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Internal Friction in Two-Stage Martensitic Transformation of Ni-Ti Alloy

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Abstract. Transformation sequences in a NiTi shape memory alloy after deformation and subsequent annealing were studied. For specimens deformed by rolling between 5 and 10% and then annealed in the range between 673 K and 823 K, the internal friction curve exhibits a broad peak in the range of martensitic transformation which is the result of the overlapping of two peaks. Similar effects were also observed using the DTA method. The Granato-Lücke analysis shows that the two peaks may be correlated with the difference in the dislocation densities existing in the specimens. It is suggested that the specific arrangements of dislocations cause non-homogeneous internal stresses which are responsible for the two-stage martensitic transformation.

1. INTRODUCTION

Deformation and subsequent annealing of NiTi alloys offers great possibilities of controlling characteristic temperatures, sequences of transformations and shape recovery. Todoroki and Tamura [1] showed that depending on annealing temperature, changes in transformation sequences in deformed alloys occur during both heating and cooling. They also observed that the cooling DSC curves exhibit two peaks in the range of the martensitic transformation for specimens annealed at 773 K. Referring to the paper of Monasevich and Pascal [2] they explained this effect as the formation of two types of martensite. The X-ray studies of the reversible martensitic transformation for the cold rolled and annealed alloy at temperatures before recrystallization starts showed that this transformation is a one-step transition and results in the forming of the monoclinic B19' martensite [3]. The purpose of this paper was to prove by internal friction (IF) measurements that the appearance of two peaks in the martensitic transformation on the DTA cooling curves may be due to the inhomogeneity in the stress field which results from the changes in the dislocation configuration during the recovery process.

2. EXPERIMENTAL PROCEDURE

A commercial NiTi shape memory alloy containing 50.6 at. %Ni was used in this study. Specimens in shape of strips were quenched from 1073 K after annealing for 1h and then cold-rolled by 5% or 10% reduction. The deformed samples were then annealed at various temperatures between 573-973 K. The transformation behaviour and the characteristic temperatures were determined using the DTA (differential thermal analysis) method on the TA 1 Mettler instrument.

The internal friction and vibration frequency as a function of temperature were measured using an acoustic frequency relaxator. To determine the dislocation density for the internal friction peak related to the particular transformations, measurements of amplitude dependences of the internal friction were carried out. The IF-amplitude dependences were conducted isothermally for the transformation peaks and at temperature related to the parent phase at $T=293$ K.

3. EXPERIMENTAL RESULTS

The effect of annealing of the deformed alloy on the course of transformation was studied using the DTA method. Fig.1 shows the DTA cooling curves for the alloy deformed by 10%, and then annealed at various temperatures.

The curves of specimens annealed at 873-973 K show only one peak corresponding to the one step transformation from the parent to martensitic phase B2→B19'.

The curve of the deformed alloy shows a strongly spread peak of martensite transformation on which overlapping of two effects may be distinguished, i.e. that of the R-phase formation and, at lower temperatures, of the martensite formation. Annealing of the alloy at 573 K causes distinct separation of those two effects. The separation becomes even more visible when the annealing temperature increases up to 823 K. Fig. 1 also illustrates changes of the characteristic temperatures of both transformations versus the annealing temperature. The increase of the annealing temperature up to 873 K causes disappearance of the R-phase peak. The DTA curves obtained during cooling of specimens annealed at 673-823 K (except for the specimen annealed at 723 K) show three peaks, one from the R-phase formation and two in the range of the martensitic transformation.

The IF curves exhibit an identical course of changes as the DTA curves. Fig. 2 shows the internal friction curve after quenching from temperature 1073 K at which one peak occurs corresponding to change B2→B19'.

Fig. 1. The DTA cooling curves for the alloy deformed by 10% and annealed at various temperatures

Fig. 2. Internal friction and the square of resonant vibrations frequency vs. temperature for NiTi quenched from 1073K

Fig. 3 shows the IF curve of a deformed specimen by 10% and annealed at $T=823$ K. The curve exhibits the presence of 3 peaks: peak (a) corresponding to the parent phase transformation into R-phase-preceding the actual martensitic transformation B2→R. The subsequent peaks (b) and (c) are related to the transformation R→B19'.

Fig. 3. Plots of curves $Q^{-1} = f(T)$ and $f^2(T)$ for NiTi deformed by 10% and annealed for 1 hour at 823 K

Fig. 4 presents the IF curves versus amplitude obtained isothermally and corresponding to the 3 peaks of transformations (a,b,c) and to the parent phase (d).

The analysis of curves obtained was carried out using the Granato-Lücke model. Fig. 5 depicts plots of $\ln(Q^{-1} \cdot \epsilon)$ vs. $1/\epsilon$ obtained from the function $Q^{-1} = f(\epsilon)$ given in Fig. 4.

Fig. 4. The IF amplitude dependences obtained for NiTi deformed by 10% and annealed at 823 K determined at: a - 250 K, b - 230 K, c - 204 K, d - 293 K

Basing on the determined parameters of lines from Fig. 5 and applying the method described earlier in [4] the ratios of dislocation densities occurring in the martensitic phase in the temperatures of peaks to the dislocation densities in the parent phase $\left(\frac{\Lambda_a}{\Lambda_d}\right)$ were established. It was found that the dislocation density at the temperature of the first B2→R transformation is 4.5 times higher than in the parent phase. In case of temperatures at which maxima b and c occur the dislocation densities are higher than in the parent phase by a factor 8.0 and 5.5, respectively.

Fig. 5. Granato-Lücke plots obtained from curves of IF amplitude dependences shown in Fig. 4

4. DISCUSSION

The specimens deformed and annealed at low temperature (573-823 K) show on the DTA and IF cooling curves a following course of transformations: B2→R→B19'. However the peak corresponding to the martensitic transformation exhibits a splitting of this peak. The Granato-Lücke analysis shows that the two peaks may be correlated with the difference in the dislocation densities existing in the specimens. These results are confirmed by electron microscopy observations which show a dislocation cell structure. Dislocation density decreases with annealing temperature and after annealing at 823 K areas of regular network of dislocations creating subgrains of low angle boundaries can be seen. These dislocation networks are due to the polygonization process. Annealing at 873 K causes full recrystallization of the deformed alloy, and for this specimen only one peak of the martensitic transformation can be seen on the DTA and IF cooling curve. This confirms the hypothesis that the two peaks on the DTA and IF cooling curves in the martensite range are caused by inhomogeneity in the stress field resulting from changes in the dislocation configuration during the recovery process. Separation of the martensitic peak into two has recently been observed, [5] during thermal cycling. The authors concluded that the martensitic transformation was affected by strain fields occurring in the untransformed parent phase and that the additional peak on the DSC curves results from locked-in transformation energy in the self accommodating martensite microstructure. Our main conclusion is that the martensitic transformation in the NiTi alloys deformed and annealed below recrystallization temperature, is a one-step transition. The presence of two peaks on the IF cooling curves in the martensitic range is caused by the inhomogeneity of the stress field resulting from changes in the dislocation configuration during the recovery process.

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