (SME) [1]. The nickel-titanium alloy was first developed in the1960s. This alloy was named Nitinol, an acronym for the elements from which the material was composed (Ni for nickel and Ti for titanium) and the location for these investigations [2]. There are many families of SMAs among which are Ni-Ti, Cu-Al-Ni, Fe-Mn-Si, and Cu-Zn-Al. The properties for these alloys can be controlled and changed by different methods of thermo mechanical processing of the composting materials so that each of these alloys has its unique properties. Ni-Ti and Cu-Zn-Al are the most shape memory alloys used in many industrial applications [3]. Many methods are used to manufacture shape memory alloys such as casting and powder technology. Powder metallurgy technology is the main depended on technology to produce and develop shape memory alloy [4,5]. In general, powder metallurgy plays an important role in technologies of materials processes and in the formation of product forms and properties of engineering materials [6]. It is considered a highly developed method of manufacturing reliable ferrous and nonferrous parts, and it is a continually and rapidly evolving technology embracing most metallic and alloy materials, and a wide variety of shapes [7]. Thus, for this fact, powder metallurgy is used to synthesize the Ni-Ti-Ag alloy in this work. Ni-Ti has an attractive interest because of its reversible shape recovery which is more than 8% [8]. The transformation of austenite (As-Af) to martensite (Ms-Mf) of shape memory alloys is reversible and usually occurs at a temperature of about 50-110 °C depending on the content of Ni in the matrix (48-50wt.%) [9]. A third element could be added to improve the phase transformation and then enlarge the applications of these alloys. For the last years, many investigations were published about ternary Ni-Ti-X alloys with X=Au, Fe, V, Pd, Pt, Cr, or Cu. These elements can improve the shape memory effect (SME), super elasticity (SE), and phase transformation. The third element substitutes either Ni atoms in the intermetallic lattice of Ni-Ti, such as Fe, pt, pd or Au, or substitutes Ti atoms, like Mn, V, or Cr. Ni substituents like pt, pd or Au, increase the phase transformation temperature, while Ti substituents like Mn, V, or Cr decrease the phase transformation temperature. The substitution element, like Cu, may be used to stabilize the phase transformation temperature [10]. Nb is partially soluble in Ni-Ti intermetallic lattice, and hence, it increases the hysteresis of phase transformation. Moreover, because of the limited amount of investigations regarding Nb, it is replaced by Ag or W element. The difference in melting temperatures of Ag (961 oC) and W (3422 oC) compared with that of Ni-Ti (1310 oC) as well as the difference in densities of the third element, (10.49g/cm3) for Ag, and (19.3g/cm3) for W compared with the density of Ni-Ti (6.45 g/cm3), it is expected that the addition of these elements will extremely affect the processing of Ni-Ti-Ag or Ni-Ti- W [11].
The present work aims at preparing Ni-Ti-nano/Ag SMA by using powder technology and to study the microstructure, hardness, and wear behavior of the resultant shape memory alloy.

2. Experimental Procedures

I. Materials used

In the present work, Ni-Ti powders (manufactured by Sky Spring Nano Materials Company - USA) were used with Ni and Ti percentage content is 50 wt.% for each. The elemental nano-Ag powder was used as an alloying element, and it was used with different weight percentages of 3, 5, 7, and 10 wt.%. The grain sizes of Ni and Ti were 40, 50 μm respectively, while the grain size of nano-Ag was 25 nm. The raw powders of Ni-Ti were blended with the elemental nanoparticles Ag for each weight percentage. The mixture of the powders was blended in a turbulent mixer for 2 hrs., with adding ceramic balls and ethanol. After the mixing process, each of the four powder mixtures was compacted by using uniaxial pressing at 600 MPa in a cylindrical steel mold with 12 mm diameter, and length of about 30 mm. The dimensions of specimens used in hardness and wear tests were 10 mm in diameter and 10 mm in length. Afterward, the green compacts were sintered at 600oC/min for 6 hrs under argon atmosphere.

II. Microstructural analysis

After the preparation of the specimens, a microstructure analysis for all specimens was done by using scanning electron microscopy (SEM). Also, the phases' formation of all of the specimens before and after adding nanoparticles Ag were examined by using X-Ray diffraction analysis with (Cu, K_α radiation with the wavelength of 1.54060A). Besides, differential scanning calorimetry type (DSC: TA-1A) (Measures were done at Amirkabir University of Technology/ Tehran - Iran) was used to

determine the phase transformation temperatures for heating and cooling cases, the temperature range was about + 300-(-100) oC.

