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Dynamic Mechanical Spectroscopy of Nanograined Thin NiTi Wires

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Introduction

Thermomechanical behaviour of NiTi SMA is due to the complex interaction between several deformation mechanisms including lattice elasticity, phase transformations between austenite (A), martensite (M) and R-phase (R), reorientations of R and/or M variants, twinning and detwinning phenomena and the usual plasticity due to dislocation movements. As regards the elasticity, it is essential to point out that, although each individual phase has its own intrinsic elastic properties, their evaluation on polycrystalline NiTi wires is complicated due to the presence of internal interfaces introduced by the martensitic transformation. As both the interphase and martensitic interfaces are responsive to stress, two major problems appear [1]. First, it becomes experimentally difficult to evaluate the intrinsic elastic properties of phases as well as macroscopic elastic moduli by conventional methods. Second, since the motion of internal interfaces upon mechanical loading results in texture evolution, effective elastic properties of phases (e.g. Young's modulus) vary as well – i.e. they would be different e.g. in tension and compression.

In this work, thermomechanical tensile behaviour of a commercial Ti-50.8at% Ni alloy wire of 0.1 mm in diameter was investigated by means of a device which combines a conventional tensile device with a dynamic mechanical and thermal analyser (DMTA). This non-conventional tensile device permits to study storage modulus (E') as functions of tensile stress, strain and temperature history.

First, E' was measured during usual isothermal tensile tests over a wide testing temperature range between -100°C and 150°C . Second, E' was measured during thermal cycling under a constant tensile stress between 100 and 1000 MPa over wide testing temperature range between -150 and 250°C .

These tests are analysed to study dependences of phase transformations on stress and temperature and to bring new information concerning the value of the elastic moduli in the different stress-temperature regions.

Experimental Results

Figure 1a) shows the evolution of the storage modulus during a tensile test at 20°C . Characteristic points (0 to 8) are identified. Some of them permit to detect phase transformations during loading and unloading. Point (1) corresponds to the A-R transformation, (2) and (3) correspond respectively to the start and finish of deformation localisation plateau associated to the forward R-M transformation. Conversely, (5) and (7) correspond respectively to the start and finish of deformation localisation plateau associated to the reverse M-R transformation, and (8) corresponds to the R-A transformation.

Figure 1b) shows the evolution of the storage modulus during cooling and heating between 100°C and -100°C under a constant stress of 300 MPa. Characteristic points ($1'$ to $11'$) are identified. During cooling point ($3'$) corresponds to the A-R transformation and points ($5'$) and ($6'$) correspond respectively to the start and finish of the R-M transformation. During heating, point ($8'$) and ($9'$) are associated to the M-R transformation and point ($10'$) corresponds to the R-A transformation.

By combining these two types of tests at different temperatures and under different stresses, the phase transformation diagram can be established for both cooling and heating. Figure 2a) and 2b) show the evolution of E' with temperature and stress, during cooling and heating, respectively. By linear fitting to extrapolate to zero stress, it is possible to determine temperature of phase transformation without stress and compare with the results obtained from differential scanning calorimetry (DSC). With the DMTA and the DSC studies, the domains of different phases are fully defined and reliable; values of the storage modulus are determined for each phase.

Figure 2 presents this new information in a form of equal moduli areas plot on top of the stress-temperature diagram showing the phase transformation conditions. Due to the intrinsic hysteresis (history dependence) we show separate diagrams for forward (cooling) and reverse (heating) transformations for the sake of clarity. The most striking feature

is the valleys of low storage modulus E' stretching along the transformation lines caused by the nonlinear decrease of storage modulus prior phase transformations.

Conclusions

These DMTA studies permit to bring some information for the values of Young's moduli of the different phases in NiTi, which can be compared with the values obtained from other experimental methods [1]. It is found that for a same phase, E' evolves with temperature and stress ; E' is found to vary within 65-85 GPa for the austenite, 35-55 GPa for the martensite and 30-60 GPa for the R phase. This evolution shows that E' is influenced far from the transformation range.

