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Heat Treatment of Ni-Ti Alloy for Improvement of Shape Memory Effect

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Abstract

Nickel-Titanium alloys with stoichiometric single phase and non-stoichiometric dual phase structures of NiTi and $NiTi+Ni_3Ti$ are produced through high speed induction melting and combustion synthesis of pure Ti/Ni elements. Both alloys are homogenized at 1273 K for two hours, rolled into thin strips of 0.3 mm thickness, solution treated at 1273 K for two hours under vacuum and finally quenched in water. Effect of ageing on austenite/martensite and intermediate phase transformation temperatures are investigated. Results show that transformation temperatures and reversible shape memory properties comparable with those required for bioengineering applications such as manufacturing of artificial hand prostheses can be obtained through careful control of the chemical composition and the heating processes.

1.0 Introduction

Shape Memory Effect (SME) is one of the most interesting behaviors associated with the martensitic transformation in Ni-Ti alloys. Ni-Ti alloys represent advantageous capabilities of memorizing both initial and final geometric shapes (1). Numerous technological applications of great attraction to biomedical engineers have been introduced during past few years (2). Design and manufacturing of miniature type medical device, orthopedic surgical implant and artificial prosthesis are a few of the many possible examples.

Numerous investigations are underway throughout the world to find the important parameters that may influence the SME behavior (3-5). One of the most important properties that must be controlled during production, is the temperature at which the alloy starts to remember its previous shape (6,7). There is not much written, however, on the effects of the method of production, the chemical composition and the ageing time and temperature on the SMA transformation temperatures. Typical results obtained from our extensive studies on these effects are reported in this paper.

2.0 Experimental Procedure

2.1 Method of Production

Ni-Ti alloy can be produced by different routes. One way is to simultaneously melt the highly pure elements in a fast-melting unit such as High Frequency Induction System (HFI) (3). Undesirable contaminations from refractory materials are, however, often inevitable in this method. "Self-Propagating High Temperature Synthesis (SHS)" also called "Combustion Synthesis (CS)" is also used as an alternative method to produce a highly desirable ultra-clean alloy (8-10). This process consists of a self-sustaining ignition reaction that synthesizes the batch of uniformly mixed powders. Both methods are tried in this research. All specimens selected for this study show the shape memory effect.

2.2 HFI Melting

Pure metal pieces cut from plates containing 99.7% Ni and ingots containing 99.4% Ti are charged into a graphite crucible and melted in an experimental HFI unit under vacuum. The alloy is cast into an iron mold, homogenized for 18 kS at 1273 K and subsequently analyzed. The resulting chemical composition is 50.2 at.% Ni balanced with Ti.

The specimens are rolled at 1173 K into thin strips of 0.35 mm thickness with intermediate annealing for 120 S at 1173 K. Then they are solution treated for 3.6 KS at 1273 K under vacuum both to release the residual stress and to produce a NiTi single phase. The specimens are then quenched in water.

Effect of ageing on the transformation temperatures is studied by heating specimens at 573, 673, 723, 773, 823 and 873 K for 3.6 kS and then quenching them in water. The electrical resistivities of the specimens are precisely measured against temperature. Effects of the ageing time, the ageing temperature and the chemical composition of the specimens on the electrical resistivities are determined.

2.3 Combustion Synthesis

Ni-Ti pellets containing 50.03, 50.23, 50.33 and 50.68 at.% Ni are made by weighing, mixing and pressing high purity powders of both elements into a metallic die. The powders have irregular shapes and their size ranges from 60 to 130 μm for Ti and under 15 μm for Ni. Each pellet is made of 10 gms of carefully mixed powders compacted to reach 65-66% of the alloy's theoretical density.

A separate graphite crucible is used to synthesize each pellet. The crucible is heated under flowing argon in a horizontal tube furnace being held at 1573 K. The result is production of a homogeneous cast alloy which is hot-rolled at 1073 K into strips of 1 mm thickness and then cold-rolled at room temperature into strips of 0.5 mm thickness without intermediate

