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TECHNICAL NOTE

# Effect of Primary Microstructures during Training Producers on TWSME in NiTi Alloys

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# 1. INTRODUCTION

The term shape memory alloy (SMA) is called to the alloys which can remember their original shape during heating. TWSME which is one of the properties of the SMAs has attracted lots of interest by researchers due to its applications in different fields [1-2]. TWSME is not an inherent property of SMAs but it can only be obtained by a suitable thermomechanical treatment, usually called training. There are several training methods in order to obtain a TWSME. Common procedures of two-way memory training include the martensitic stress induced transformation (a pseudoelastic training), thermal cycling training under a constant stress and shape memory training (thermal cycling with loading and unloading) [1,3]. It is commonly accepted that through the training procedures, an anisotropic dislocation structure is developed in the matrix. This dislocation structure creates an anisotropic stress in the matrix which leads to the formation of martensite into various preferential orientations. This phenomena leads to introduce internal plastic deformation. It has been suggested that the internal plastic deformation is necessary to create two-

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# ABSTRACT

The influence of the martensitic, martensitic+austenitic and austenitic structures during bending training on two-way shape memory effect (TWSME) in Ni-50.8 at %Ti and Ni-49.9 at %Ti alloys has been studied. In addition, the effect of pre-strain, plastic strain, training cycle and training temperature on the TWSME has been investigated. Specimens were trained in martensitic, martensitic+austenitic and austenitic states with bending deformation. The results showed that in martensitic state, TWSME was more than the other states. Although the number of training cycles in martensitic state(10-15 cycle) were less than the other training methods (15-25 cycle), the obtained optimum pre-strain in martensitic state (16%) was more than other training methods (12%). In addition, it was shown that with increasing the training temperature in austenitic state, TWSME decrease.

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way shape memory effect. The magnitude of the twoway shape memory is found to depend on whether the training producers involve the formation of stress induced martensite or martensite reorientation [4, 5].

The influence of training parameters such as training cycle, pre-strain and primary microstructures on TWSME in Ni-49.8 at %Ti, Ni-50.2 at %Ti and TiNiCu shape memory alloys in tensile deformation were investigated by other researchers [5-7]. Furthermore, the bending deformation in martensitic state in TiNiHf alloy was studied by other researches [3]. The purpose of present work is to study the influence of training producers in martensitic and austenitic states by performing bending deformation in Ni and Ti-rich shape memory alloys.

### **2. EXPERIMENTAL**

Binary NiTi alloy with a nominal composition of Ni-49.9 at %Ti and Ni-50.8 at %Ti were prepared from technically pure metals by using vacuum induction melting in high density graphite crucible. The ingots were solution-treated and hot-rolled. The samples were then cut in  $4.5 \times 40 \times 100$  mm. The transformation behavior of samples was determined using differential scanning calorimetry (DSC) with the heating/cooling rate of 10°C/min. The microstructure observation was carried out with optical microscopy (OM) and scanning electron microscopy (SEM). XRD analysis was carried out to study the composition of the phases. In order to investigate the two-way memory behavior, the samples were bent against different cylindrical rods to introduce the different pre-strains (Figure 1). The pre-strain was calculated by  $\varepsilon_d = h/(h + d)$  where, h is the thickness of the sample and d is the diameter of the rod. The deformation strains in the study are 3, 6, 9, 12, 16 and 20%, respectively. The two-way shape memory strain is measured by  $\varepsilon_{tw} = (\theta_w - \theta_h)\varepsilon_d/180$ , where  $\theta_h$  is angle in heating position,  $\theta_w$  is angle in quenching position and  $\varepsilon_d$  is pre-strain. The plastic strain in the first heating cycle was calculated by  $\varepsilon_p=\theta_h \varepsilon_d/180$  [3,8].

The samples were trained in bending deformation in the martensitic, martensitic+austenitic and austenitic structures. Training in martensitic state was discussed elsewhere [8]. In martensitic+austenitic and austenitic structures, training procedures were carried out in full constrain i.e. the thermal cyclings were carried out, after bending the sample against the cylindrical rod (without unloading it). In full constrain methods, (1) the specimens were heated between  $A_s$ <T< $A_f$  (80°C) and above  $A_f$  (100 °C) (2) the specimens were bent against the cylindrical rod to the deformation position (3) then the thermal cycling was performed. The specimens were trained by repeating the mentioned steps for 30 times.



