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DISLOCATION RELAXATION IN THE MARTENSITIC PHASE OF THE THERMOELASTIC NiTi AND NiTiCu ALLOYS

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Abstract. - The internal friction has been measured in the martensitic phase of NiTi(Cu) alloys at 1 Hz and 4 kHz. The detailed study of the strain amplitude dependence of the internal friction and the whole spectrum as a function of the temperature in the martensitic phase have allowed to attribute to dislocations the main source of internal friction.

1. Introduction. - The alloys which undergo a thermoelastic martensitic transformation exhibit during the transformation and in their martensitic phase a high value of the internal friction (fig.1),(1,2). This phenomenon has been related to the viscous motion of interfaces between the different variants of martensite under the effect of an applied stress, and more precisely in the NiTi(Cu) alloys (2), to the viscous motion of twinning dislocations inside the martensite plates. However, the precise nature of these mechanisms is still not well known.

Internal friction measurements as a function of temperature or applied stress allow to clarify certain mechanisms of damping due to dislocation motion. The internal friction spectrum as a function of temperature displays several peaks related to the dislocation motion, i.e. Bordoni and Hasigutti peaks (3). Measurements as a function of the strain can be compared with the Granato-Lücke theory (4) or the De Jonghe model (5). These models permit to discuss experiments conducted in the martensitic phase of NiTi(Cu) alloys in terms of dislocations.

2. Experimental. - The studied alloys, provided by Brown Boveri (Baden), have been elaborated according to the standard technique described in (2). The internal friction Q⁻¹ and the frequency f have been measured as a function of the temperature T during linear heating (or cooling) or as a function of the strain amplitude ε. The measurements made at 1 Hz have been done in an inverted torsional pendulum (6) on 50 mm long and 1 mm diameter wires. The measurements at 4 kHz were done in a resonant bar apparatus (7) on 40 mm x 5 mm x 1.5 mm plates.

3. Results. - A) Measurements as a function of the strain
The measurements in the range of low strains have been carried out at 4 kHz in a near *Present address : Abt. Sicherheit 32, Eidg. Institut für Reaktorforschung, CH-5303 Mülrenlingen, Switzerland
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equiatomic NiTi alloy which has undergone two different treatments: annealing 30 minutes at 600°C then furnace cooled (fig.1a, curve 1), annealing 2 hours at 600°C then quenched in cold water (fig.1a, curve 2). In each case (fig.2) the value of $Q^{-1}$ initially low increases for strains between $10^{-7}$ and $5 \times 10^{-7}$, then remains nearly constant for strains up to $3 \times 10^{-6}$. These results reported in a Granato-Lücke plot $\ln(Q^{-1},\varepsilon) - \varepsilon^{-1}$ (fig.2) show that the behaviour of $Q^{-1}$ in the range of strains $10^{-7} - 5 \times 10^{-5}$ is consistent with the Granato-Lücke theory (linear part of the plot). This means that a depinning of the dislocations from weak pinners occurs in this range of strains. At higher strain, the applied stress is sufficient to induce a dislocation motion by successive depinning and $Q^{-1}$ is nearly strain independent. From these results, it appears that the dislocations are responsible for the internal friction in the range of strains $10^{-7} - 3 \times 10^{-6}$.

In the range of high strains, the same kind of measurements have been carried out at 1 Hz in the martensitic phase of a wt % 47 Ni 46 Ti 7 Cu previously annealed at 690°C then quenched in cold water (fig.3). $Q^{-1}$ is constant in the range of strains $10^{-6} - 2 \times 10^{-5}$, which is in good agreement with the previous measurements, then increases linearly with the strain from $2 \times 10^{-5}$ to $2 \times 10^{-4}$. This last result is consistent with the De Jonghe model which predicts a linear dependence of $Q^{-1}$ with the stress if this stress is larger than a critical one for the martensite reorientation. The reorientation of the martensite is provided by the motion of interface dislocations between two variants. Thus, the internal friction in the high strain range should be analysed in terms of long range motion of dislocations.

B. Dislocations and relaxation peaks

The low frequency internal friction spectrum measured between 15K and 260K is reported in fig.4 for two different alloys. In the at.% 50 Ni 50 Ti alloy which was previously annealed 1 hour at 590°C then quenched in cold water (curve 1), $Q^{-1}$ increases continuously from 15K to 80K and then remains constant for temperatures up to 150K above which a peak appears. The wt% 49.7 Ni 45.3 Ti 5 Cu alloy was degassed during 8 hours at 1000°C under $2 \times 10^{-10}$ torr in order to eliminate interstitial impurities. In this alloy, the behaviour of $Q^{-1}$ is similar to the previous one between 15K and 150K, but no peak is observed at higher temperatures. For both alloys, no noticeable effects appear on the frequency spectrum and $Q^{-1}$ is strain independent in the range of strains $10^{-6} - 2 \times 10^{-5}$. $Q^{-1}$ between 15K and 150K should be interpreted by a dislocation relaxation (Bordoni type peak) for which the relaxation time is the sum of two terms (B,3): the first one $\tau_1$ related to the temperature by an Arrhenius law is responsible for the increasing part of $Q^{-1}$ (low temperature side of the peak) and the second one $\tau_2$ related to the viscous friction of the dislocations with the impurities and slightly temperature dependent is responsible for the constant part of $Q^{-1}$.
The general expression of these peaks is

