CHARACTERISTICS OF DEFORMATION AND TRANSFORMATION PSEUDOELASTICITY IN Ti-Ni ALLOYS

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Abstract. — Although the Ti-Ni alloy is the most important material for applications, there has been no systematic work on its deformation behavior. In the present report, the effects of ageing, Ni-content and annealing right after cold work on the shape memory effect and on the pseudoelasticity are systematically investigated by tensile tests at various temperatures. Based on the obtained results the differences in deformation and transformation pseudoelasticity characteristics between Ti-Ni alloys and other β-phase alloys are discussed.

Introduction. — The Ti-Ni alloy is the most important material among many shape memory alloys for the applications of both the shape memory effect and the pseudo-elasticity, because only this one deforms more than 50% strain prior to fracture and the strain as large as 8% recovers by heating above A_f or unloading in a poly-crystalline state (1). However no systematic work has been done from fundamental view point on the characteristics of deformation and transformation pseudoelasticity for this alloy, except for a few pioneering work (2)∿(5). In fact, there are many factors affecting the deformation behavior of this alloy, i.e. ageing effect, Ni-content, annealing right after cold work, and so on. In the present report the effects of these factors are examined systematically by tensile tests at various temperatures (77K∿373K).

It is well known that the characteristics of deformation and transformation pseudoelasticity in Ti-Ni alloys are remarkably different from those in other β-phase alloys. For example, the formers are very ductile (1,6). while the latters are not and shows the intergranular fracture (6,7). The transformation pseudoelasticity is always observed in the latters at temperatures above A_f (8)∿(10), while it is not necessarily so in the former, i.e. a Ti-50at%Ni alloy usually does not exhibit the pseudoelasticity even above A_f, while a Ti-51at%Ni alloy does so. This distinct difference will be explained by the fundamental deformation characteristics of Ti-Ni alloys, which are consistent with the above mentioned various effects.

Specimens and Experimental Procedures.— The alloy preparation was described in the previous paper (1). The compositions of the alloys used were determined by chemical analysis to be Ti-49.8at%Ni, Ti-50.1at%Ni, Ti-50.6at%Ni and Ti-51.6at%Ni (±0.1at%Ni)(the nominal compositions were Ti-50.0at%Ni, Ti-50.7at%Ni,Ti-51.0at%Ni and Ti-51.5at%Ni, respectively). The ingots of these alloys were hot rolled and then cold drawn at R.T. to wire specimen with diameter of 1.0 mm. The specimens were then heat-treated for 3.6ks at various temperatures in a range from 473K to 1273K in a vacuum of 10^{-3} Pa, and then rapidly quenched into ice water in order to investigate the effect of annealing temperature (from here the heat-treatment is abbreviated as 1273K IQ for example). In order to investigate the ageing effect, the same alloy specimens were aged for 3.6ks below 873K followed by quenching into ice water after the heat treatment of 1273K IQ (from here this heat treatment is abbreviated as 1273K IQ → XK IQ, where X ranges from 1073K to 473K). After these heat-treatments, wire specimens with diameter of 1.0 mm and with gauge length of 30 mm long were made by electropolishing. Tensile tests

were carried out with an Instron type tensile machine, Shimadzu Autograph DSS-10T-S type. The details of the specimen preparation and the tensile tests have been reported elsewhere (1,11).

Results.- As mentioned in the previous section, there are many factors affecting the deformation behavior of Ti-Ni alloys. In this section the effects of (a) ageing, (b) Ni-content and (c) annealing right after cold work are described in the follwing in that order.

(a) Effect of ageing.- Heat treatments of shape memory alloys have generally been conducted by solution treatment at high temperature followed by quenching rapidly into water in order to obtain a high temperature single phase, which causes the shape memory effect to appear. A Ti-Ni alloy, however, is an exceptional case which shows the shape memory effect regardless of the heat-treatment, i.e. rapid quenching into water or gradual cooling in a furnace. Moreover, the characteristics of pseudoelasticity of this alloy in a slowly cooled specimen are better than those in a rapidly quenched one (unpublished work by present authors). This effect can be attributed to the ageing effect at low temperature during cooling, which produces fine precipitates. The presence of fine precipitates raises the flow stress for slip, and thus makes the stress-induced transfomation pseudoelastic as shown later (Fig. 2).

Fig. 1 Effect of ageing temperature on the electrical resistance-temperature curve in a Ti-50.6at%Ni alloy.

