Evaluation of Degradation of Critical Stress for Inducing Martensite by Residual Martensite Volume Fraction in Ti-Ni Alloys

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The effect of cyclic deformation on the critical stress for inducing martensite in Ti-Ni alloys was investigated by performing isothermal cyclic tensile tests at temperatures of above A_r . The compositions of alloys were Ti-50.1Ni, Ti-50.4Ni and Ti-50.6Ni (at%), annealed at 623, 673, 723 and 773 K for 3.6 ks after cold drawing with 34% reduction. Furthermore, the effects of Ni content, shape memory treatment temperature, maximum strain and testing temperature on the critical stress for inducing martensite were investigated in relation to the residual martensitic volume fraction subjected to slip deformation. Results show that the residual martensite volume fraction represents changes in the critical stress for inducing martensites with number of cycles, Ni content, shape memory treatment temperature, maximum strain and testing temperature that the martensite volume fraction subjected to slip deformation subjected to slip deformation strain and testing temperature. Thus it can be stated that the martensite volume fraction subjected to slip deformation is capable of representing the effects of these conditions on the degradation of materials functions.

Key words: Ti-Ni alloy, critical stress for inducing martensite, residual martensite volume fraction, Ni content, shape memory treatment temperature

1. INTRODUCTION

The shape memory alloy is receiving attention in various fields. In areas such as engineering and medicines, its applications are being studied and practically used [1-3]. Ti-Ni alloy is practically used because it is superior to cyclic deformation, and so on. Therefore, the degradation of materials functions and fatigue properties have been important concerns, and the degradation of functions with number of cycles has been investigated [4-6]. It has been reported that it is effective for suppressing the degradation of functions to give a cold processing or optimize the heat treatment [4].

Authors have reported that the degradation of some functions in Ti-Ni alloys was caused by the residual martensite volume fraction subjected to slip deformation in specimens and the residual martensitic volume fraction is capable of representing the degradation of functions [7-9]. However, there was few works to investigate systematically the effects of test condition, composition, heat treatment condition on cyclic deformation. Therefore, it is practically important to clarify the these effects on cyclic behavior in Ti-Ni alloys.

The purpose of the present paper is to investigate the effects of cyclic deformation, maximum strain, testing temperature, shape memory treatment temperature and Ni content on the degradation of the critical stress for inducing martensite in Ti-Ni alloy wires.

2. EXPERIMENTAL PROCEDURE

Ti-Ni alloy ingots were made using a high frequency induction vacuum furnace. The compositions of the alloys were Ti-50.1Ni, Ti-50.4Ni and Ti-50.6Ni (at%), respectively. The ingots were hot-forged and hot-extruded followed by cold drawing and intermediate annealing to make wires with a diameter of 1.0 mm. The wires were cold-drawn with the reduction of 34% and were cut to 120 mm in length. The wire specimens were annealed at $T_{\rm HT}$ = 623, 673, 723 and 773 K for 3.6 ks. Surface layer of specimen was removed by pickling and electropolishing except for both ends of specimen which were clamping sections. The representative values of phase transformation

Table I Transformation temperatures and elastic moduli of Ti-50.6at%Ni alloy.

т _{нт} [K]	А _f [K]	А, [K]	М s [K]	м, [K]	E _A (parent) [GPa]	Е _м (martensite) [GPa]
623	321	285	257	198	60.9	22.5
673	314	288	251	200	67.1	18.9
723	315	301	254	210	67.4	17.4
773	307	293	249	221	67.8	18.0

Table II Transformation temperatures and elastic moduli of Ti-Ni alloys annealed at 673K.

Ni [at%]	A , [K]	A s [K]	М s [K]	М _f [K]	E _A (parent) [GPa]	Е _м (martensite) [GPa]
50.1	356	331	319	273	63.4	22.1
50.4	338	323	289	240	65.9	21.0
50.6	314	288	251	200	67.1	18.9



Fig. 1 Schematic drawing of stress-strain curve representing superelasticity characteristics.

temperatures and elastic moduli are listed in Table I and Table II. The transformation temperatures were measured by DSC (Differential Scanning Calorimetry). The elastic modulus E_A of parent phase and E_M of martensitic phase were obtained by isothermal tensile tests at temperatures of above A_f and 283K, respectively.

