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Short communication

Optimum temperature for recovery and recrystallization of 52Ni48Ti shape memory alloy

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Abstract

Results of recent studies on the effect of heat treatment after cold rolling on mechanical properties and transformation temperatures of the cast 52Ni48Ti alloys are described. It is seen that stress-induced martensite is formed when both the as-rolled and the low-temperature (⩽400 °C) heat-treated samples are strained. Martensite reorientation due to the stress action occurs in samples heat treated at 500 °C and higher. Optimum recovery occurs below 600 °C resulting in hardness reduction. Recrystallization starts at 600 °C resulting in parent phase yield stress enhancement. Yield stress starts to decrease when grain growth and order–disorder transformation both occur above 700 °C.
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1. Introduction

Mechanical properties and transformation temperatures of NiTi shape memory alloys are influenced by deformation processing and heat treatment of the specimens [1,2]. Determination of the optimum temperatures for heat treatment after cold working is of special interest to scientists and production engineers [3]. Structural defects caused by cold rolling may affect on the phase transformation temperatures, shape memory properties, superelasticity and thermoelastic martensitic phase changes that occur in the NiTi systems [1,3].

The thermoelastic interface of the austenite (parent phase) with the martensite (twin phase) is a completely mobile boundary layer [4,5]. It can thus depress the shape recovery effect. To overcome this problem, the alloy must be heat treated in an appropriate temperature range [6]. The recovery/recrystallization procedure is thus of considerable significance [3]. There is no systematic information, however, available on this subject throughout the literature.

Heat treatment in a temperature range above that of the recrystallization will result in grain growth and order–disorder transformation that will adversely affect on the mechanical properties of the NiTi shape memory alloy (SMA) [7,8]. The objective of this investigation is to determine the most suitable temperature range applicable to recovery and recrystallization of the nickel rich NiTi SMA.

2. Experimental procedure

Cast ingots produced in a VIM (vacuum induction melting) unit were used to produce standard mechanical test-specimens. Sponge titanium particles together with cathodic nickel plates were melted to produce NiTi ingots. Details of the production procedure were described elsewhere [9]. Particle induced X-ray emission (PIXE) [10] analyses were used to determine the analysis of the as-cast samples. They contained 42.70 wt% of titanium and 57.06 wt% of nickel.

The ingots were rolled at 1000 °C from 10 cm × 1.5 cm × 1.5 cm to 11 cm × 1.1 cm × 1.8 cm. Rolled samples were machined to their eventual dimensions. They were then surrounded with pure iron powders to protect them from oxidation and were heat-treated for 1 h at different temperatures from 100 to 800 °C. They were quenched then into water and their tensile strength and hardness were measured.

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3. Results and discussion

Fig. 1 illustrates the austenite start and finish temperatures determined by DSC heating test. Stress–strain curves of the heat-treated samples are illustrated in Fig. 2. Mechanical response of the shape memory alloy depends on the prior thermal processes applied to the specimens. With the 10% reduction in area applied to the ingot samples of this research, the stored cold working corresponds to a strain-hardening exponent of 0.23. This value is evaluated based on the method described by Rozner and Buehler [4].

Quenching a specimen from above Af (austenite finish transformation temperature in heating) to a temperature between Ms (martensite start transformation temperature in cooling) and As (austenite start transformation temperature in heating) results in formation of an austenitic microstructure corresponding to pattern I of Fig. 3. The specimens quenched to a lower than Mf temperature and then heated to a temperature between Ms and As show, however, a martensitic phase dominance corresponding to pattern II of Fig. 3 [5]. This curve corresponds to martensite reorientation due to the stress action.