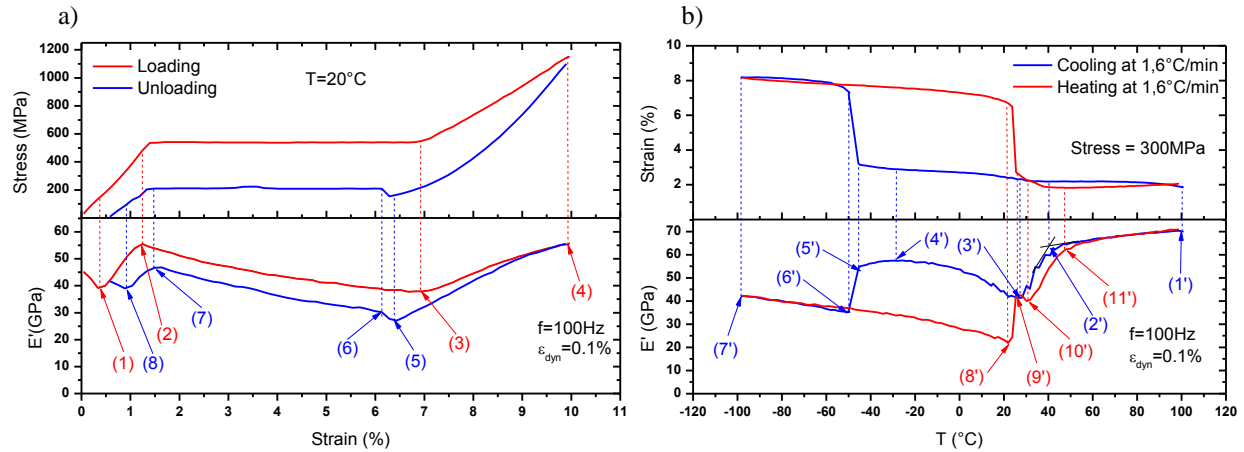


Figure 1: Dynamic mechanical testing analysis of the stress-induced transformation behaviour of NiTi. a) isothermal tensile stress-strain curve at 20°C (upper figure) and evolution of E' during the tensile deformation (lower figure); b) temperature-strain hysteresis loop under a static tensile stress of 300MPa (upper curve) and evolution of E' during the thermal cycle.

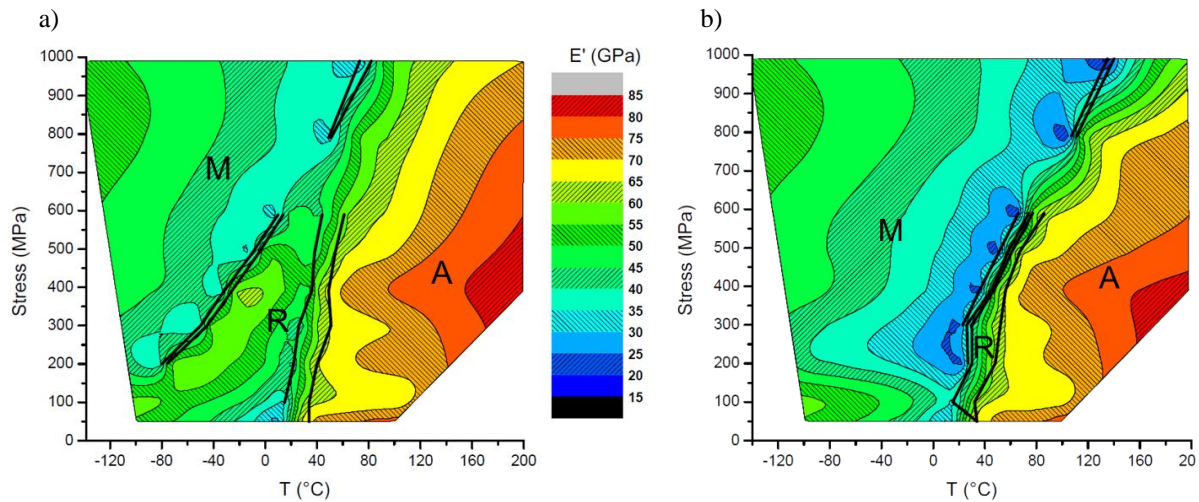


Figure 2: Storage modulus E' evaluated from DMTA experiments (Fig. 1) plot on top of the stress-temperature diagram showing the phase transformation conditions a) during forward b) reverse

References

- [1] Sittner, P. et al, "Young's modulus of austenite and martensite phases in superelastic NiTi wires", SMST 2013