

EFFECTS OF HEAT TREATMENT ON TENSILE PROPERTIES AND SHAPE MEMORY EFFECTS OF Ni-Ti-Nb ALLOY^①

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ABSTRACT The effects of heat treatment on tensile properties and shape memory effects of Ni-Ti-Nb alloy have been studied by tensile test at different temperatures and electrical resistance measurement. It has been shown that below 900 °C with the increase of solid solution temperature, cooling rate and aging temperature, yield stress decreases whereas elongation increases; yet above 900 °C, yield stress shows no substantial change whereas elongation decreases abruptly. Once specimens deformed at ($M_s + 30$ °C) to 16%, transformation hysteresis width around 150 °C can be achieved and shape recovery ratio still remains 70% ~ 80% no matter which kind of heat treatment is taken.

Key words Ni-Ti-Nb alloy heat treatment tensile properties shape memory effect

1 INTRODUCTION

Ni-Ti-Nb shape memory alloys have attracted considerable attention in recent years, since they exhibit a wide transformation hysteresis which is useful in the applications for coupling and sealing^[1,2]. Ni₄₇Ti₄₄Nb₉ alloy is the typical one among the alloys. Zhao *et al*^[3,4] reported in detail its transformation behavior and microstructure. Zhang *et al*^[5,6] studied the effects of deformation on the transformation hysteresis and shape memory effect and found that there exist a characteristic deformation temperature ($M_s + 30$ °C) and a critical strain range (14% ~ 20%) at which deformation can effectively increase the transformation hysteresis with the strain recovery ratio remaining high enough at the same time.

The change of the microstructure and the transformation temperature of Ni-Ti-Nb alloys as a function of composition were investigated by Piao *et al*^[7,8]. Recently the work^[9] by the pre-

sent authors had shown that heat treatment regulation can affect the martensitic transformation behavior of the Ni-Ti-Nb alloy. But there is no report about the influence of heat-treatment on the mechanical behavior. The purpose of the present work is to investigate the influence of heat treatment on tensile properties, transformation hysteresis and shape memory effect.

2 EXPERIMENTAL PROCEDURE

2.1 Material

The composition of the material was 47.3% Ni, 43.8% Ti and 8.9% Nb. The ingots were swagged and rolled at about 850 °C to a form of 6 mm sheet. Specimens (0.5 mm × 2 mm × 50 mm) were cut carefully from the sheet and then heat-treated in quartz capsules evacuated to 0.1 Pa. In order to investigate the effect of solid solution temperature, the specimens were heat-treated at various temperatures in a range from 800 °C ~ 1 050 °C for 1 h and then rapidly quenched into water (hereafter the heat-treat-

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ment is abbreviated as 900 °C 1 h W Q, for example). In order to investigate the aging effect, some specimens were aged for 2 h below 800 °C (from 800 °C to 300 °C) followed by quenching into water, after the heat treatment of 900 °C, 1 h W Q. Several cooling conditions used in the experiment were ice brine quenching (I B Q), water quenching (W Q), air cooling (A C) and furnace cooling (F C).

2.2 Testing methods

The transformation temperatures were determined by electrical resistance measurement. The tensile tests were carried out on an Instron 1186 testing machine at a strain rate of $4.1 \times 10^{-4} \text{ s}^{-1}$ with temperature range between -90 °C and room temperature. Tensile specimens with 30 mm \times 2 mm \times 0.5 mm gauge portions were fixed by clamps that go through the constant temperature cabinet and were enveloped in a cooling atmosphere achieved by ejecting liquid nitrogen into the cabinet with high pressure nitrogen. Temperature in the cabinet was equalized by stirring air in the cabinet with an electric fan. The accuracy of the test temperature was controlled within ± 1 °C. In order to determine the shape memory effect, two parallel scratches were drawn near the ends of the gauge portion. The strains were determined directly by the length change between the scratches at room temperature. Supposing L_0 as the length before loading, L as the length at room temperature after loading, L_T as the length after loading and unloading then heating well above A_f temperature, the shape recovery ratio can be calculated from the following equation:

$$R = (L - L_T) / (L - L_0) \times 100\%$$

3 RESULTS AND DISCUSSION

3.1 Effect of heat treatment on mechanical properties

Fig. 1 shows the effect of solid solution temperatures on the yield stress $\sigma_{0.2}$ and the elongation δ . With the increase of solid solution temperatures, the yield stress goes down rapidly

then does not change almost after surpassing 950 °C. The elongation shows no significant change when annealing specimens among 800 ~ 900 °C and then decreases rapidly when annealing specimens above 900 °C.