\[ Q^{-1} = g^2 \frac{A l^2}{6} \frac{1+\beta \exp(\Delta x)}{2\text{ch}(\Delta x)+\beta^2 \exp(\Delta x)+2\beta} \]

with \( \Delta x = \frac{E}{K} \left( \frac{1}{T_\text{p}} - \frac{1}{T} \right) \) and \( \beta = \omega \tau_2 \)

where \( g^2 \) = orientation factor, \( A \) = dislocation density, \( l \) = dislocation length, \( E \) = relaxation energy, \( K \) = Boltzmann constant, \( T_\text{p} \) = peak temperature, \( \omega \) = pulsation. Theoretical curves of \( Q^{-1} \) as a function of \( \Delta x \) for several values of \( \beta \) are shown in fig.5. The comparison with the experimental results between 15K and 150K (fig.4) permits to estimate \( \beta > 1 \) and to situate the peak temperature between 50K and 80K. The density of mobile dislocations \( A \) can be evaluated to \( 10^{19} \text{ cm/cm}^3 \) with the following reasonable values of the parameters: \( g^2 = 0.1, \ l = 10^{-2} \text{ cm}, \ \beta = 1 \). This value of \( A \) is too high if ordinary slip dislocations are supposed to be involved in this peak. The involved dislocations are rather imperfect dislocations with Burgers vector smaller than the lattice parameter, that is to say, twinning dislocations \([2] \). However the dislocations situated at the interface between the different variants of martensite, at present stage, cannot be ruled out.

In the kHz range, a corresponding peak should occur at higher temperature than in the Hz range. The measurements carried out in the near equiatomic NiTi alloy reported in fig.1a show an incertitude about the presence of a low temperature peak, except perhaps after quenching (curve 1) where \( Q^{-1} \) goes through a maximum between 80K and 130K. If we attribute to the peak situated between 50K and 80K at 1 Hz a value of the frequency factor of \( 10^{10} \text{ Hz} \) and an activation energy between 0.1 and 0.2 eV, the peak should occur in the range of temperatures 80K-130K. This is observed in fig.1, curve 1.

4. Discussion.- It has been shown that the main source of internal friction in the martensitic phase of the NiTi(Cu) alloys is arising from imperfect dislocations. The motion of such dislocations is sensitive to pinner defects and to viscous friction arising from the lattice or some distribution of surrounding defects. Furthermore, the long range motion of these dislocations should provide a good explanation for martensite reorientation. This assumption is in good agreement with results obtained by tensile tests which show that the reorientation occurs at lower stresses than that required for plastic deformation \([9,10] \). The presence of a low temperature peak (Bordoni type peak) due to the motion of these dislocations dealing with a Burgers vector smaller than the lattice parameter should be affected by a Peierls potential high enough to hinder martensite reorientation towards low temperature. This assumption is confirmed by tensile tests results \([10]\) which show an increase of the critical stress for martensite reorientation when the temperature decreases.
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Fig. 1 Internal friction and frequency spectra as a function of temperature during cooling in a near equiatomic NiTi alloy at 4 kHz (a) and in a wt% 47 Ni 46 Ni 46Ti 7 Cu alloy at 1 Hz (b). In the two cases, the three characteristic regions appear: parent phase region (low internal friction), transformation region (peak), martensitic region (high internal friction).
Fig. 2 Internal friction as a function of the strain $\varepsilon$ measured at about 4 kHz at 130 K in a near equiatomic NiTi alloy and the corresponding Granato-Lücke plot $\ln (Q^{-1}\varepsilon) - \varepsilon^{-1}$

Fig. 3 Internal friction as a function of the strain $\varepsilon$ in the martensitic phase of the wt% 47 Ni48Ti7Cu alloy of fig. 1 at 1 Hz

Fig. 4 Internal friction and frequency spectra as a function of the temperature for a near equiatomic NiTi alloy and a wt% 45.3Ni49.7Ti5Cu alloy

Fig. 5 Theoretical plot of the internal friction $Q^{-1}$ as a function of $\Delta \chi$ for the two relaxation times model (8)