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### How the evolution of the dynamic elastic modulus during isothermal tensile tests can bring new information on mechanisms deformation of a NiTi superelastic wire

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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 usual plasticity due to dislocation movements. These phenomena are difficult to identify by considering only stress-strain curves of tensile test. Classically the Dynamical Mechanical Analysis (DMA) is performed during a temperature sweep under a constant low stress and storage modulus E' is measured as function of temperature. In this presentation, superelastic deformation behaviour of NiTi thin wire was investigated by using a non-conventional DMA which allows to measure the storage modulus as function of strain during isothermal tensile tests at high level of stress. DMA tests were performed during superelastic tensile tests up to 10% of strain and up to failure at three temperatures above Af (the reverse martensitic transformation finish temperature). The goal is to identify and study elasticity, phases transformations, localisation, plasticity, by analysing storage modulus evolution during isothermal tensile tests. This model and the results of the three isothermal tests are used to determine the Clausius-Clapeyron coefficients for the forward A-M and reverse M-A transformations.

#### **Experimental Results**

Sress-strain curve (Fig. 1a) exhibits a classical superelastic behaviour. During loading, E' decreases monotonically between (1) and (2) with an initial value of 70 GPa. At point (2), matching with the start of the loading stress plateau, E' reaches 52 GPa. Between (2) and (3), E' continues to decrease almost linearly down to  $E'_3=35$  GPa. At the end of the plateau in (3), E' starts to increase monotonically to reach 52 GPa in (4) to become constant between (4) and (5). Fig. 3b shows the evolution of E' as function of stress.



Figure 1: DMA-SS test at  $T = 60^{\circ}C a$ ) stress-strain and storage modulus-, b) storage modulus-stress

#### Discussion

Identification of deformation phenomena present in NiTi wire from stress and storage modulus E' evolution Analysing both stress E' during a tensile test, allows to identify different zone (I to IV) and associated phenomena. E' evolution brings new information and clarifies the ambiguities we may have by analysing only stress as function of strain. Table 1 shows the evolution of stress and E' to identify elasticity, transformation, localisation and plasticity during loading. To simplify interpretation, arrows are used to code evolution.  $\Rightarrow, \heartsuit, \heartsuit$  mean that the value is constant, increases and decreases with increasing strain successively. For the NiTi wire studied, there is no stage with constant value of the storage modulus ; it means that there is no deformation stage for which the deformation is purely elastic or purely plastic, even for tests performed in the temperature range of superelastic behaviour.

σ(ε)	$E'(\varepsilon)$	Phenomena
$\nabla$	仓	Elasticity
⇒	$\hat{r}$	Plasticity
$\nabla$	Ŷ	Starting transformation
$\nabla$	\ \ \ \	End transformation
⇒	mostly linear	Localisation

Table 1: Identification of different phenomena from stress and storage modulus evolution during loading

#### Elastic moduli of austenite and martensite

Elastic moduli of austenite and martensite are usually measured by analysing the slope of the stress-strain curve. However the continuous decrease of the storage modulus E' between (1) and (2) shows the difficulty to measure elastic modulus of austenite from this initial slope. Estimating the elastic modulus from the initial stress-strain slope leads to underestimate elastic modulus of austenite. Elastic modulus of oriented martensite is often estimated from the slope of the stress-strain curve after the stress plateau The continuous increase of E' between (3) and (5) shows the impossibility to measure the elastic modulus of martensite by measuring this slope. A better approximation is obtained by considering the values of the storage moduli E' at high temperature and low stress for the austenite and at low temperature and very high stress for martensite.

Table 2: Elastic moduli of austenite and martensite determined by the slope of stress-strain curve and by DMA method

Phase	E by slope method	E from E'
Austenite	62 GPa	70 GPa
Martensite	25 GPa	60 GPa

#### Modelling of DMA results to determine Clausius-Clapeyron coefficients

A model is proposed for different zone and especially for zone (III+IV). First, zone (III+IV) at T=60°c was fitting by equation (1) (fitting parameter :  $K_3$ ) and was plotted in Fig. 1b. Then, fitting was extended to other temperature with equation (2) thanks to the Clausius Clapeyron coefficient  $C_{AM}$ . The coefficient  $C_{AM}$  is chosen to minimize the gap with experimental curves at 40°C and 80°C. Figure 2 shows the result with  $C_{AM}$ .=7.9 MPa.C<sup>-1</sup>.

$$E'(\sigma, T_0 = 60^{\circ}c) = E'_3 + (E'_M - E'_3) \tanh\left[K_3 \frac{\sigma - \sigma_3}{E'_M - E'_3}\right] (1)$$
  
$$E'(\sigma, T) = E'_3 + (E'_M - E'_3) \tanh\left[K_3 \frac{\sigma - \left[\sigma_3 + C_{AM} \left(T - T_0\right)\right]}{E'_M - E'_3}\right] (2)$$



Figure 2: storage modulus-stress at 40°C,60°C,80°C. Model with  $C_{AM}$  = 7.9 MPa.C<sup>-1</sup>

#### Conclusion

Evolution of E' during isothermal tensile test allows to :

- identify clearly different phenomena (elasticity, localisation, transformation, plasticity)
- determine elastic modulus of austenite and martensite without using the slope of the stress-strain curve which is influenced by transformation phenomenon
- determine Clausius Clapeyron coefficient of forward transformation without using stress plateau which can be difficult to identify if the transformation occurs without localisation.