It is convenient to examine the electrical resistence-temperature curve as a function of ageing temperature in order to assess the most effective temperature for ageing. Figure 1 shows the curves of Ti-50.6at%Ni alloy specimens, which were subjected to the heat-treatment : 1273K IQ → XK IQ, where X ranges from 1073K to 473K. By ageing at temperatures between 1073K and 873K the curves change little. But, by ageing at temperatures between 773K and 573K the shape of the curves depend strongly on ageing temperature, especially that aged at 673K shows the biggest change among them. The specimens aged at higher temperatures scarcely showed the pseudoelasticity at any deformation temperature. However, the specimens aged at low temperatures showed perfect pseudoelasticity above A_f. The critical stresses for inducing
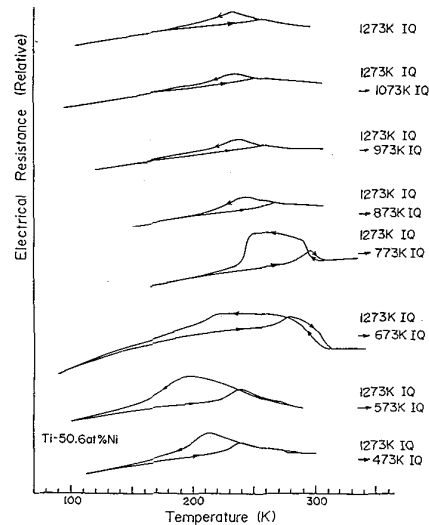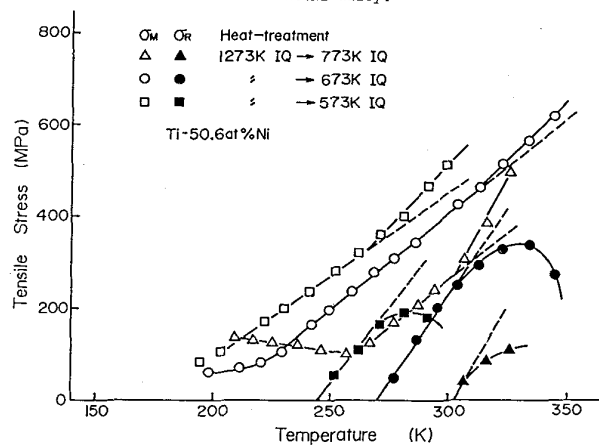

Fig. 2 Effect of ageing on the critical stresses for inducing martensites(σ_M) and for reverse transformation(σ_R) in a Ti-50.6at%Ni alloy.

martensites (σ_M) and for the completion of the reverse transformation (σ_R) are plotted against deformation temperature in Fig. 2. Both the stresses were found to follow the Clausius-Clapeyron relationship in the temperature region $A_f < T < T_C$. Here T_C represents the critical temperature where the plastic deformation by dislocation motion starts, and thus above T_C the critical stresses deviate from the linear relation with temperature as shown in the figure. The specimens aged at 673K showed the perfect pseudoelasticity without residual strain until σ_M reaches about 500MPa, which is the largest value among those of aged specimens. It is concluded from these results that the ageing treatment at 673K is most effective in improving the characteristics of pseudoelasticity.

(b) Effect of Ni-content. –
Although the phase diagram of the Ti-Ni system is not well-established as yet, there is a general agreement on the presence of some precipitate phases such as $TiNi_3$ and/or $Ti_{42}Ni_{58}$ on the Ni-rich side of these diagrams (12,13). Thus the ageing effect may also depend on Ni content as well as on ageing temperature. The electrical resistance-temperature curves of specimens with various Ni-content whose heat-treatments are 1273K IQ and 1273K IQ $\rightarrow$ 673K IQ, respectively, are shown in Fig. 3. The shape of curves and transformation temperatures are almost unchanged by the ageing treatment in a Ti-49.8at%Ni alloy. But, the change in the shape of curves by ageing at 673K are more remarkable with increasing Ni-content. The critical stresses σ_M and σ_R are shown as a function of deformation temperature in Fig. 4. The values σ_R in Ti-49.8at%Ni and Ti-50.1at%Ni alloys are absent, because the pseudoelasticity was not observed in these specimens. But in Ti-50.6at%Ni and Ti-51.6at%Ni alloys the pseudoelasticity is observed in a wide temperature range, and the deviation of the stress σ_M from the linear relation with temperature is observed at about 600MPa in a Ti-51.6at%Ni alloy, which is a clear indication for the onset of slip. From these results it is concluded that the effect of ageing is more prominent in a high Ni-content specimen than in a low Ni-content one.
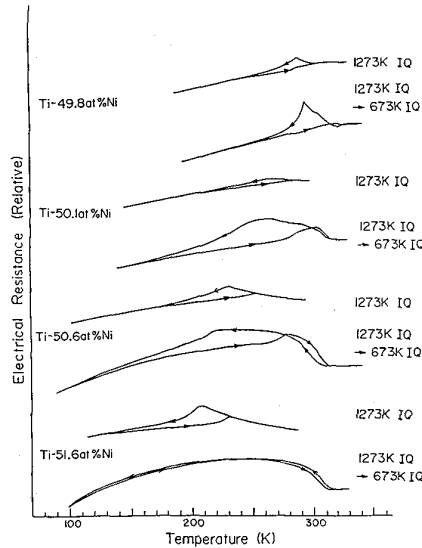