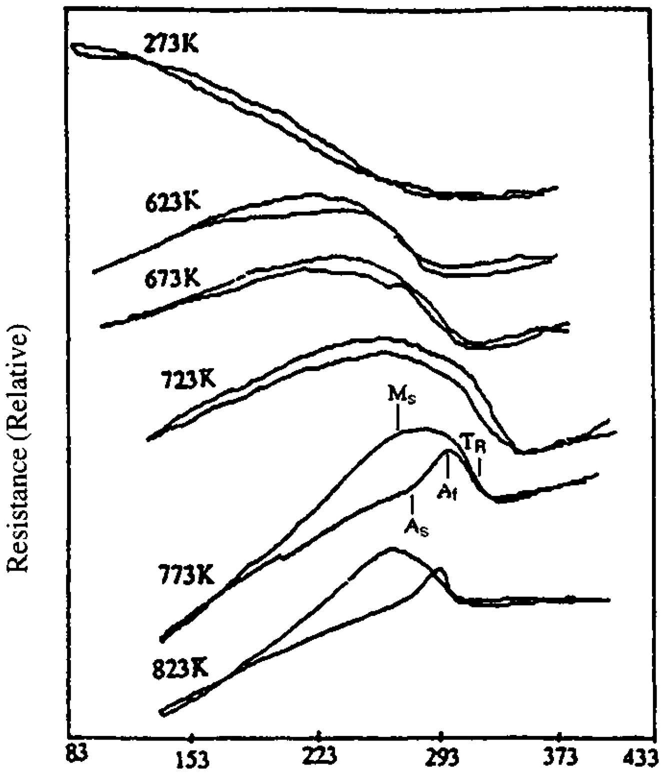


Figure 1: Effect of ageing temperature on the electrical resistance of 50.23% Ni alloy. All specimens except the one plotted at the top of the diagram are aged for 3.6 kS. The one at the top is not aged. All samples are produced by CS method.

annealing. The strips are solution treated in a high vacuum furnace at 1273 K for 3.6 kS and then quenched in water. They are eventually aged under atmospheric conditions at various temperatures and for different periods of time.

The microstructures of the specimens are examined using an optical microscope. The samples are etched in a solution containing H_2O, HNO_3 and HF with volumetric ratios of 5, 4 and 1, respectively. The transformation temperatures are measured by electrical resistance determination. Cu-K_α radiation is used for x-ray diffractometery of the samples.

3.0 Results

3.1 Transformation Temperatures

Variation of the electrical resistivity versus temperature for 50.23 at.% Ni alloy aged at 623, 673, 723, 773 and 823 K for 3.6 kS is demonstrated in Figure 1. No ageing is done for the curve shown at the top of the figure. This curve is seen to be concave upwards, indicating no austenite

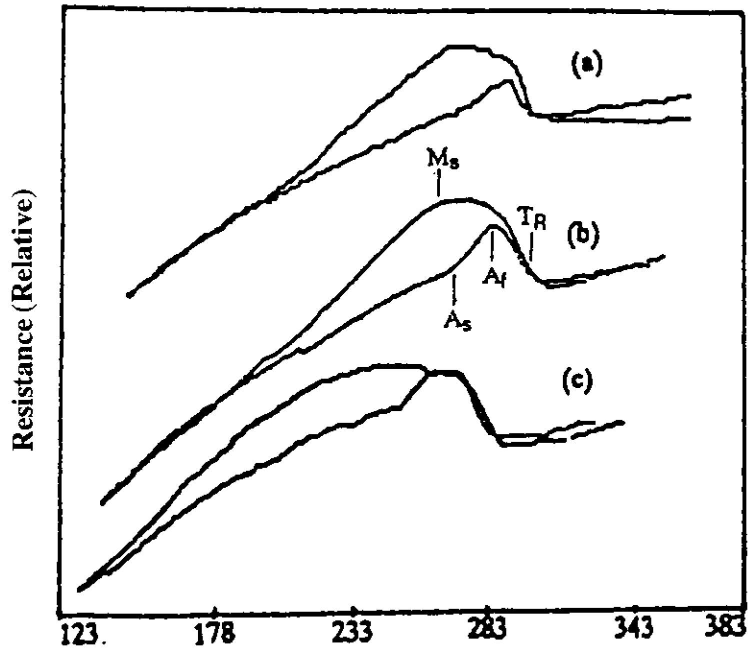


Figure 2: Effects of the chemical composition and the ageing time on the electrical resistence of (a) 50.33 at.% Ni alloy aged for 3.6 kS, (b) 50.23 at.% Ni alloy for 3.6 kS, and (c) 50.03 at.% Ni alloy aged for 7.2 kS. All samples are produced by CS method.