Fig. 2 and Fig. 3 show the effect of cooling conditions on the yield stress and the elongation, respectively. It can be seen that the yield stress decreases whereas the elongation increases with the increment of the cooling rate from F C to A C then both of them tend towards stability under more rapid cooling rate. Another phe-

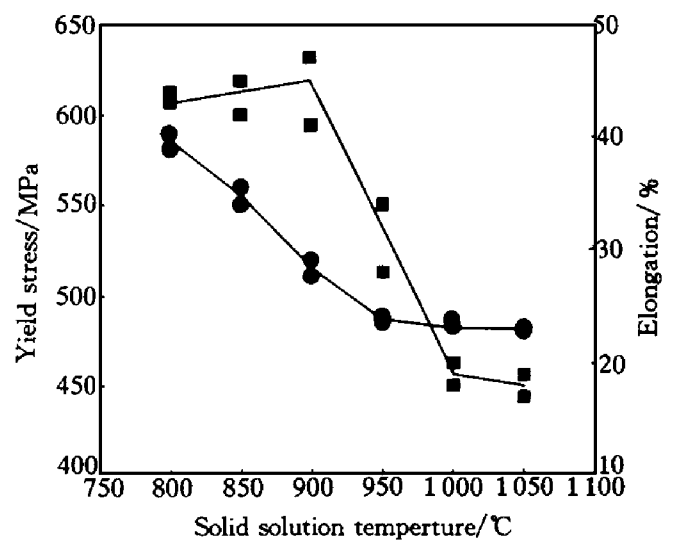


Fig. 1 Effect of solid solution temperature on yield stress $\sigma_{0.2}$ and elongation

δ (deformation temperature = 20 °C)
1 — $\sigma_{0.2}$; 2 — δ

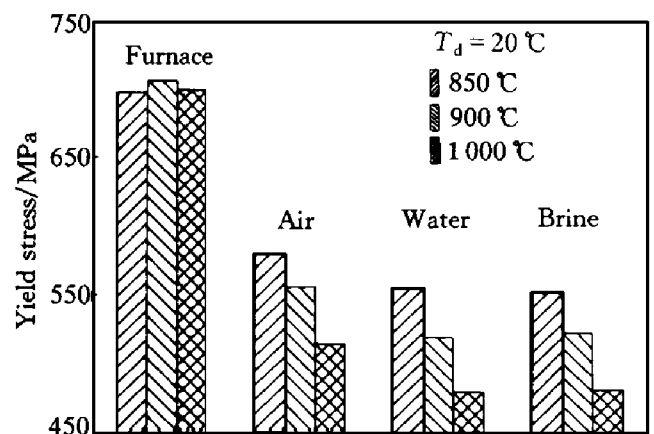


Fig. 2 Effect of cooling conditions on yield stress $\sigma_{0.2}$

(T_d — deformation temperature)

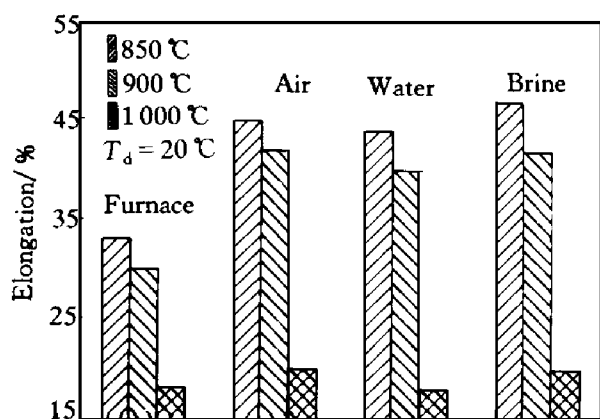


Fig. 3 Effect of cooling conditions on elongation δ

nomenon can be observed is that all specimens were solid solution treated at 1000 °C show lower elongation comparing with specimens annealed below 900 °C irrespective of cooling rate. This indicates that solid solution treating the alloy above 900 °C would deteriorate its ductility.