III. Hardness Test

Hardness test before and after adding Ag nanoparticles has been carried out by using Vicker's hardness apparatus according to ASTM E384 standard. Three readings at least were taken for each specimen at the surface of the specimen, and then the readings were averaged to achieve the average diameter of the indentation Vickers hardness [12].

IV. Wear test

A wear test was carried out by using a pin-on-disc technique to evaluate the wear rate for the specimens before and after adding Ag nanoparticles. Wear test was performed according to ASTMG99, and it was carried out by changing the load as 2, 4, 6, 8, and 10 N. The sliding wear was carried out by using a disc made of hardened tool steel at hardness 45 HRC and rotating at 2950 rpm. Wear rates were measured before and after adding Ag nanoparticles by weighing specimens using a sensitive electronic balance with an accuracy of about 0.01 mg. The type of the device used was; mettle AE-60 (china made). Wear rates for all of the specimens were calculated using the following formula:

$$\text{Wear rate} = \square(\Delta w/(2\pi.r.n.t)) \quad (1)$$

$$\Delta W = w\_1 - w\_2$$

Where:

ΔW: the difference in weight (g).

w_1, w_2: the weight of the specimens before and after wear test(g) respectively.

r: the radius of the distance from the center of the specimen to the center of the disc.

n: rotating number of the disc.

t: time of the test (min)

V. Shape Memory Effect Test

The shape memory effect of the specimen containing 10% Ag nanoparticles was determined by compacting the specimen by 0.06% from its original length [13] using compression test device with 1mm/min displacement and followed by heating the specimen in the furnace at 120 C° for 5 Min and cooling in air. Then the shape memory effect was measured using the following equation [14].

$$SME\% = (L\_2 - L\_1)/(L\_0 - L\_1) \times 100 \quad (2)$$

Where:

L_0: the original length of the specimen (mm).

L_1: the length of the specimen after 0.06% compaction.

L_2: the return length of the specimen after heating.

3. Results and Discussion

I. Microstructure analysis

Figure 1 shows the microstructure of the compacted specimens before and after adding Ag nanoparticles with different concentrations. The images of SEM show the homogeneous distribution of Ag nanoparticles in the Ni-Ti matrix with a little number of pores. Since the melting temperature of nanoparticle Ag is low, the Ag nanoparticles are virtually melted and fill interstitial voids between Ni-Ti particles. It is observed that Ag particles surrounded Ni-Ti particles and there is no interdiffusion between them. This agrees with ref. [15].

The shape and distribution of the created pores depend extremely on the sintering temperature. This temperature causes good coalesce of Ag nanoparticles with Ni-Ti particles.