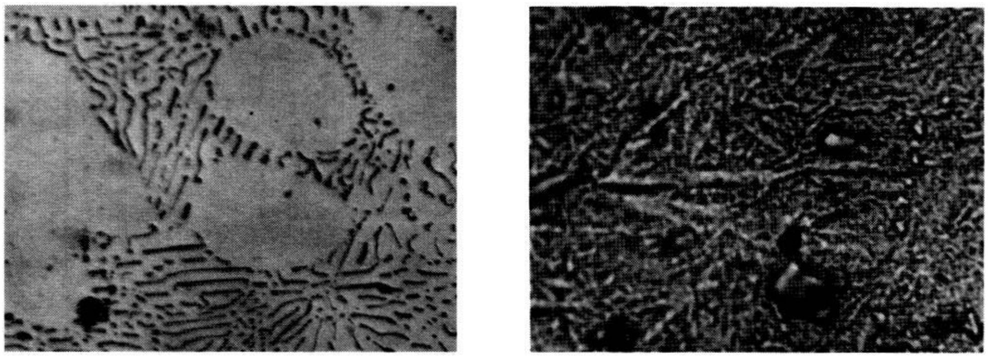
to martensite transformation to occur above 77 K, but indicating that austenite to incommensurate phase B2↔IC, transforms above 77 K. The samples with greater ageing temperatures show that the transformation into martensite and R-phase can occur at the experimental range of temperatures indicated in the figure.

Effects of the chemical composition and the ageing time on the transformation temperatures MS and TR are shown in Figure 2. Curve (a) and curve (b) indicate that MS and TR both decrease by decreasing Ni content of the specimens while curve (c) shows a further decrease in MS and TR in spite of the double increase observed in the aging time.

3.2 Metallography and X-ray Diffractometery

Figure 3 shows typical morphologies of CS specimens containing 50.60 at.% Ni (a) before rolling and (b) after rolling. Photo micrograph (a) shows a lamellar eutectic structure containing NiTi (light) and Ni_3Ti (dark) flakes embedded in a single NiTi phase. The CS specimens are readily hot-rolled into very thin plates. As can be seen from part (b) of the figure, the process destroys the eutectic structure and martensite is produced. The production of this phase stimulates the appearance of the shape memory effect.

In Figure 4, a comparison is made of the x-ray diffraction patterns of 50.33 at.%



(a) (b)

Figure 3: Photo micrographs of 50.60 at.% Ni specimens produced by CS process (a) as cast and (b) after hot-rolling.

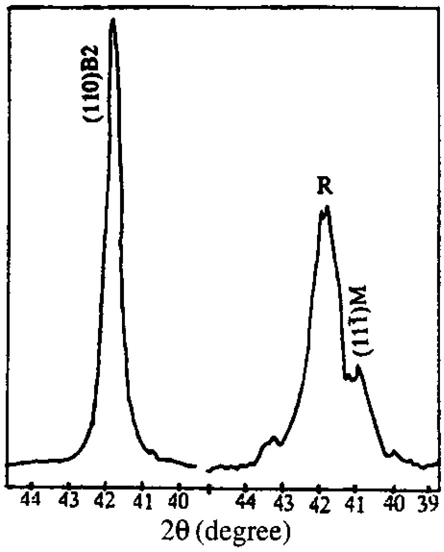


Figure 4: X-ray diffraction patterns of the 50.33 at.% Ni alloy, (a) solution treated at 1273 K for 3.6 kS and quenched in water and (b) aged at 773 K for 25.2 kS.

Ni samples (a) solution treated and (b) aged for 25.2 kS at 773K. It can be inferred from the figure that the only existing phase in the solution treated state is the parent B2 phase that transforms into martensite and R-phase as a result of the ageing which is accompanied with reduction of the Ni content of the matrix.

4.0 Discussion

Effects of ageing on the MS transformation temperature of different samples with different compositions and production methods are demonstrated in Figures 5 and 6. Figure 5 shows that at a constant aging temperature, a decrease in

