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High Temperature Internal Friction in Ni Ti Alloy

V. Pelosin, F. Deborde and A. Rivière

Laboratoire de Mécanique et Physique des Matériaux, URA 863 du CNRS, ENSMA, BP. 109, 86960 Futuroscope cedex, France

Former internal friction studies concerning NiTi alloys are currently limited to the temperature range 300 K-400 K relative to the martensitic transition. This paper describes results obtained by isothermal mechanical spectroscopy between 400 K and 1050 K with a sample in austenitic phase after different thermomechanical treatments: cold rolled, deformed by tension, submitted to successive transition cycles or annealed at high temperature. Two relaxation peaks superimposed to a low frequency (i.e. high temperature) background were found. For the first one (520 K at 1 Hz) both, the variation of the relaxation strength with the measurement temperatures and the relaxation parameters ($E_a = 1.35$ eV and $\tau_0 = 2.5 \times 10^{-14}$ s) correspond to a Zener relaxation. The high temperature peak (870 K at 1 Hz) was associated with a relaxation due to dislocation segment motion.

I. INTRODUCTION

A large volume of data on internal friction measurements in NiTi has been published [1-3] but they mainly concern the low temperatures behaviour that correspond to the martensitic or premartensitic transition (200K-400K). Unlike the martensite which exhibits an high damping capacity, the internal friction measured in the austenitic state is low. However, between 500K and 600K it increases sharply. The purpose of this work is to identify the present effect, that is to say the manifestation of two different peaks superimposed to an important background.

2. EXPERIMENTAL PROCEDURE

A commercial NiTi shape memory alloy was used in this study (49.6 at. % Ni). The samples were cut in flat bars of dimension $50 \times 6 \times 1 \text{ mm}^3$ from 1mm sheets obtained by cold rolling. The characteristic transformation temperatures measured by DSC are $M_s=350\text{K}$, $M_f=325\text{K}$, $A_s=360\text{K}$ and $A_f=380\text{K}$. Internal friction measurements were carried out with an inverted torsional pendulum subjected to subresonant forced vibrations over a large frequency range (10^{-4} to 40Hz) with ten discrete frequencies per decade. The damping is then calculated from the determination of $\tan\phi$ where ϕ is the phase angle between the applied stress and the resulting strain: $Q^{-1} = \tan\phi$. Isothermal measurements are performed after the stabilization of the temperature, and above all, of the sample microstructure. Q^{-1} was determined for a maximum vibration amplitude of 5×10^{-6} .

3. RESULTS

3-1. Annealing temperature 473K-573K

Figure 1 shows experimental values of internal friction versus frequency realized in isothermal conditions at 523K. The curve is typical of spectra obtained between 513K and 573K. It could be observed that the low frequency background is increased (considering the other results, it has been found that the increasing depends on the temperature). Moreover, a high frequency peak is superimposed to the background. A correct treatment of the data enables to reveal the peak (logarithmic background subtraction). Figure 2 gathers the peaks obtained successively at 513K, 523K, 531K and 542 K (in isothermal condition). The peak position is shifted toward the high frequencies when the temperature is increased: such a behavior is typical of a relaxation process.

Figure 1.(a): Internal friction measured at 523K in NiTi. Dashed line: background and (b): resulting peak.

Figure 2 Relaxation peaks observed in isothermal conditions at successively 513K, 523K, 531K and 542 K.

The same experiments have been performed on a sample annealed at 1073K during ten hours then furnace cooled at 4K/min. As it is shown on figure 3 (curve a), the damping measured at 541K leveled between 5 and 10×10^{-4} in the whole frequency range and the presence of a peak has not been detected. Then, this sample has been submitted to thirty transition cycles, by performing annealing runs between 300K and 423K. The spectrum recorded at 524K (curve b) indicates that the background and the peak are restored by the previous thermomechanical treatment. Curve c, illustrates the damping evolution at 541K of the initial state sample (cold rolled). It is clear that both the background and the peak are evidenced by introduction of defects due to the cold rolling or to the successive transition cyclings. A more detailed study shows that the peak amplitude is considerably reduced in the case of the cycled sample by comparison to the initial state.

