

DMA characterization of Ni-Ti Shape-Memory Alloys (SMA)

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ABSTRACT

The novel performances found for Ni-Ti shape memory alloys (SMAs) strongly depend on their microstructure and their thermo-mechanical history.

The Dynamic Mechanical Analyzer (DMA) is an excellent tool to investigate existing correlations between temperature, stress and thermo-elastic transformation. It is possible to study the transitions in both stressed and unstressed conditions over a wide temperature range (typically from -150°C to 600°C) as well as the viscoelastic properties as a function of strain and frequency.

INTRODUCTION

During the last decade, smart materials systems have received increasing attention due to their great scientific and technological significance. Shape-memory materials (SMM)⁽¹⁾ are the major representatives of intelligent/smart composites because of their unusual properties, such as the shape-memory effect (SME), pseudo-elasticity or large recoverable strain with high damping capacity. In particular Ni-Ti system alloys have been intensively investigated and nowadays are the most important commercial SMAs because of their exclusive shape-memory performance, good processability and excellent mechanical properties. The Ni-Ti alloys can be readily fabricated into various forms or sizes and they exhibit some exciting application potentials in microelectro-mechanical systems⁽²⁾, medical implants, intelligent materials and structural systems⁽³⁾. These materials undergo thermally induced structural transformation (martensitic)⁽⁴⁾ on cooling be-

yond a critical temperature as well as stress induced transformation when the appropriate stress is applied at a particular temperature. All these thermo-mechanical transformations⁽³⁾ and their influence on mechanical properties and specimen shapes are dependent on alloy composition, processing procedures and thermo-mechanical treatments. The purpose of using DMA to characterize these materials is to establish correlations between material structure and properties.

EXPERIMENTAL

The test samples were thin fibers (70 or 80 μm in diameter) and thin bars (17 x 5 x 0.45 mm). Depending of the test procedure and specimen shape, either film/fiber or single cantilever clamps were used.

The fiber samples were used to perform strain recovery tests (figure 2) and stress/strain experiments (figure 4 and figure 5). During the strain recovery test, the temperature was ramped at $5^{\circ}\text{C}/$

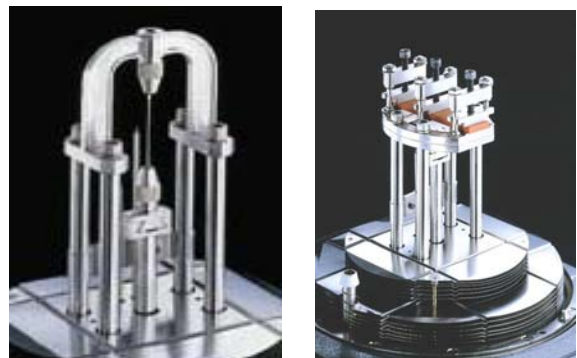


Figure 1: Tensile fibers and single cantilever clamps of the TA Instruments DMA Q800.

min between $-30\text{ }^{\circ}\text{C}$ and $150\text{ }^{\circ}\text{C}$ whereas in the stress/strain experiments the temperature was kept constant. The strain rate used in stress/strain experiments was $0.5\text{ } \%/ \text{min}$.

Thin samples bars were tested in single cantilever bending (figure 3). The applied deformation amplitude was $2.25\text{ }\mu\text{m}$ at a frequency of 1 Hz and a heating rate of $2\text{ }^{\circ}\text{C}/\text{min}$ between $-100\text{ }^{\circ}\text{C}$ and $100\text{ }^{\circ}\text{C}$.

As usual, all the clamps have been calibrated to account for compliance.

RESULTS AND DISCUSSION

A key experiment to evaluate the memory-shape effect is the temperature cycling test. With a constant stress of 100 MPa applied, the fiber is heated and cooled at $5\text{ }^{\circ}\text{C}/\text{min}$ and the dimension changes (i.e. strain) are measured. As shown in figure 2, the fiber contracts during heating followed by an increase of the stiffness. The opposite happens during cooling. The amplitudes of these periodic changes define the properties of these particular SMA.

An important property of SMA materials is the damping capacity and the ease to change it by simple thermal treatments. In figure 3 the dissipation factor, $\tan\delta$ (loss tangent) is shown for a temperature ramp test at a constant frequency of 1 Hz and a strain amplitude of $0.001\text{ } \%$ (ca. $2.25\text{ }\mu\text{m}$). The plain ternary Ni-Ti alloy (with Cu) (curve 3) shows a very strong damping peak around room temperature. After hydrogen doping (curves 1 & 2, different hy-

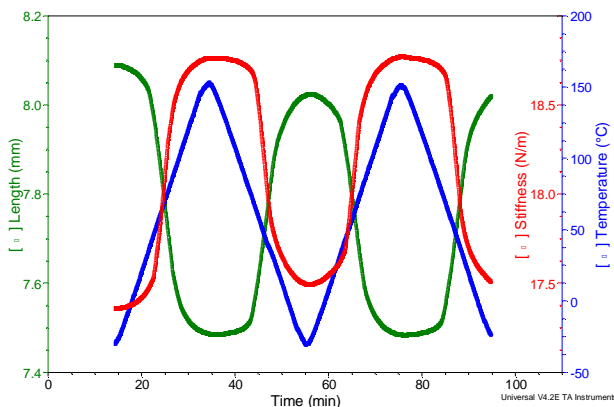


Figure 2: Strain recovery cycles under constant load versus temperature; Temperature rate in $\% \text{ }^{\circ}\text{C}/\text{min}$; Fiber diameter= $70\text{ }\mu\text{m}$; Stress applied 100 MPa

