During the 12 shifts of the MA-358 experiments two types of measurement were performed:

- Microstructure changes in deformed NiTi shape-set structures
- Kinetics of annealing processes occurring during heat treatments of thin wires NiTi

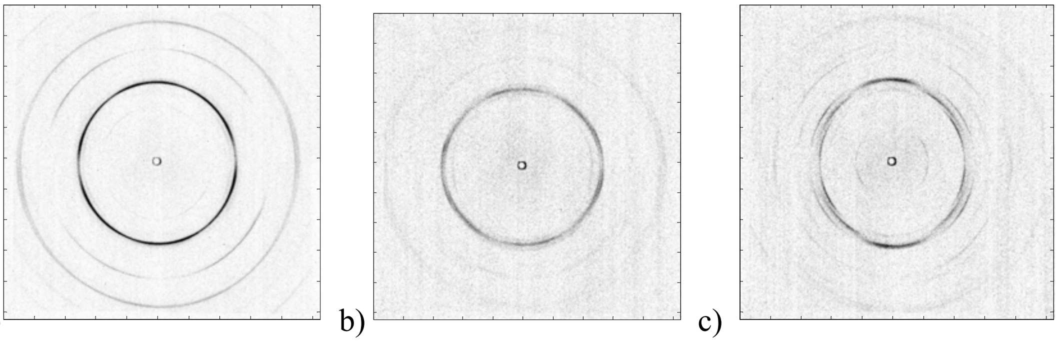
I/ Microstructure changes in deformed NiTi shape-set structures

I.1/ BACKGROUND AND MOTIVATION

The goal was the possibility to scan across the wire (i.e. to record diffraction patterns from different 10µm cross-paths through the wire) at a few places along the snake-shaped spring as thin as 150µm (with a different curvature). The patterns were recorded for several strain states.

I.2/ RESULTS

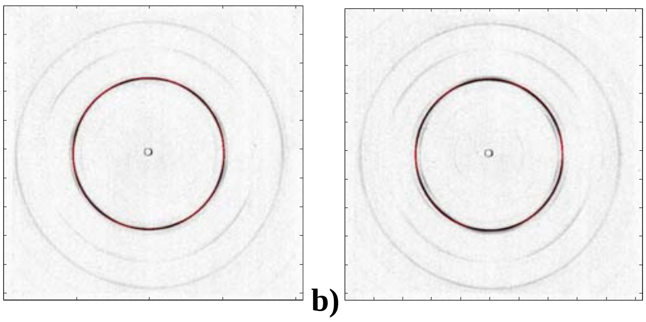
On Figure 1 typical patterns of austenite (a) and martensite (b,c) are shown.



a) b) c)

Figure 1: *Diffraction pattern recorded from shape set spring under tensile load in the center (a), on the outer surface of a bend (b) and in the inner surface of a bend (c).*

The preferred variants (and thus texture) of the martensite phase depend on the nature of the uniaxial stress applied, i.e. if it's compressive (b) or tensile (c). Figure 2 shows the austenite reflection on the vicinity of martensitic transformation.



a) **b)**

Figure 2: *Figure of austenite diffraction on the vicinity of martensitic transformation in compression (a) and tension (b). The austenitic reflection 110 Debye circle is contoured with red circle.*

The shape of Debye circle is rather elliptic due to the elastic strain present during the deformation of the spring. The horizontal and vertical axes diameters are 1030 and 1048 pixels respectively for austenite in pressure (with respect to the wire axis) and 1006 and 1060 pixels for austenite in tension; this corresponds with about −0.7% compressive and +1.7% tensile deformations. Vertical direction more or less corresponds with the wire axis.

The apparent difference between patterns on Figure 1 allows for a simple human-driven recognition of a part of the spring obeying tensile or compressive deformation and, yet more simply, a qualitative description of a prevailing type of phase composition (austenite / pressured martensite / tensile martensite). Figure 3 shows three such maps of prevailing phases in a two-dimensional scan (across/along a single bend of spring).

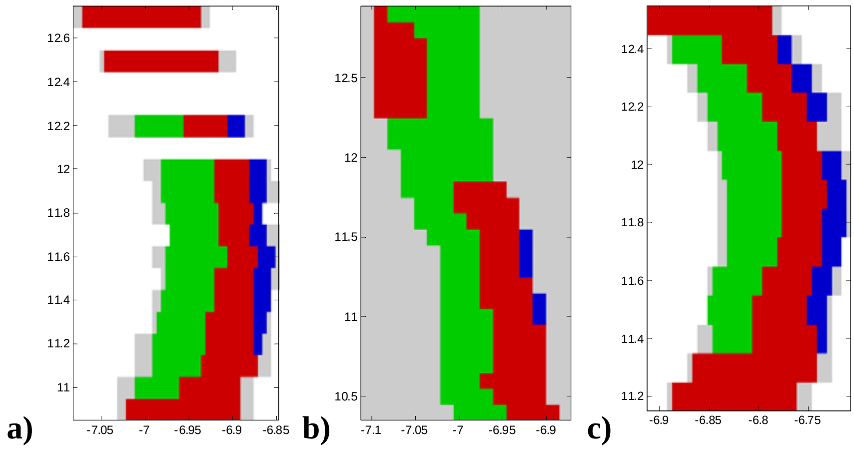


Figure 3: *Two-dimensional map of phase composition of shape-set structure under deformation. Not measured area is white, measured with no signal is gray, prevailing austenite signal is red, tensile martensite green and compressive martensite is blue. Axes units are millimeters.*

The three pictures correspond to spring elastically deformed with a force of ~0MPa (a), 590MPa (b) and '~0MPa unloading' (c). The 'zero stress' states refer to only bending deformation of the spring with practically zero mean tension in the wire. Obviously the volume of compressed martensite is smaller and almost vanishes under strong deformation, when the spring is almost straightened (Figure 3b).