Fig. 3 Effect of Ni-content on the electrical resistance-temperature curve in Ti-Ni alloys.
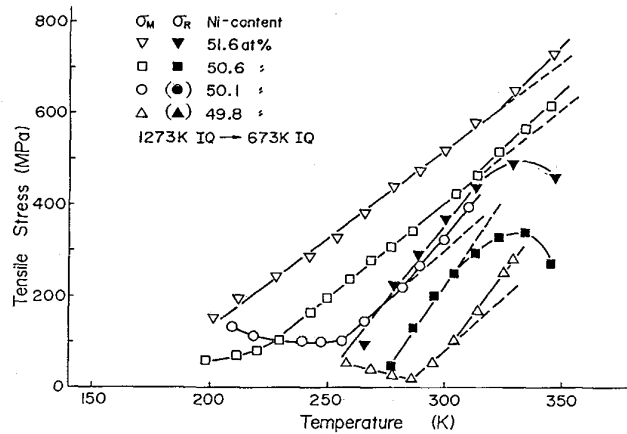

Fig. 4 Effect of Ni-content on the critical stresses for inducing martensites(σ_M) and for reverse transformation (σ_R).

(c) <u>Effect of annealing right after cold work</u>. – As mentioned previously the effect of ageing was most prominent in a Ti-51.6at%Ni alloy and was not appreciable in a Ti-49.8at%Ni alloy. This means that a specimen with low Ni-content does not exhibit the pseudoelasticity even above A_f, while that with high Ni-content does so. However, we found another effect which improved remarkably the characteristics of pseudoelasticity even in a specimen with low Ni-content. Figure 5 shows the effect of annealing right after cold work on the electrical resistance-temperature curve of a Ti-49.8at%Ni alloy specimen, which was annealed at each temperature without solution treatment after cold drawn. The curve exhibits little change in a specimen annealed above 873K. However, the curves for specimens annealed below 773K show very conspicuous change which is very similar to that observed in the Ti-50.6at%Ni alloy aged below 773K. The critical stresses σ_M and σ_R in the specimens annealed at 1273K, 773K and 673K, respectively, are plotted against deformation temperature in Fig. 6. The specimen annealed at 1273K did not show the pseudoelasticity at any deformation temperature and, thus, σ_R was absent. However, the specimens annealed at lower temperatures showed the pseudoelasticity, and especially that annealed at 673K showed the most superior characteristics of pseudoelasticity. The stress-strain curves as a function of deformation temperature for a specimen annealed at 673K and those for a specimen annealed at 1273K and then annealed at 673K are compared in Fig. 7. The specimens were loaded and unloaded at each deformation temperature, and then heated to 373K(>A_f) in order to make the total strain to be separated into the recoverable strain and the residual strain, as shown by the dotted curve. Permanent residual strain more than one percent took place as shown by curve (a) in the specimen annealed at 1273K followed by annealing at 673K. This residual strain caused the specimen to show the two way shape memory effect as shown by curve (b) when it was deformed after the preceding deformation. On the contrary, in the specimen annealed at 673K the perfect shape memory effect was observed below A_s and the pseudoelasticity was observed
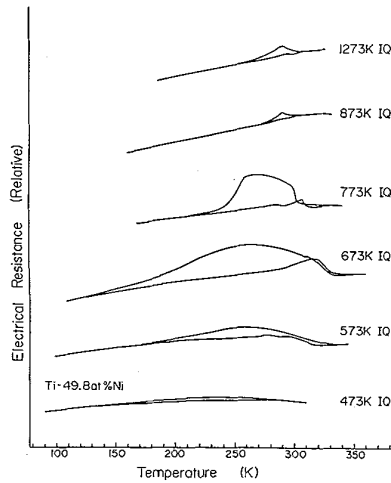