The testing temperatures $T_{\rm H}$ were 343, 353 and 363 K. The specimen was elongated up to a given maximum strain $\varepsilon_{\rm max}$ (=2, 4, 6%) under a constant strain rate (0.16 %/sec). The cyclic tests were performed repeatedly until the number of cycles N=100.

Figure 1 shows the schematic stress-strain curve representing the superelastic behavior for the Nth cycle. In the figure, σ_{Ms} is the critical stress for inducing martensite. ε_{Ms} is the strain which starts stress induced martensitic transformation. ε_{max} is the applied maximum strain. The area U is the strain energy per unit volume during loading (area of a-b-c-d-a). The area U_{MS} is the strain energy per unit volume which is needed to induce martensitic transformation (area of a-b-e-a).

3. RESULTS AND DISCUSSION

3.1 Cyclic behavior

Figure 2 shows the effect of cyclic deformation on the critical stress for inducing martensite σ_{Ms} for various (a) maximum strains ε_{max} (b) testing temperatures $T_{H'}$ (c) shape memory treatment temperatures T_{HT} and (d) Ni contents. The values are normalized against those of the first cycle and plotted as a function of number of cycles. The change of σ_{Ms} with number of cycles does not depend on ε_{max} . However, the change of σ_{Ms} with number of cycles depends on $T_{H'}$ and Ni content. σ_{Ms} decreases with increasing T_{H} and T_{HT} . The effect of Ni contents on the change of σ_{Ms} is significant and σ_{Ms} decreases with decreasing Ni content. It has been reported that the superelasticity characteristics against cyclic deformation are stabilized by the precipitation of Ti₃Ni₄ formed by aging treatment in Ti-Ni alloys with nickel content exceeding 50.5 at%[4]. So that, the change



Fig. 2 Effect of cyclic deformation on the critical stress for inducing martensite with various (a) ε_{max} , (b) T_{H} , (c) T_{HT} and (d) Ni content.



Fig. 3 Effect of cyclic deformation on the strain energies per unit volume at ε_{max} =4% and T_{H} =363K in Ti-50.6at%Ni annealed at 673K.

of $\sigma_{\rm M}$ in Ti-50.6at%Ni alloy is smaller than those of Ni contents below 50.4at%.

Figure 3 shows the effect of cyclic deformation on the strain energies U and $U_{\rm MS}$ at $\varepsilon_{\rm max}$ =4% and $T_{\rm H}$ =363 K in Ti-50.6at%Ni annealed at 673K. The energies U and $U_{\rm MS}$ decrease with increasing number of cycles. On the other



Fig.4 Relationship between $U_{Ms}(1)$ - $U_{Ms}(N)$ and the total strain enegy per unit volume during loading with various (a) ε_{max} , (b) T_{H} , (c) T_{HT} and (d) Ni content.

hand, the energy difference $U_{\rm MS}(1)$ - $U_{\rm MS}(N)$ increases. This energy difference means a stored energy in specimen after unloading. Therefore, it is thought that $\sigma_{\rm Ms}$ decrease with increasing $U_{\rm MS}(1)$ - $U_{\rm MS}(N)$.

3.2 Relationship between the strain energy and the critical stress for inducing martensite

Figure 4 shows the relationship between the stored energy and the total strain energy ΣU per unit volume during loading with various (a) ε_{max} , (b) $T_{\rm H}$, (c) $T_{\rm HT}$ and (d) Ni content. The stored energy increases with increasing the total strain energy, and shows the dependence on $T_{\rm H}$, $T_{\rm HT}$ and Ni content. The stored energy increases with increasing $T_{\rm H}$ and $T_{\rm HT}$, and decreases with increasing Ni content.