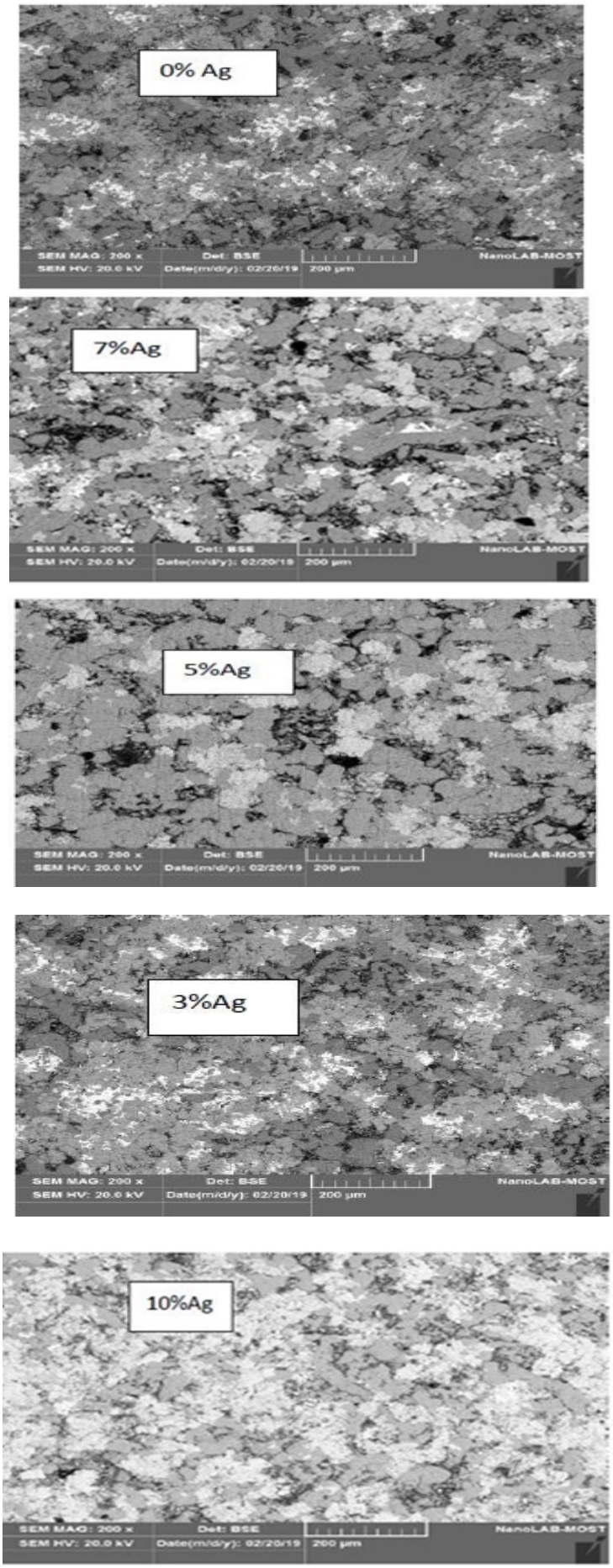
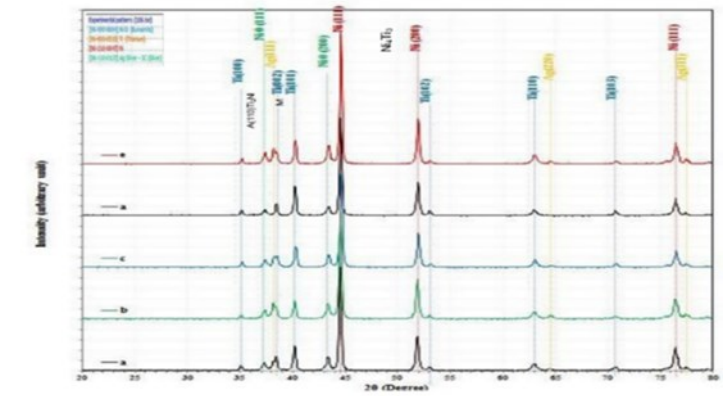
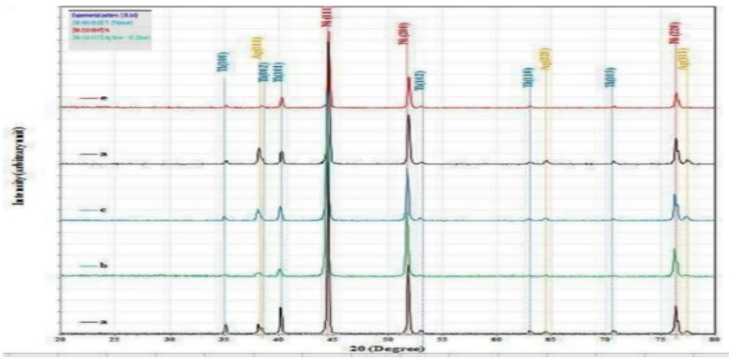


Figure 1: SEM Images of Ni-Ti-Ag SMA with Different Amounts of Ag Nanoparticals



(a)



(b)

(a) XRD before sintering; (b): XRD after sintering

Figure 2: X-Ray Diffraction.

II. X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) for the specimens before adding Ag nanoparticles and after adding Ag nanoparticles shows the creation of different phases after the sintering process. Before adding Ag nanoparticles and after sintering, the phases are austenite phase (Ti_2Ni) which is known as a cubic phase and this phase appears at heating. While, the other phase created as a result of phases transformation temperatures is a martensite phase (Ti 002) which is known as the monoclinic phase, and this phase appears on cooling the specimen in the furnace [16]. Furthermore, Ni_4Ti_3 phase may be created during the slow cooling of the specimens in the furnace. This phase can be removed by post-heat treatment to complete the phase transformation. The results of this work agreed with [17]. The XRD test was done for all of the sintered specimens before and after adding Ag nanoparticles. Figure 2 shows the XRD results with the basic data for all of the specimens after the sintering process with different amounts of Ag nanoparticles.