**Figure 1.** Schematic illustration of pre-strain, plastic strain and two-way shape memory effect measurement [10]



**Figure 2.** DSC curves of 1) Ni-49.9 at %Ti, 2) Ni-50.8 at %Ti samples on heating/cooling

# **3. RESULTS AND DISCUSSIONS**

3. 1. The Effect of Chemical Composition on Transformation Temperature DSC results for all hot-rolled and annealed samples are shown in Figure 2. The martensite start and finish temperatures (M<sub>S</sub> and M<sub>f</sub> ) and the austenite start and finish temperatures ( $A_s$  and A<sub>f</sub>) in all cases were determined by DSC. As it is seen, during the heating and cooling cycles in all samples, a single peak is observed corresponding to the B19 $\rightarrow$  B2 and the B2 $\rightarrow$  B19' phase formation. Furthermore, it is clear that the M<sub>s</sub> temperature increases when Ti content increases. As the phase transition temperature of NiTi SMA depends on the chemical composition [9], this behavior can be attributed to the relationship between Ni content and M<sub>s</sub> temperature. In fact Ni is austenite stabilizer. Therefore, the M<sub>s</sub> temperature in Ni-rich alloy in comparison with Ti-rich alloy decreases leads to martensitic transformation.

Figures 3 and 4 illustrate the OM and SEM microstructures of the specimens. According to XRD analysis (see Figure 5), it is clear that  $Ni_3Ti$  and  $Ti_2Ni$  in Ni-rich and Ti-rich alloys have been precipitated, respectively. Figure 3 shows the martensite variants. The XRD results admit the presence of martensite phase in the structure.



**Figure 3.** Optical microstructures of (a) Ni-49.9 at %Ti alloy, (b) Ni-50.8 at %Ti alloy



Figure 4. SEM microstructures of (a) Ni-49.9 at %Ti, (b) Ni-50.8 at %Ti



**Figure 5.** X-ray diffraction analysis of (a) Ni-49.9 at %Ti, (b) Ni-50.8 at %Ti



**Figure 6.** The effect of pre-strain on (a)the two-way and (b) plastic strains in Ni-50.8 at %Ti alloy

**3. 2. The Effect of the Primary Structure on the Optimum Pre-strain and the Plastic Strain** The effect of pre-strain on the two-way and plastic strain in the Ti-rich alloy by performing different training producers is shown in Figure 6 in the martensitic structure. In addition, as the specimen pre-strained 20% failed after 5 training cycles, the curves of two-way strain vs. pre-strain is illustrated up to 5 training cycles. However, in other states the curves are depicted up to 30 cycles.

Generally, it is shown that at the beginning, by increasing pre-strain the two-way strain increases up to a maximum value of 16 %, 12% and 12% pre-strains in martensitic, martensitic+austenitic and austenitic states respectively and then it will decrease. While, the plastic strain increases continuously with increase in pre-strain at all states (see Figure 6-b). Furthermore, it is shown that the plastic strain in austenitic and austenitic+martensitic states is more than martensitic state. When the shape memory alloy is trained in austenitic state, the amount of the stress-induced martensite variants are less than the thermaly-induced martensite variants. Therefore, the plastic deformation happens ready [6, 7], and this leads to degradation of the TWSME in austenitic state rather than in martensitic state. Since in the austenitic+martensitic state, two mechanisms of reorientation and stress-induced martensite variants are acting at the same time, the maximum TWSME is obtained by training at martensitic structure and austenitic one.