Figure 5 shows the relationship between the degradation of σ_{Ms} and the stored energy as a function of (a) test conditions of ε_{max} and T_{H^2} (b) Ni content and (c) T_{HT} . Values are normalized against those of the first cycle. The degradation of σ_{Ms} increases with increasing the stored energy. From these results, it is understood that the stored energy helps the martensitic transformation induced by stress.

Furthermore, as shown in Fig.5 (a) and (b), the stored energy represents the effects of the test conditions of ε_{max^3} $T_{\rm H}$ and Ni content on the degradation of $\sigma_{\rm Ms}$. However, the relationship between the degradation of $\sigma_{\rm Ms}$ and the stored energy depends on $T_{\rm HT}$ in Fig. 5 (c). It has been reported that the dislocation density formed by cold drawing suppresses the slip deformation and decreases with increasing $T_{\rm HT}$ [10]. It has also been reported that the increase in $T_{\rm HT}$ enlarges a precipitation size[11], and when the size of the precipitation becomes large, the effect which suppresses the slip deformation by the precipitation becomes small [12]. Thus it is thought that the synergistic effect of the dislocation density formed by cold drawing and the size of precipitation of Ti_3Ni_4 appears in the relationship between the degradation of σ_{Ms} and the stored energy.



Fig. 5 Relationship between $(\sigma_{Ms}(1)-\sigma_{Ms}(N))/\sigma_{Ms}(1)$ and $(U_{Ms}(1)-U_{Ms}(N))/U_{Ms}(1)$ with various (a) test condition, (b) Ni contents and (c) T_{HT} .

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Fig. 6 Relationship between $(\sigma_{Ms}(1)-\sigma_{Ms}(N))/\sigma_{Ms}(1)$ and ξ with various (a) T_{H} and T_{HT} and (b) Ni content.

3.3 Evaluation by residual martensite volume fraction

Change in superelasticity characteristics, i.e., the critical stress for inducing martensite and the dissipated strain energy per unit volume are caused by the slip deformation. Therefore, in this section, the discussion will focus on the influences of $T_{\rm HT}$ and Ni content on cyclic behavior. And the effects of $T_{\rm HT}$ and Ni content on change in the critical stress for inducing martensite with number of cycles will be discussed in relation to the martensite volume fraction ξ subjected to slip deformation. ξ is given by the following equation [7]. Where, $E_{\rm L}$ is the elastic modulus during elastic deformation in the strain range below 0.4%.

$$\xi = \frac{E_M \left(E_A - E_L \right)}{E_L \left(E_A - E_M \right)} \tag{1}$$

Figure 6 shows the relationship between $(\sigma_{MS}(1)-\sigma_{MS}(N))/\sigma_{MS}(1)$ and the martensite volume fraction subjected to slip deformation ξ as a function of (a) $T_{\rm H}$ and $T_{\rm HT}$ and (b) Ni content. From Fig.6, The degradation of critical stress for inducing martensite shows a strong dependence on the martensite volume fraction and increases with increasing ξ . The martensite volume fraction subjected to slip deformation is a basic degradation factor in SMA. Thus the change in the critical stress for inducing martensite in alloy with various Ni contents and shape memory treatment temperatures can be estimated by the martensite volume

fraction subjected to slip deformation.

4. CONCLUSION

The following conclusions were derived from the results and discussion:

(1) The stored strain energy in specimens after unloading represents changes in the critical stress for inducing martensite with number of cycles, Ni content, maximum applied strain and testing temperature. However, when shape memory treatment temperature is different in Ti-Ni alloys, it is not possible to represent unitedly the change in the critical stress for inducing martensite by using the stored energy.

(2) The martensite volume fraction subjected to slip deformation represents changes in the critical stress for inducing martensite with number of cycles, Ni content, shape memory treatment temperature, maximum applied strain and testing temperature. Thus it can be stated that the martensite volume fraction subjected to slip deformation is capable of representing these influences on the degradation of materials functions.

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