I.3/ CONCLUSION

These measurements show the feasibility of measurements with step of 10µm from the bulk volume along the snake-shaped spring of the NiTi wire as thin as 150µm with different curvatures. Since now the analyses allow for a simple human-driven recognition of a part of the spring obeying tensile or compressive deformation and, yet more simply, a qualitative description of a prevailing type of phase composition (austenite / pressured martensite / tensile martensite). These analyses aren't yet finished and we will publish the new results soon.

II/ Kinetics of annealing processes occurring during heat treatments of thin wires NiTi

II.1/ BACKGROUND AND MOTIVATION

Thin wires made of NiTi shape memory alloy exhibit excellent functional properties (e.g. pseudoelastic strains up to 6% above 100ºC, generation of stresses ~800MPa upon heating, strength up to 2GPa, etc.) due to their fine grained microstructure (less than 1µm).

If the shape of the NiTi wire is mechanically constrained during the heat treatment, it acquires that shape in the parent austenite phase. Heat treatment under mechanical constraint is called "shape setting" if the wire is contained in a fixture or "straight annealing". The functional properties of the NiTi wire can be adjusted through a combination of : i) amount of the cold work performed in the last wire drawing step, ii) temperature of the heat treatment and iii) time of heat treatment.

With the ultimate aim to develop suitable manufacturing routes for NiTi hybrid textiles, we have been working on the optimization of the heat treatment procedures applied to NiTi wires using conventional furnace heating and Joule heating. Although there is only a very limited knowledge of the physical processes taking place during the heat treatments (internal stress relief, reverse martensitic transformation, annealing out of point defects, dislocations, precipitation, recrystalisation etc.).

II.2/ RESULTS

Tests on shape setting with Joule heating have been done during the experiment MA359 on the beamtime ID11 (figure 4). We used a wire in a vertical position and the Frelon Camera with the frame and binning mode to obtain time resolution as fine as 10ms.

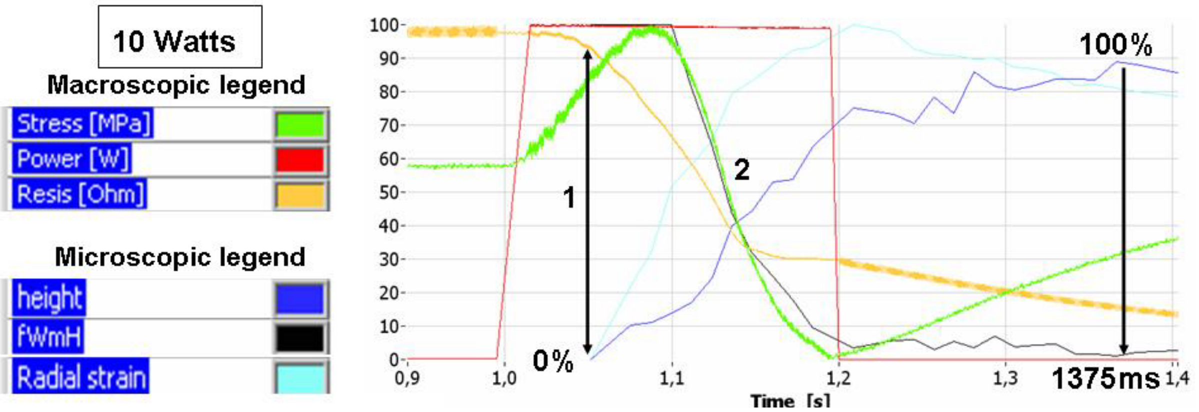


Figure 4: *Preliminary results at 10Watts with a 100µm NiTi wire in vertical position*

As we can see in figure 1 there is quite good correspondence between macroscopic behavior and structure sensitive information: **1/** In-situ heating experiment: At 10Watts the height of the austenitic peak increases after 50ms and reach 100% in 375ms. **2/** There is a significant parallel between decrease of the macroscopic stress and the Full Width at Half Maximum (FWHM) of the austenitic peak.

II.3/ CONCLUSION

These tests show the feasibility of measurements on the recovery processes from the bulk volume of the NiTi wire as thin as 100µm under applied stress up to 400MPa, at high temperature up to 800ºC with time resolved of 10ms. More diffraction information is however needed for reliable interpretation in terms of the activity of various physical processes during the heat treatment.The plan now is to make in-situ diffraction experiments on various as-drawn NiTi in the vertical position and in the horizontal position to know about the axial and radial strain (new proposal 18874).