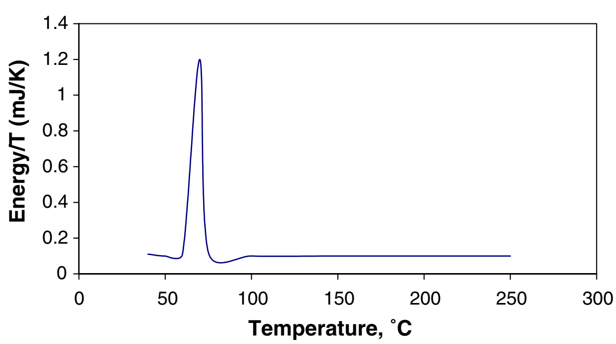

Fig. 1. DSC curve of the rolled specimen.

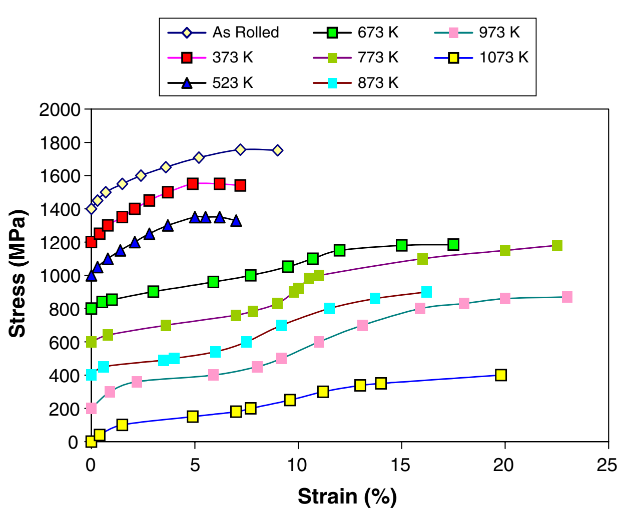

Fig. 2. Stress–strain curves of the rolled specimens heat-treated for an hour at different temperatures. A 200 MPa shifting has separated the curves.

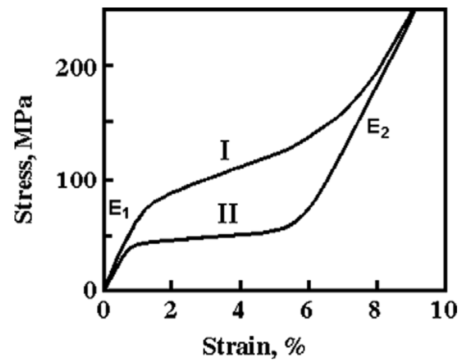

Fig. 3. Two stress–strain patterns for a specimen having a temperature between Ms and As: (I) quenched from above Af and (II) heated from lower than Mf.

Effect of annealing on resistivity vs. temperature curves of the samples is illustrated in Fig. 4. Cooling curves indicate the appearance of both rhombohedral and martensite phases during cooling of the samples. These curves indicate a martensite start transformation temperature of around 310 K and a martensite finish temperature of about 280 K. Variations with annealing of transformation temperatures are plotted against annealing temperature in Fig. 5. These results are similar to those determined for nickel rich NiTi alloys during previous investigations [2,11].

Stress–strain curves of the as-received as well as 100–300 °C annealed samples observed in Fig. 2 look similar to pattern I of Fig. 3. They correspond with stress-induced martensite (SIM) transformation indicating prevalence of remained austenite within the samples. Austenite phase dominance after cooling from the rolling and/or annealing temperature to that of the ambient brings about this behavior. Small differences between plastic flow, start-strains of the as-rolled and heat-treated specimens shown in Fig. 2 seems due to the stress hardening effects. Recovery, recrys-