The effect of aging temperatures on the yield stress and the elongation are shown in Fig. 4. It can be found that the yield stress goes down whereas the elongation goes up with the elevation of the aging temperature.

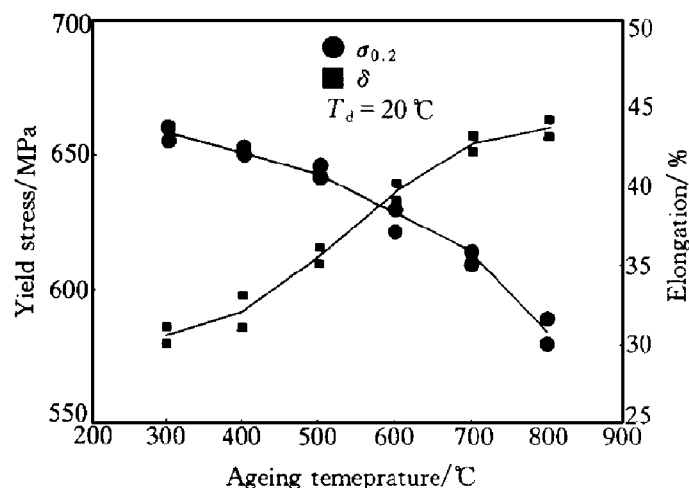


Fig. 4 Effect of aging temperature on yield stress $\sigma_{0.2}$ and elongation δ

From the results above, it can be found that the yield stress reduces near linearly in the following sequence: specimen F C \rightarrow specimen aged at low temperature \rightarrow specimen aged at high tem-

perature \rightarrow specimen A C \rightarrow specimen W Q (solid solution treatment at low temperature) \rightarrow specimen W Q (solid solution treatment at high temperature) \rightarrow specimen I B Q. This is consistent with the variation of the M_s temperature to heat treatment condition^[9]. TEM observations show no new kinds of precipitated phase were found in the B2 parent phase after heat treatment, but EPMA shows the content of nickel (or Ni/Ti ratio) in the B2 matrix changed. The main reason for the change of transformation temperature lies in the change of the matrix composition (or Ni/Ti ratio) with the variation of heat treatment procedure and the precipitation of the β -Nb from the NiTi matrix was considered to be the reason of the change in the composition of the matrix phase. Yet the quantity of new β -Nb precipitate is too little. No obvious difference exists among the microstructure of specimens undergone different heat treatment at room temperature^[9]. So the change of the microstructure with the heat-treatment condition can not be the main reason for the change of $\sigma_{0.2}$ and δ .

As well-known, martensite can be formed above the M_s temperature with the application of external stress. The critical stress to form martensite increases linearly with an increase in temperature from M_s to M_s^0 ^[10]. All the specimen are under the condition of the parent phase at room temperature^[9]. The M_s^0 temperature which represents the highest temperature for the formation of SIM is about 60 °C for the alloy in this experiment^[5]. So tensile at room temperature will induce the forming of martensite, the bigger the difference between the ambient temperature and the M_s temperature, the more the stress is needed to induce SIM, correspondingly the higher the yield stress is. So the change of $\sigma_{0.2}$ is just a reflection of the change of the M_s temperature.