Figure 3. Curve (a): internal friction measured at 541K in a NiTi sample cooled slowly from 1073K. Curve b: measurements performed at 524K on the same sample which has been submitted to thirty transition cycle and curve c: internal friction of a cold rolled sample

Figure 4. Arrhenius plot obtained from the relaxation peak of the cycled, and the cold rolled samples

The activation energy, E_a , and the relaxation time, τ_0 , are calculated from the Arrhenius plot (figure 4). In the two cases, highly strained sample or cycled one, the experimental data are lined up then the process could be the same. The calculated values obtained for the activation energy and the time relaxation are $E_a=1.35\text{eV}$ and $\tau_0=2.5 \times 10^{-14}\text{ s}$.

3-2 Annealing temperature 745K-1065K

The annealing temperature has been increased on the initial state sample (cold rolled). The internal friction spectra obtained at various temperatures are reported on figure 5. Once again, one could observe on the curves a, b and c, a low frequency increase which depends on temperature. The background of the curve d, realized at 866K, seems to be stabilized as it looks not so different from the one measured at 834K, curve c. But, the most striking feature happens at 925K (curve c) where the background collapses dramatically and a peak (already visible on curve d), is clearly evidenced around 10Hz.

Figure 5. Internal friction spectra obtained on increasing temperature at 745K: a, 783K: b, 834K: c, 866K: d and 925K: e.

Figure 6. Evolution of the relaxation peak with the annealing temperature. Curves b, c, d and e corresponds to the same temperatures than on figure 5.

Figure 7. Arrhenius plot corresponding to the peak obtained on cooling after annealing at 958K and at 1065K.

Figure 6 gives the evolution of the peak height and position with the annealing temperature. As shown, the peak is shifted to the high frequencies and its amplitude growth up when the temperature is increased. The mechanisms responsible of the effect could be reasonably ascribed to a thermally activated relaxation process. Curve a has not been reported on figure 6 as no peaks could be extracted from the data. The sample has to be heated at 750K at least in order to induced the appropriate structural modifications, which give rise to the relaxation phenomenon. Some complementary information is given by the damping measured on cooling after annealings performed at 958K and at 1065K. In the two cases the peak is still present but after the 1065K annealing its amplitude is reduced and, as shown on figure 7, its frequency is lowered. The activation energy calculated from the Arrhenius plot (figure 7) are $E_a=3.2\text{eV}$ after the two annealings. The relaxation time is slightly changed from 1.5×10^{-19} s (after 958K annealed) unto 2.7×10^{-19} s (after 1065K annealed).

4. DISCUSSION AND CONCLUSION

The first peak observed is closely linked to structural disorder. Indeed the process exists only for sample which have been submitted to specific thermomechanical treatments as cold rolling, fast cooling or several transition cyclings. All these two treatments are well known to introduced precipitates and structural defects in a general way [4-5]. We could then assumed that a relaxation of a Zener type could happen in local disorder area. The peak width is larger than a Debye's peak one, a partial ordering is then more probable than an orientation of the substitutional atoms [6].

Concerning the second peak, observed at higher temperature, several remarks can be done. First, on heating, its presence is associated with a dramatic decrease of the low frequency background. It is undoubtedly the manifestation of a thermally activated relaxation process as it is shifted to the high frequencies when the temperature is increased. The peak amplitude is slightly decreased with further annealings, 958K then 1065K, and the relaxation frequency is diminished. On the other hand the activation energy is not changed by the thermal treatments. This behavior is quite similar to this observed in pure aluminum [7]. In that case, the authors had assumed that the background collapse was due to the ordering of the strain hardening cells. By analogy with this work, we have assumed that a convenient annealing enables the arrangements of the grain orientation. Then the dislocation segments could growth up and move easily in a reversible manner that gives rise to the relaxation peak.

Nevertheless the interpretation of the two peaks described in this paper needs further experimental confirmations to conclude definitively on their origins.

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