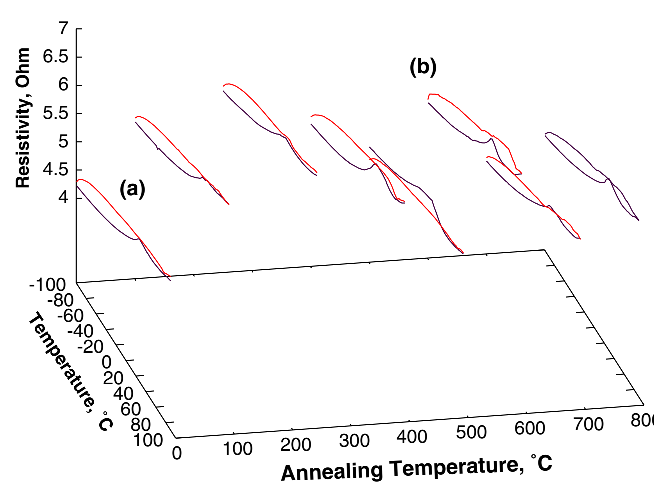

Fig. 4. Effect of 1 h annealing on resistivity–temperature curves of the samples used in this investigation: (a) as-rolled and (b) annealed at temperatures specified on the diagram.

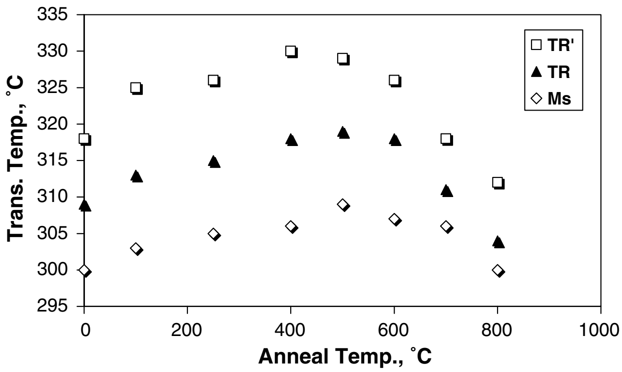

Fig. 5. Variation of martensite start (Ms), R-phase start (TR′) and R-phase finish (TR) temperatures with the annealing temperature. The annealing time has been 1 h.

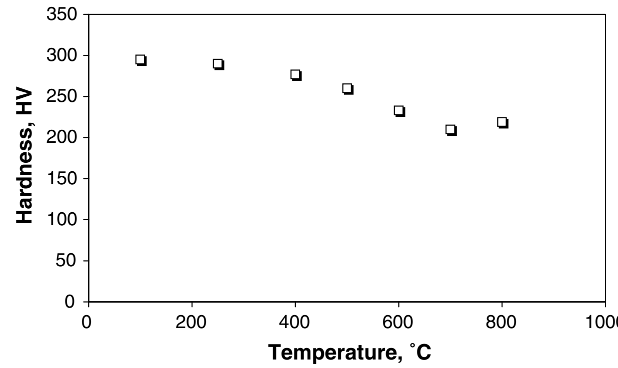

Fig. 6. Effect of 1-h heat treatment on hardness of the hot rolled NiTi samples.

tallization and order–disorder transformation are different competing mechanisms that affect mechanical behavior and transformation temperatures of the specimens.

Fig. 5 shows an increasing trend in Ms, TR′ and TR with the annealing temperature up to around 500 °C and a subsequent decrease thereafter. This indicates an increasing volume of martensite plus R-phase to austenite ratio within the samples up to 500 °C. A higher martensite plus R-phase to austenite ratio means a closer to pattern II (see Fig. 3) stress–strain curve. This effect is clearly observable by comparing the results shown in Figs. 2 and 3.

3.1. Recovery and recrystallization

Ya et al. [8] have shown that recovery and recrystallization of Ni–Ti–Pd alloys are related to the parent phase transformation temperatures. This is not the case, however, for NiTi alloys because their transformation temperatures are well below those of recovery and recrystallization [7,8].

Recrystallization results in hardness change of the samples [7]. One can determine, therefore, the temperature range for recovery and recrystallization by hardness measurement. Effect of heat treatment on the overall hardness of the specimens is given in Fig. 6. It is seen that higher annealing temperature causes a lower hardness in the sample. Hardness decrease is due to different processes:

(a) R-phase formation,
(b) Martensite formation,
(c) Recovery of the microstructure,
(d) Order–disorder transformation.