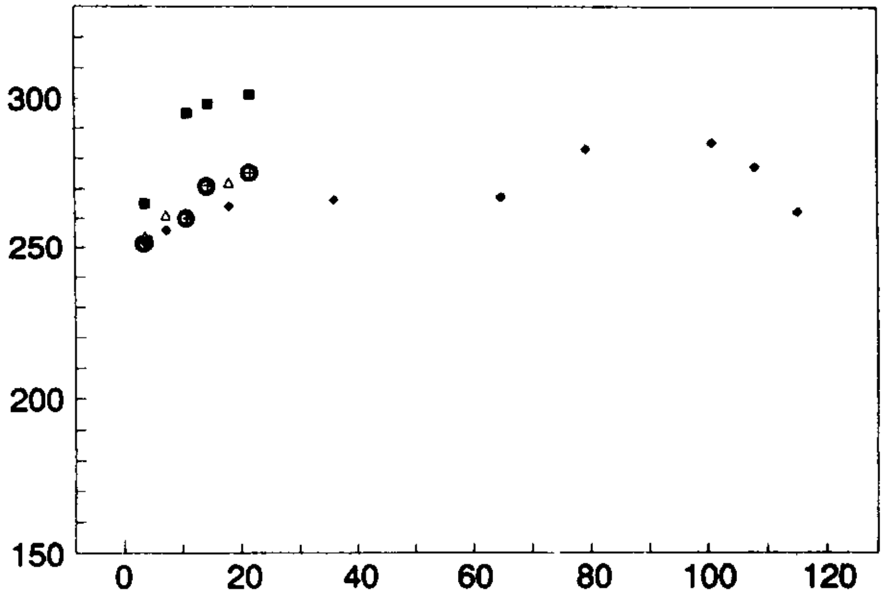


Figure 5: Effect of aging time on M_s temperature, ■ CS, 50.33,⊕CS, 50.23, ♦HFI, 50.20, Δ HFI, 50.10. All specimens are solution treated and aged at 723 K.

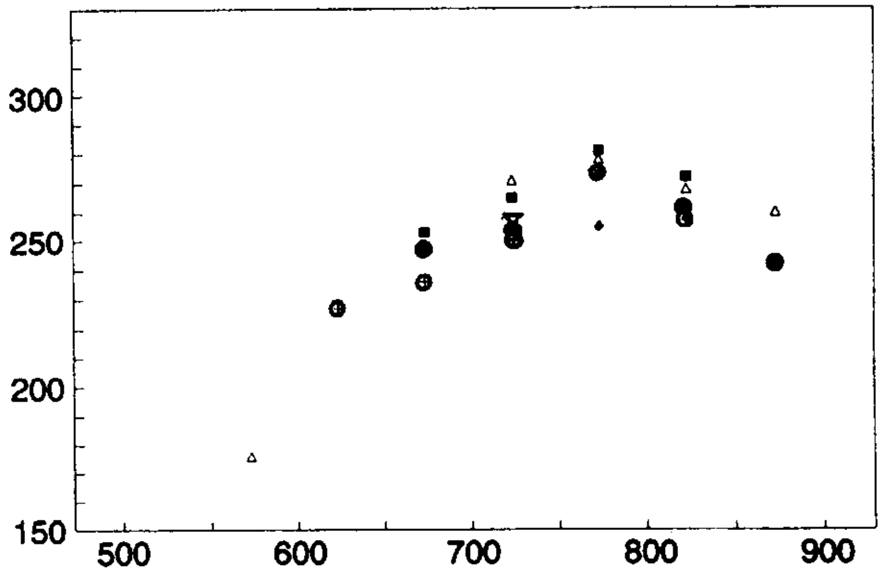


Figure 6: Effect of aging temperature on M_s: ■ CS, 50.33, ⊖ CS, 50.03, ♦ CS, 50.03 Δ HFI, 50.20, • HFI, 50.20, ∇ HFI, 50.10. All specimens are solution treated and aged for 3.6 kS. The fourth specimen is cold worked before aging.