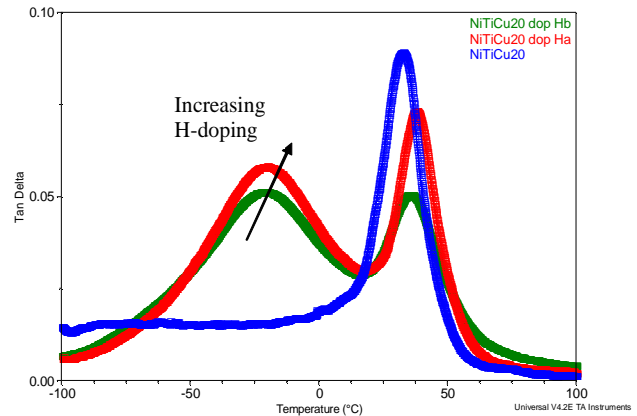


Figure 3: $\tan\delta$ behavior for Ni-Ti bar samples with different level of hydrogen-doping. Temperature ramp tests at a ramp rate of $2\text{ }^{\circ}\text{C}/\text{min}$, a frequency of 1 Hz and a strain of $0.001\text{ } \%$ ($2.25\text{ }\mu\text{m}$ amplitude) using single cantilever clamps.

drogen-doping treatments) an additional, very strong damping region below $0\text{ }^{\circ}\text{C}$ appears. The DMA is a very sensitive analytical technique that gives easy access to the material's damping behavior. A more complete characterization involves also testing at different frequencies to study the frequency dependence of the damping properties.

A typical experimental procedure to characterize SMA materials, used for electronic devices and medical applications is the stress-strain test. The diameter of the fibers used in SMA devices is very small (from $100\text{ }\mu\text{m}$ down to $10\text{-}15\text{ }\mu\text{m}$). Due to the air-bearing motor (minimum load = 10^{-5} N) and the optical encoder, able to measure the strain with a sensitivity of 1 nm , the Q800 is capable to control very small strains and loads as shown in figure 4 and 5.

The SMA alloy when deformed, yields after an initial elastic deformation (1) with the stress remaining approximately constant. The strain in the plateau region (2) is not slip strain, but is due to a kind of twinning. If the stress is unloaded (3) the elastic portion of the strain recovers, the shape of the strain recovery curve being dependent on the composition and thermo-mechanical treatment. The fibers with different chemical composition (different amount of Cu in the ternary alloy) shown in figure 4, exhibit different stress-strain behavior with different stress relaxation curves while the strain is decreasing.

The thermo-mechanical properties of these sensible materials are strongly depending on the temperature. In figure 5, the stress-strain behavior as a function of temperature is shown. Increasing the temperature of the SMA fibers (80 μ m in diameter), while applying a strain rate of 0.5 %/min up to 4% and reverse (back to 0 stress) shows distinct differences in the mechanical response.

SUMMARY

DMA is a very suitable tool to characterize metals and alloys in terms of their thermo-mechanical behavior. In particular, shape-memory alloys represent a very interesting category of new “smart” materials for different applications like electronic sensors, medical sutures and devices. Under both, static as well as dynamic loading, the DMA technique allows to characterize all the aspects of SMA materials over a wide temperature range (from – 150 °C up to 600°C) and with a very high strain and stress sensitivity. By using different sample holders (i.e. film/fibers, compression, bending or cantilever) all types of specimen shapes (fibers, thin films, and bars) can be analyzed in respect of their particular application field.

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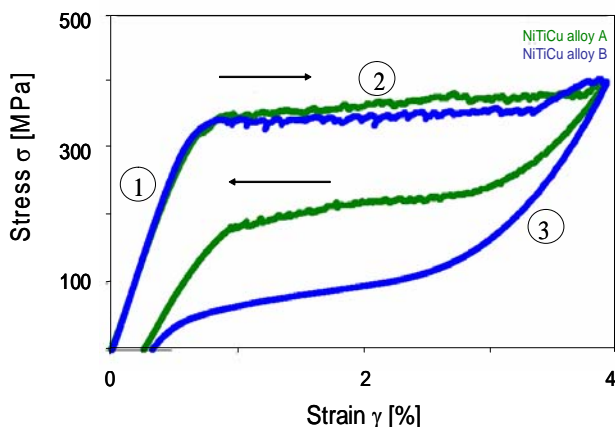


Figure 4: Stress-strain behavior for two Ni-Ti alloys with different amount of Cu. SMA fibers (80 μ m in diameter) were subjected to strain rates of 0.5 %/min up to 4 % and reverse, using fibers clamps at a temperature of 72 °C.

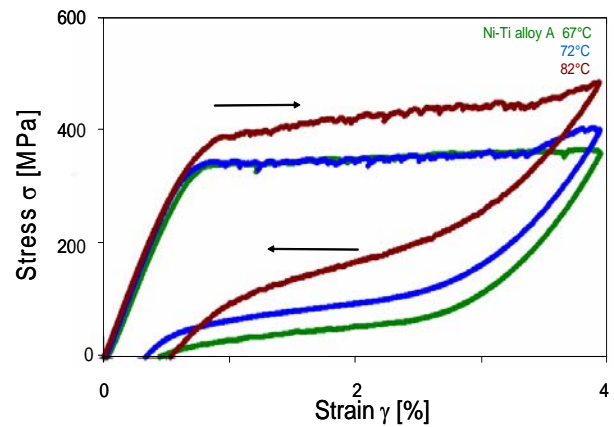


Figure 5: Stress-strain behavior for Ni-Ti alloys as a function of temperature.

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