3.2 Effect of heat treatment on transformation hysteresis and shape memory effect

Some specimen are deformed at its test temperature ($M_s + 30$ °C) to a total strain of 16%. Figs. 5(a) ~ (d) show electrical resistance versus temperature curves of several typical specimens

after deformation. In comparison to specimens undeformed (see Fig. 5(e)), deformed specimens show a big increase of its A_s temperature with no change of its M_s temperature. It can also be found that with the variation of the M_s temperature from -120°C to -60°C , as shown in Fig. 5(a) ~ (d), the shape of R - T curves after deformation shows no substantial change.

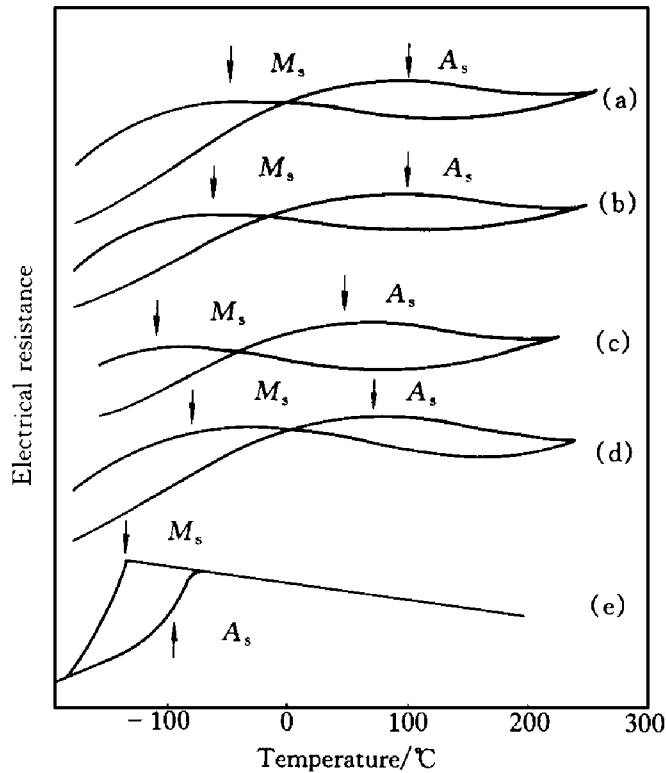


Fig. 5 Electrical resistance versus temperature curves of specimens after deformed at ($M_s + 30^\circ\text{C}$) to 16% total strain

- (a) -900°C , 1 h W Q;
- (b) -900°C , 1 h A C;
- (c) -900°C , 1 h F C;
- (d) -900°C , 1 h W Q $\rightarrow 500^\circ\text{C}$, 2 h W Q;
- (e) -900°C , 1 h F C

Fig. 6, Fig. 7 and Fig. 8 respectively show the effect of solid solution temperatures, cooling conditions, aging temperatures on the transformation temperature hysteresis and the shape recovery ratio. Obviously, once specimens deformed at ($M_s + 30^\circ\text{C}$) to a total strain of 16%, transformation hysteresis width around 150°C can be achieved and the shape recovery ratio still

remains 70% ~ 80%, no matter which kind of heat treatment regulations is taken previously.

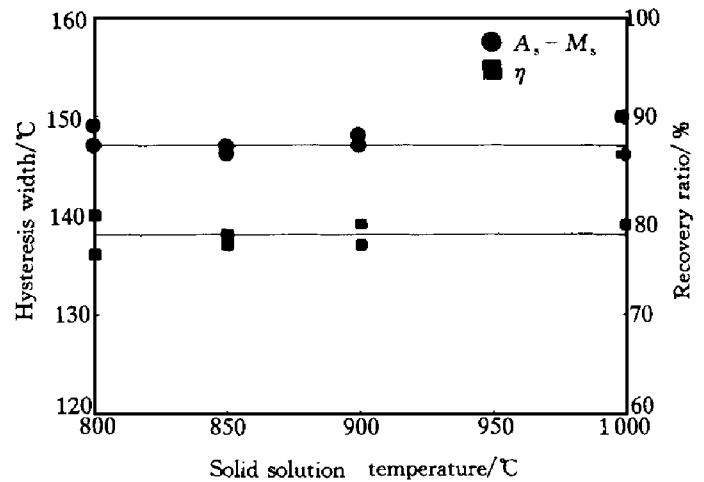


Fig. 6 Effect of solid solution temperatures on transformation hysteresis ($A_s - M_s$) and shape recovery ratio (η)

