ESRF	**Experiment title:** Low temperature shape setting of NiTi filaments for smart textiles	**Experiment number**: **MA-1309**
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Report:

Thin filaments of NiTi shape memory alloy are currently used to manufacture smart hybrid NiTi textiles which transform their functional properties to 2D forms. Before their textile processing, cold worked NiTi filaments have to be heat treated to display functional properties as superelasticity or shape memory effects. Conventional heat treatment, which consists of exposing the NiTi wire to temperature as high as 500ºC in electrical furnace for a relatively long time, is not applicable to NiTi hybrid textiles because of the presence of polymeric yarns which would be damaged at temperatures higher than 200^{o}C. This point has been a crucial problem for further development of hybrid woven or knitted NiTi textiles.

The experiment MA1309 was performed as a part of wider research prograam aimed at developing heat treatment methods applicable at temperatures lower than 200 ^{o}C. The method basically consists in selecting an optimized microstructure of the treated wire, transforming its microstructure into an oriented martensite state and exposing it to heat treatment at relatively low temperatures (below 300^{o}C). The goal of this in-situ X-ray diffraction experiment was to find out what happens with the martensitic microstructure during the exposure of the wire to low temperature cycles.

A purposely made deformation rig capable of simultanous application of controlled load and direct electric current heating of thin superelastic NiTi filaments with precisely controlled microstructures [1-3] (8 samples, 2 geometrical orientation of the rig with respect to the X-ray beam) was installed on the ID31 high resolution powder diffractometer (Fig. 1).

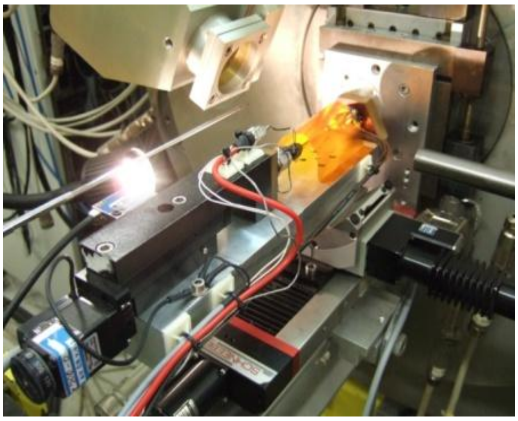

Fig. 1: Tensile rig installed on ID 31

The wires (d = 0.1 mm, l = 10 mm) were stretched (7%) beyond the superelastic plateau (Fig 2a) and subsequently heated by electric current in a step wise manner. The force and electric resistance of the wire evolved upon heating at constant length as shown in figure 2c (single continuous heating/cooling cycle). X-ray diffraction patterns were periodically recorded prior the heat pulse and after the heat pulse and unloading. Diffraction patterns were, then, analysed for peak positions, peak widths and integrated intensities using single peak fitting.

The analysis has shown that the diffraction pattern corresponding to stress induced monoclinic B19' martensite prior the heat pulse (Fig.2) changes into the pattern of cubic B2 austenite (Fig. 2d) in the temperature range 100 - 250°C denoted as LTSS in figure 2c. The austenite peaks in the diffraction pattern recorded after treatment are broader and shifted from their original positions suggesting the presence of internal stresses in the microstructure of the treated wire.

It comes out that, following such low temperature heat treatment LTSS (T < 250 °C) , the NiTi wire is austenitic, shape set (4% longer), exhibits excellent superelasticity (Fig. 2b) – i.e. exactly what was required for hybrid NiTi textiles. It is thus confirmed by the in-situ X-ray experiment that stress induced martensite in the stretched NiTi wire with properly adjusted austenitic microstructure can be converted to plastically deformed but still perfectly superelastic austenite by the heat pulse generated by electric current. This conversion is called low temperature shape setting – LTSS. The mechanism involved utilizes the chemical energy stored in the stress induced martensite raised by the supplied heat to form/process the wire The results of this research were presented in a keynote talk of P. Sittner at SMST 2011 conference [4] and, at least, one journal publication is in preparation.