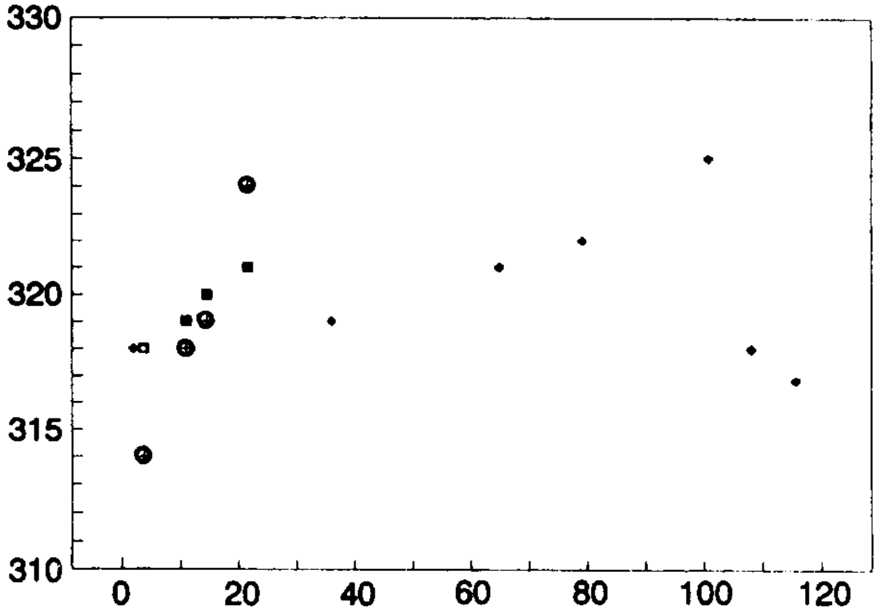


Figure 7: Effect of aging time on T_R temperature.■ CS, 50.33, ⊖CS, 50.23, ♦HFI, 50.20. All specimens are aged at 723 K.

the Ni content will reduce M_s temperature of the samples made through CS route. Similar results are obtained for reverse martensitic transformation.

This is not the case, however, for samples made through other routes as reported by T. Honma et al. (11), M. Nishida and T. Honma (12) and G. Jinfang et al. (13). The contradiction may be due to the absorption of impurities associated with alternative production processes. The HIF curves illustrated in Figures 5 and 6 indicate, for example, results different with CS curves.

According to Nishida and Honma (12), the R-phase transformation can only occur when the Ni content is higher than 50.30 at.%. It is, however, important to notice that this transformation occurs even in 50.03 at.% Ni alloy illustrated in Figure 2(c). This means that in specimens produced by combustion synthesis, MS can be so much suppressed that the stress due to the coherency of the precipitation can result in formation of the R-phase before that of martensite. T_R transformation temperature as shown in Figures 7 and 8, is not so sensitive to composition as M_s.

5.0 Summary

NiTi shape memory alloys are produced by high frequency induction melting and combustion synthesis. The real-time electrical resistivity versus temperature measurements are done as a means to determine the transformation temperatures of the alloys. Transformation temperatures comparable with those

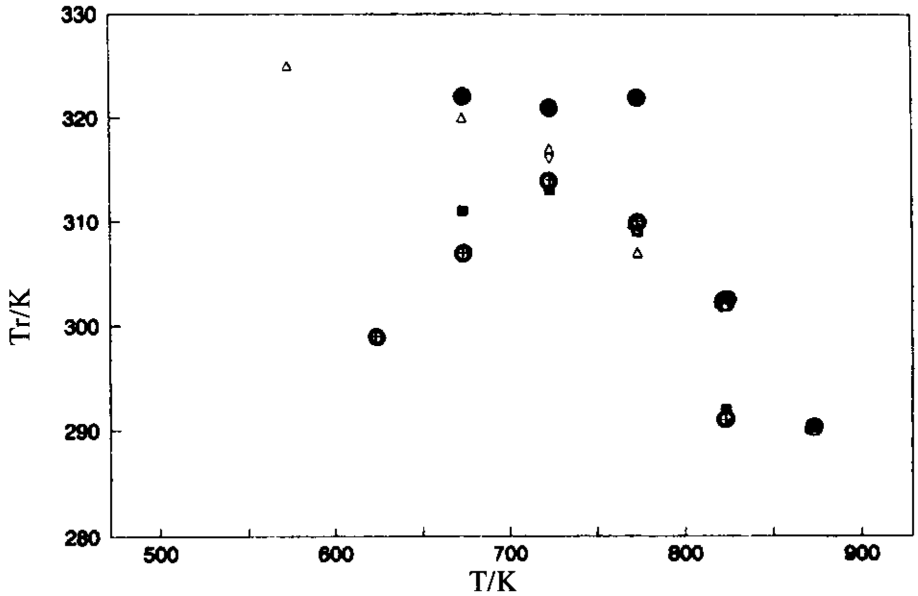


Figure 8: Effect of aging temperature on T_R. ■ CS, 50.33, ⊕ CS, 50.23, ♦ CS, 50.03 Δ HFI, 50.20, • HFI, 50.20, ∇ HFI, 50.10. All specimens are solution treated and aged for 3.6 kS. The fourth specimen is cold worked before aging.

required for bioengineering applications such as manufacturing of artificial hand prostheses are obtained. Effects of the chemical composition, method of production and thermal treatment on the austenitic/martensitic and intermediate phase transformation temperatures are investigated.

6.0 References

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