Order–disorder transformation normally occurs at temperatures higher than 800 °C [12]. DSC curve (Fig. 1) shows an austenite formation temperature of around 70 °C, it is therefore concluded that the decreasing of the hardness is due to martensite formation, R-phase formation and recovery of the samples. It is said that the hardness change is not because of the martensite transformation [8,11].

3.1.1. Parent phase yield stress

Parent phase yield stress (PPYS) is the stress at the end of the first linear E_1 part in the σ–ε curves of Fig. 3. As it can be seen in Fig. 7, below 600 °C PPYS decreases with the annealing temperature. This is similar to what Liu and McCormic [6] have reported. It is usually attributed to recovery of the rolled specimen that seems proceeding below 600 °C. Above 600 °C, PPYS increases with heat treatment of the samples. Completion of recovery at 600 °C may be the reason for this effect.

Recovery can cause formation of crystal defects like dislocations below 600 °C. This phenomenon results in increasing of the density of the defects. With increasing of the annealing temperature, the ability of the parent/martensite interface for moving enhances. This results in the parent phase yield stress reduction. Above 600 °C, PPYS increases due to recrystallization of the austenitic phase. Finally, yield stress decreases with grain growth and order–disorder transformation, both occurring above 700 °C [11].

3.1.2. Ratio of elastic modules

According to Ford and White [13], the ratio of elastic modules of the linear region of curve I (E_1) to the elastic modules of the linear region of curve II (E_2) in tension curve of Fig. 3 varies with post rolling heat treatment temperatures. As it is shown in Fig. 8, E_1/E_2 ratio first decreases with increasing of the temperature to 550 °C, increases up to 800 °C and decreases again. The reason for the first E_1/E_2 decrease is structural recovery resulting

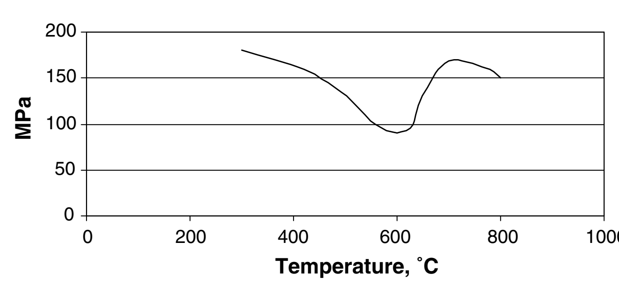

Fig. 7. Effect of heat treatment on parent phase yield stress.

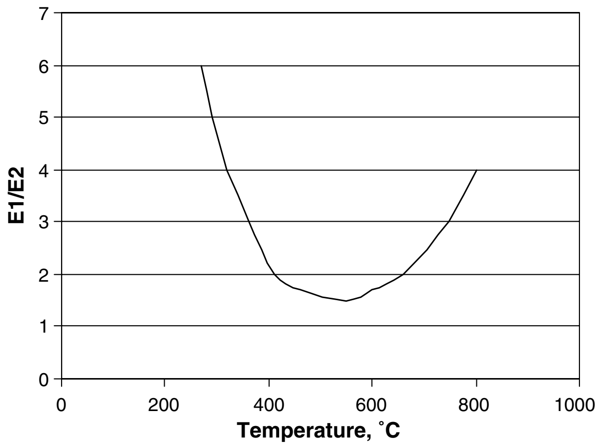

Fig. 8. Variation of $\frac{E_1}{E_2}$ ratio with the annealing temperature.

in disappearance of the dislocations. This does not change the parent phase/martensite interface. Above 600 °C, recrystallization results in decreasing of the grain sizes. This will reduce mobility of the parent phase/martensite interface and increases the E_1/E_2 ratio up to 800 °C.