Fig. 5 Effect of annealing temperature on the electrical resistance-temperature curve for Ti-49.8at%Ni alloy specimens, which were annealed at each temperature without prior solution treatment after cold work.
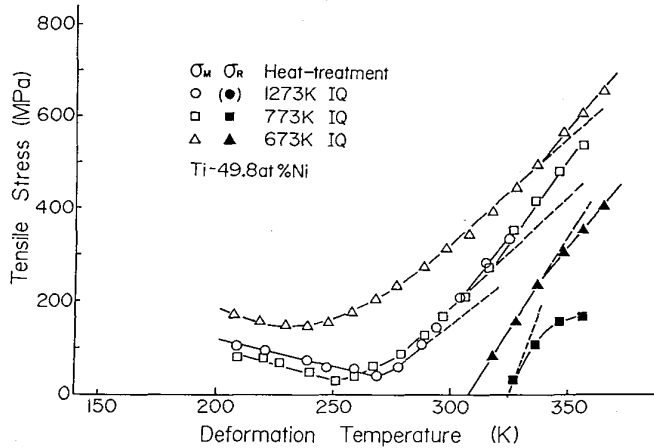

Fig. 6 Effect of annealing temperature on the critical stresses for inducing martensites(σ_M) and for reverse transformation(σ_R) in Ti-49.8at%Ni alloy specimens, which were annealed at each temperature without prior solution treatment after cold work.
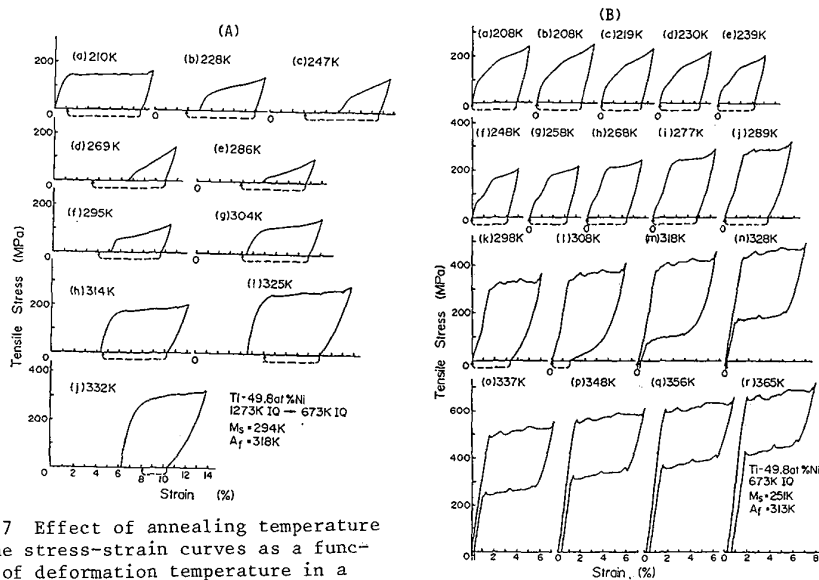
Fig. 7 Effect of annealing temperature on the stress-strain curves as a function of deformation temperature in a Ti-49.8at%Ni alloy.
(A) Heat-treatment : 1273K IQ — 673K IQ.
(B) Heat-treatment : 673K IQ.

in a wide temperature range as shown in the right-hand side of Fig. 7. The improvement of the characteristics of pseudoelasticity can not be attributed to the ageing effect due to precipitation, because both specimens were aged for 3.6ks at 673K. Therefore, it is most likely that the effect of annealing at lower temperatures is caused by the internal structure of dislocations which were introduced during drawing at R.T. and then rearranged by annealing so as to diminish the internal strain energy. The presence of such rearranged dislocations again raises the flow stress for slip, and thus results in the appearance of pseudoelasticity at temperatures above A_f.

It is also noticed in the figure that there are two plateaus in the stress-strain curves in low temperature range. The critical stress for the second plateau corresponds to the stress σ_M, but that for the first plateau was found to be the stress for rearrangement of the rhombohedral phase variants, which appear prior to the onset of the martensitic transformation during cooling in the absence of stress (5,14,15). This is confirmed by the fact that the extrapolated point of the latter stress corresponds to the temperature at which the rhombohedral phase is induced thermally. The details about this will be published soon elsewhere.

Discussion. — As mentioned in the introduction, a Ti-Ni alloy is very ductile and the transgranular fracture with many dimples on the fracture surface is observed, while a Cu-Al-Ni alloy is very brittle in a polycrystalline state and the typical intergranular fracture occurs. These differences of deformation and fracture behavior in both alloys were attributed to the large difference in the elastic anisotropy of these alloys (6,16,17). The elastic anisotropy of a Cu-Al-Ni alloy is known to be about 13 (18), while that of a Ti-Ni alloy is about 2 (19). However, there is another important factor which controls the deformation and fracture behavior. If a deformation mode is available at low stress levels the stress at grain boundaries is easily relaxed by this deformation mode. In a Ti-Ni alloy which is not given a special heat-treatment, dislocations move easily at a stress of about 100MPa as

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