The phases of Ni and Ti elements are transformed into Ni-Ti martensite phase (known as monoclinic phase), Ti_2Ni austenite phase (known as cubic phase), and Ni_4Ti_3 phase (known as rhombohedra phase). The results show that there are two phases with high-intensity Ni-Ti martensite phase and Ti_2Ni austenite phase. Also, the results reflected the appearance of NiO phase as a result of sintering conditions. Also, after adding Ag nanoparticles it was noted the appearance of Ag phase which is responsible for the pseudo elasticity and shape memory effect. These results agree with those of ref. [18].

III. Differential scanning calorimeter (dsc) results

Differential scanning calorimeter examination was carried out for the sintered specimens before and after adding Ag nanoparticles to evaluate the phase transformation temperatures as starting and finishing austenite phase (As, Af), also the starting and finishing martensitic phases (Ms, Mf). Figure 3 shows the peaks which estimate the temperatures of the austenite and martensite transformations after heating or cooling. The given results are for the specimens containing 7% and 10% nono Ag.

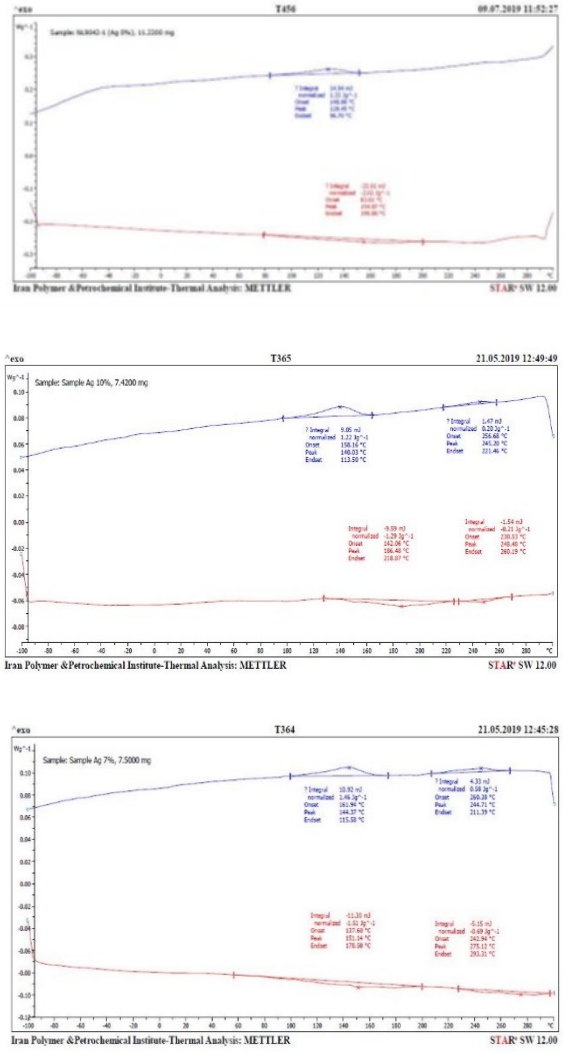


Figure 3: Results of DSC test for the specimens (tests were done in Iran)

For the specimens containing 7% nano-Ag, the onset and ending temperatures during cooling are about (161.94) ^{o}C and (115.58) ^{o}C, and the onset and ending temperatures during heating are about (137.60) ^{o}C and (178.50) ^{o}C While for the specimens containing 10% nano-Ag, the onset and ending temperatures during cooling are about (158.16) ^{o}C and (113.50) ^{o}C, and the onset and ending temperatures during heating are about (142.06) ^{o}C and (218.87) ^{o}C. These cases are listed in Table 1.