References

1. B. Malard, J. Pilch, P. Sittner, R. Delville, C. Curfs, Acta Materialia, 59, 2011, 1542–1556
2. B. Malard, J. Pilch, P. Šittner, R. Delville, C. Curfs, Sol. State Phenom. 172-174 (2011) 1243 - 1248.
3. Delville, B. Malard, J. Pilch, P. Šittner, D. Schryvers, Sol. State Phenom. 172-174 (2011) 682 - 687.
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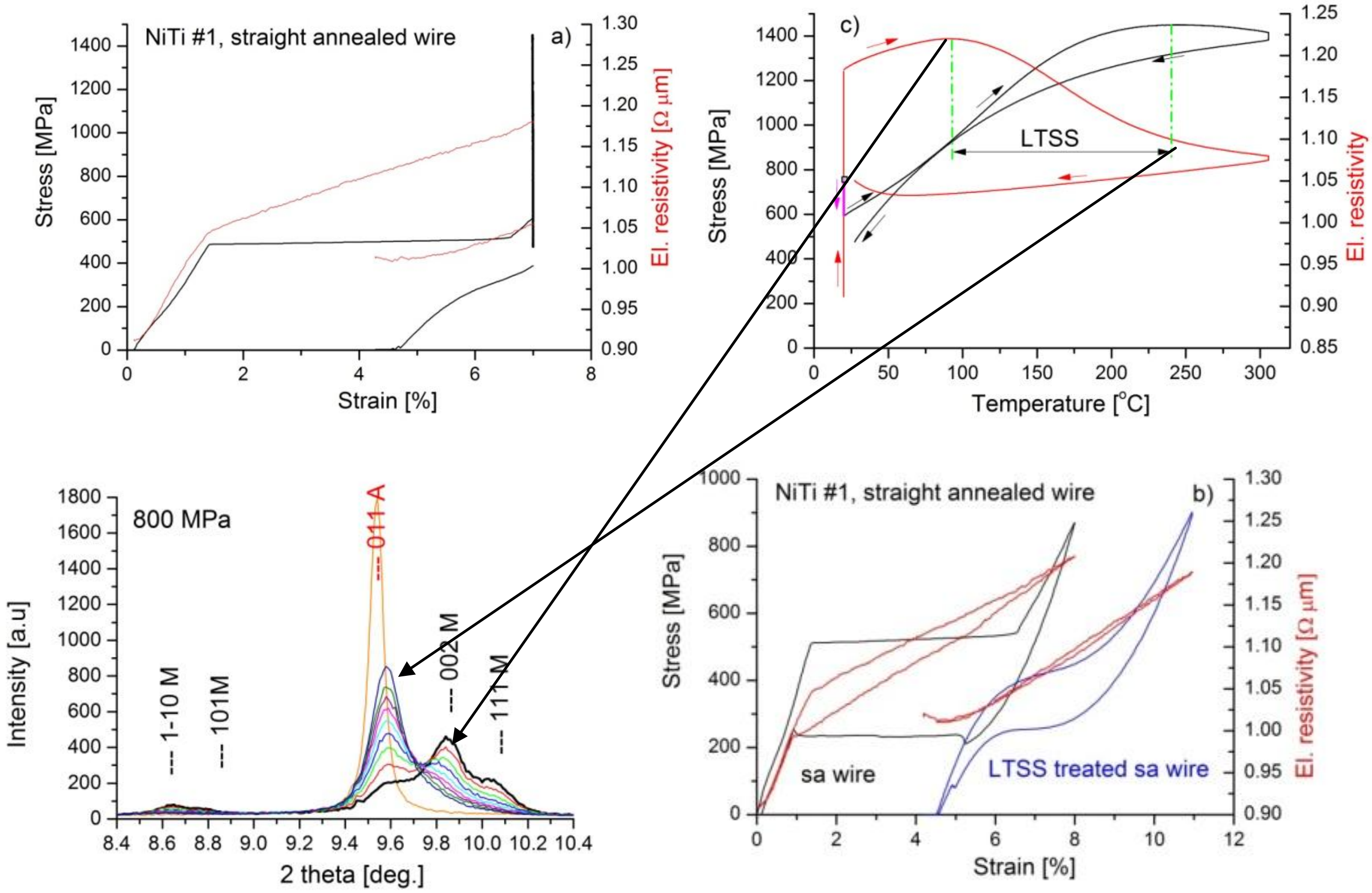

Fig. 2: Example of in-situ X-ray diffraction experiment during thermomechanical test on superelastic NiTi wire. The wire (d = 0.1 mm, l = 10 mm) was stretched beyond the superelastic plateau (a) and heated by electric current in a step wise manner while diffraction pattern were recorded prior the heat pulses with subsequently increasing power (maximal achived temperature increased stepwise).