3. 3. The Effect of the Primary Structures on the **Optimum Training Cycles** The effect of training cycle on TWSME in Ni-49.9 at %Ti both in the martensitic and austenitic states is shown in Figure 7. As it is seen, increase in the cycle numbers leads to rapid increase in two-way strain up to the steady state manner. Moreover, it can be seen that optimum training cycles are 10-15 and 15-25 in martensitic and austenitic states, respectively. This can be attributed to the yield stress of the primary structure. Because in austenitic and austenitic+martensitic states, the yield stress of the alloy are higher than martensitic state [10]. As a result to induce two-way shape memory, more pre-strain is needed. Therefore, in these training producers, at constant pre-strain, more training cycles are needed.

**3. 4. The Effect of the Primary Structure on the TWSME** Maximum values of the two-way strains in 12% and 16% pre-strains, for all training producers are shown in Figure 8. The results obtained are consistent with the other works [4]. In martensitic training producer, the specimen is cooled below  $M_f$  under no strain, then the pre-strain is applied. In this condition, the thermal martensite is reoriented. In austenitic state,

the specimen is heated up to  $A_f$  and then the strain is applied. In this condition, stress-induced martensite is formed. In martensite+austenite state both mechanisms are acting simultaneously. So, as mentioned in section (3.2), according to the amount of the plastic strain induced in each training producer, two-way strains are obtained.



**Figure 7.** The effect of training cycles on two-way memory strain in Ni-50.8 at %Ti, pre-strain: martensitic 16%, other primary structures:12%



Figure 8. Max two-way strain for training in different primary microstructures, Ni-50.8 at % Ti

**3. 5. The Effect of the Training Temperature on the TWSME and the Plastic Strain** Figure 9 shows the effect of the pre-strain on the TWSME and the plastic strain in the Ni-rich alloy trained in austenitic state, (at two training temperatures constraint is carried out during cooling). It can be seen that increase in training temperature (higher than  $A_f$ , at 100°C and 125°C temperatures), results in decline and increase in two-way and plastic strains, respectively. The results are consistent with other researchers' findings [7]. Above  $A_f$ ,  $e_0/e_{max}$  ( $e_0$ : residual elongation,  $e_{max}$ : maximum residual elongation) mainly consists of residual

elongation due to slip deformation. This parameter increases with increase in temperature. This is attributed to the increase in applied stress for martensitic transformation with temperature in early stages following the Clausius-Clapeyron relationship. Therefore, due to the increasing slip deformation increase in the training temperature leads to decrease in two-way strain.



**Figure 9.** The effect of training temperature on (a) the TWSME and (b) the plastic strain in the Ni-49.9 at %Ti alloy trained in austenitic state

### 4. CONCLUSION

**1.** The optimum pre-strain obtained in martensitic state is 16%, while in other states is 12%.

**2.** The plastic strain increases continuously with increasing pre-strain at different training producers.

**3.** The optimum number of training cycles in martensitic state is 10-15 which is less than, martensitic+austenitic and austenitic states (15-25).

**4.** The order of obtained two-way strains is as below: Martensitic > Martensitic +Austenitic >Austenite 5. In austenite state, by increasing the training temperature, two-way strain decreases.

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Keywords: Shape Memory Alloy Training Primary Structure TiNi تأثیر ساختارهای مارتنزیتی، مارتنزیت استنیت و آستنیتی طی تربیت کردن خمشی روی اثر حافظهداری دوطرفه آلیاژهای Ti %Ni-50.8at و Ni-49.9at مطالعه شد. بعلاوه اثر کرنش اولیه، کرنش پلاستیک، سیکلهای تربیت کردن و دمای تربیت کردن روی اثر حافظهداری دوطرفه بررسی شد. نمونهها در حالت های مارتنزیتی، مارتنزیت استنیت و آستنیتی با تغییرشکل خمشی تربیت شدند. با وجود آنکه تعداد سیکلهای تربیت کردن در حالت مارتنزیتی (۱۰–۱۰ سیکل) کمتر از سایر روشهای تربیت کردن (۲۵–۱۵ سیکل) بود، کرنش اولیه در حالت مارتنزیتی، اثر حافظهداری روشها (۱۲٪) بدست آمد. همچنین مشاهده شد که با افزایش دمای تربیت کردن در حالت آستنیتی، اثر حافظهداری دوطرفه افزایش مییابد.

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چکيده