Table 1: Values of temperature for DSC test (at heating and cooling)

	Heating		**Cooling**	
Ag nanoparticle %	Starting (c°)	Finishing (c°)	Starting (c°)	Finishing (c°)
0	83.6	195.8	148.8	96.7
7	137.6	178.5	161.94	115.8
10	142.06	218.87	158.16	113.5

IV. Vickers hardness results

Vickers hardness test was carried out for all of the specimens before and after adding nanoparticle Ag with different weight percentages. The results of this test show that the hardness decreases with increasing the weight percentage of nano-Ag as shown in Figure 4. This is due to the increased shape memory effect (SME) which is about (89.95%). Also, one of the observations was a decrease in the defects in the resultant shape memory alloy such as micro cracks and porosity which are dependent extremely on the compacting pressure and the sintering temperature and then affect directly the hardness values. Another

physical phenomenon perhaps affects the hardness value such as binding energy and wettability between nano-Ag particles and Ni-Ti grains.

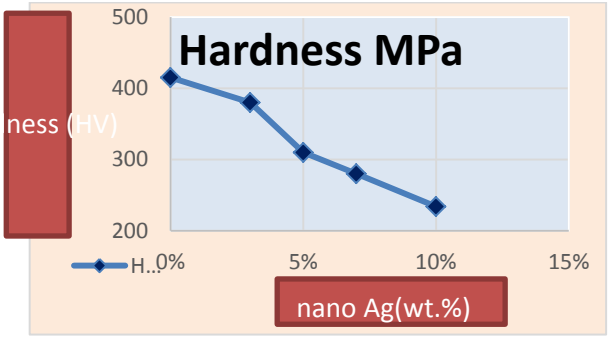


Figure 4: Variation of Vickers Hardness with Amount of Ag

V. Wear characteristics

Wear rates are increased with the increasing of the applied loads for all the specimens and as shown in Figure 5. It is clear that the wear rate slightly increases with increasing the amount of nano-Ag for all applied loads. The increase in wear rate is attributed to decreased hardness which in turn is due to the increasing Shape Memory Effect (SME) of Ni-Ti-nano/Ag shape memory alloy. The nonlinearity of curves will increase with increasing the applied loads, which as well rises the temperature between the pin and disc as a result of friction between them.

Increasing the applied load creates wear debris as a result of cracking and forming cavities at the surface. Also, increasing the load will break the oxide films and plough them to form the wear debris, and causes delamination and abrasive wear. As well, increasing the applied load will increase the shear stresses at the interfaces between Ag nanoparticles and the Ni-Ti matrix. The wear rate for Ni-Ti-Ag is slightly higher than Ni-Ti matrix. Moreover, the increase in the applied load leads to transferring wear mechanisms from mild wear to serve wear and makes more damage in the worn surfaces.

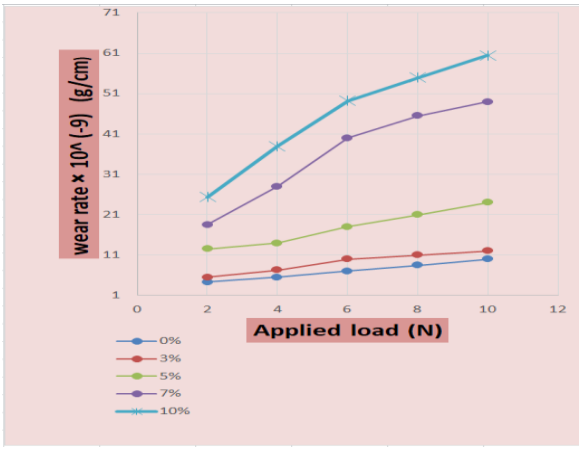


Figure 5: Relationship between applied load and wear rate for different Percentages of Ag Nanoparticles

4. Conclusions

This work aimed to synthesize and testing a Ni-Ti–nano/Ag shape memory alloy using a powder metallurgy route. Some of the concluded outcomes of the work are:

1. Increasing the percentage of Ag nanoparticles leads to improving shape memory effect (89.95%).
2. Wear rate is increased and the hardness is decreased with increasing Ag in Ni-Tii shapes memory alloy.
3. XRD results demonstrated the presence of austenite and martensitic phases with a little amount of NiO.

4.DSC results show that, for the specimen with 10wt.% Ag, the onset and ending phase transformation temperatures during heating are about (142.06) oC and (218.87) oC while the onset and ending during cooling are about (158.16) oC ,(113.5) oC .

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