Based on the reported melting range (1240–1310 °C) [14,15], the recrystallization start temperature can be estimated from this investigation to be 0.55–0.57 times T_m. This temperature is reported to be between 0.45 and 0.5 T_m by Belyaev [16] and 450–600 °C (i.e. 0.48–0.55 times T_m) by Ya and his co-workers [8].

4. Conclusions

1. Recovery and recrystallization temperatures of the as-cast 52Ni48Ti shape memory alloys after hot and cold rolling were rigorously studied. A recrystallization temperature of about 600 °C was as a result obtained. This was the optimum heat treatment temperature after cold rolling inducing proper combination of mechanical and shape memory properties in the 52Ni48Ti samples.
2. The small difference between As and Af and the hardness decreasing of the samples with the annealing temperature was due to the recovery of the alloy below 600 °C (0.55–0.57 T_m).
3. Below 600 °C, E_1/E_2 decreased. This was another reason for occurrence of recovery below recrystallization temperature.
4. Above 600 °C, the parent phase yield stress increased due to fine grains produced during recrystallization.
5. E_1/E_2 increased above 600 °C showing another recrystallization effect.
6. The optimum heat treatment temperature after cold rolling was concluded to be around 600 °C.

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References

[1] Khelfaoui F, Thollet G, Guenin G. Microstructural evolution kinetics after plastic deformation of equiatomic Ti–Ni alloy during isothermal annealings. Mater Sci Eng A 2002;338(1–2):305–12.
[2] Sadrnezhaad SK, Mashhadi F, Sharghi R. Heat treatment of Ni–Ti alloy for improvement of shape memory effect. Mater Manuf Process 1997;12(1):107–15.
[3] Khelfaoui F, Guéni G. Influence of the recovery and recrystallization processes on the martensitic transformation of cold worked equiatomic Ti–Ni alloy. Mater Sci Eng A 2003;355(1–2):292–8.
[4] Rozner AG, Buehler WJ. Effect of cold work on room-temperature tensile properties of TiNi intermetallic compound. Trans ASM 1966;59:350–2.
[5] Melton KM, Mercier O. The mechanical properties of NiTi-based shape memory alloys. Acta Metall 1981;29:393–8.
[6] Liu Y, McCormic PG. Influence of heat treatment on mechanical behavior of NiTi alloy. ISIJ Int 1989;29:417–22.
[7] Ya X, Otsuka K. CJ SMA97. In: Youyi C, Otsuka K, editors. Proceeding of bilateral symposium on shape memory alloys; 1997.
[8] Ya X, Shimizu S, Suzuki Y, Otsuka K, Ueki T, Mitose K. Recovery and recrystallization processes in Ti–Pd–Ni high-temperature shape memory alloys. Acta Mater 1997;45(4):1503–11.
[9] Badakhshan Raz S, Sadrnezhaad SK. Effects of VIM frequency on chemical composition, homogeneity and microstructure of NiTi shape memory alloy. Mater Sci Technol 2004;20(5):593–8.
[10] Sadrnezhaad SK, Badakhshan Raz S. Interaction between refractory crucible materials and the melted NiTi shape memory alloy. Metall Mater Trans B 2005;36B(3):395–403.
[11] Zarandi FMH, Sadrnezhaad K. Thermomechanical study in combustion synthesized Ti–Ni shape memory alloy. Mater Manuf Process 1997;12(6):1093–105.
[12] Mashadi F. Studying shape memory phenomenon for medical applications, MSc Thesis, Department of Materials Science and Engineering, Sharif University of Technology; 1995.
[13] Ford DS, White SR. Thermomechanical behavior of 55Ni45Ti nitinol. Acta Mater 1996;44(6):2295–307.
[14] Besselink PA. In: Torra V, editor. The science and technology of shape memory alloys. Barcelona; 1987.
[15] Van Humbeek J, Delay L. In: Hornbogen E, Jost N, editors. The martensitic transformation in science and technology. DGM; 1989.
[16] Belyaev S. Available from: <html://www.Mechanic.Tu-berilin.de>.