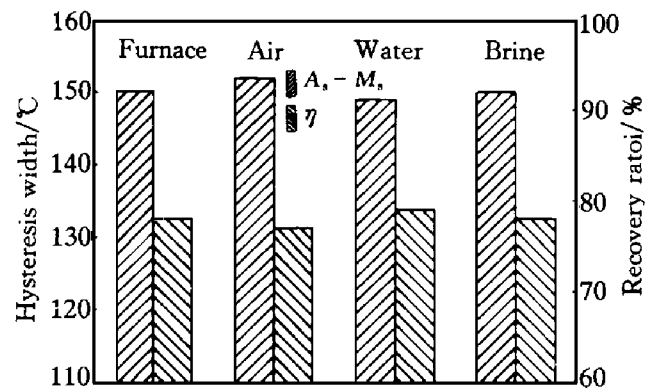


Fig. 7 Effect of cooling conditions on transformation hysteresis ($A_s - M_s$) and shape recovery ratio (η)

Appropriate transformation temperature, wide transformation hysteresis and good shape memory effect are key factors for the engineering application of NiTiNb alloy. The previous work by the present author showed that the change of the M_s temperature caused by the drift of the composition during melting can be eliminated by heat-treatment^[9]. A remelting process can be left out for regulating Ni_{47.3}Ti_{43.8}Nb_{8.9} alloy to Ni₄₇Ti₄₄Nb₉ alloy by adding pure constituent elements. Besides all facts above, it can

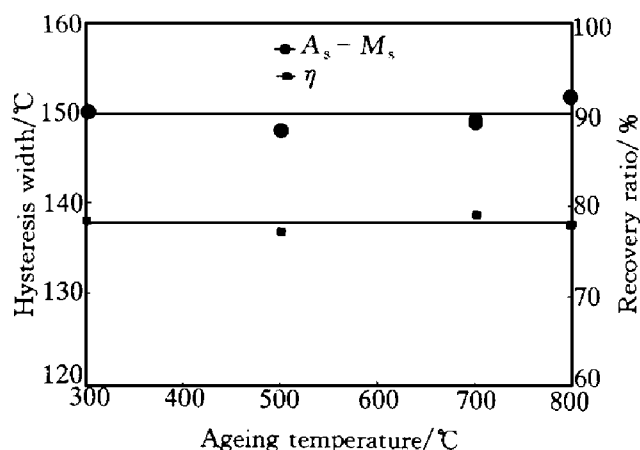


Fig. 8 Effect of aging temperatures on transformation hysteresis ($A_s - M_s$) and shape recovery ratio (η)

be concluded that, under the condition that there exists a slight deflection of composition from the designed composition, through heat-treatment we can regulate the M_s temperature to the designed M_s temperature without deteriorating the performance of wide transformation hysteresis and good shape memory effect after deformation. It can also be proposed that other NiTiNb alloys with near composition to Ni₄₇Ti₄₄Nb₉ alloy can be used in practical application as Ni₄₇Ti₄₄Nb₉ alloy after regulating its M_s temperature to that of Ni₄₇Ti₄₄Nb₉ alloy by heat-treatment.

4 CONCLUSIONS

(1) With the increase of solid solution temperature, cooling rate and aging temperature below 900 °C, yield stress decrease and elongation

increase; yet above 900 °C, yield stress shows no substantial change whereas elongation decreases abruptly.

(2) Once specimens deformed at the temperature ($M_s + 30$ °C) to 16%, transformation hysteresis width around 150 °C can be achieved and shape recovery ratio still remains 70% ~ 80% no matter which kind of heat treatment is taken.

(3) It is practicable to use NiTiNb alloys with near composition to Ni₄₇Ti₄₄Nb₉ alloy as Ni₄₇Ti₄₄Nb₉ alloy for engineering applications after certain heat treatment.

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