Microstructural effects of strain aging on NiTi pseudoelastic wires by synchrotron X-ray micro-diffraction

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Abstract: Consequences of strain aging pseudoelastic NiTi wires (2.46 mm diameter) at moderate temperatures (near 100ºC)?

Changes of stress-strain on aged, compared with non-aged wires, and small changes in thermal transformation, suggest existence of relevant strain fields. The fact that thinner wires perform differently on aging suggests radial dependence of strain fields. Structural measurements at CELLS-ALBA synchrotron, BL04 Materials Science Powder Diffraction beam-line. X-ray micro-diffraction for near 10 (μm)[2] (FWHM) zones on cross-sections of samples done, aiming to detect residual strains as function of radial position on the wire.

Results show very small changes of diffraction peaks position, with more relevant variations near the surface, as well as increase of line-width of peaks near surface. This might be interpreted as larger distribution of residual stresses at surface of wire, showing a high sensitivity of the transformation on residual stresses enabling defined paths of transformation

Introduction: Pseudoelastic NiTi wires with the same composition and transformation temperature but different diameter have been shown to perform differently. We look here for crystal structure differences and stress (strain) distribution in different diameter wires.

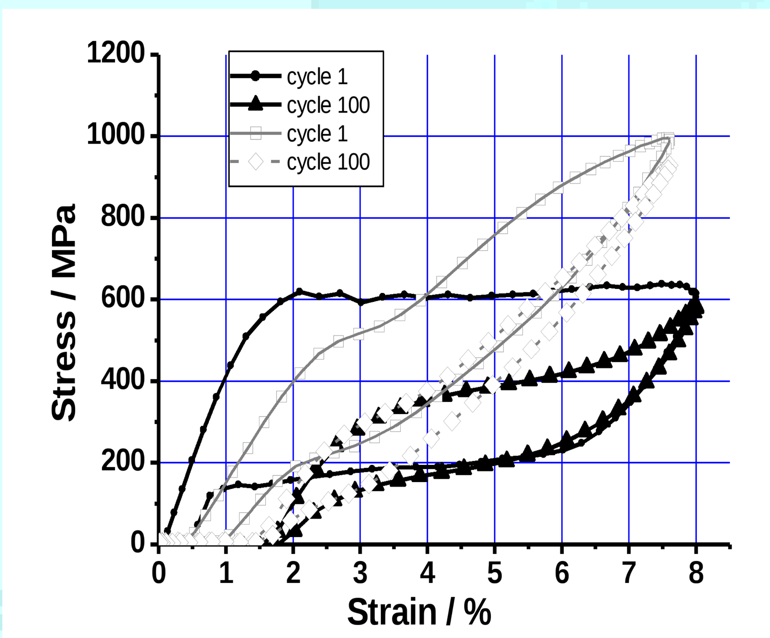
Experimental: Pseudo-elastic NiTi from Memry (CT, USA), a division of SAES Getters (Italy), previously form Special Metals Corp. (New Hartford, New York, USA). Straight-annealed wires 2.46 mm and 0.5 mm diameter, Ni-rich (55.95 wt % Ni, balance Ti). As by DSC was 237 K. Wires in light oxide surface state as furnished.

MTS 810 testing machine, in air-conditioned room. Speed of cycling: 100 s per cycle for the 2.46 mm diameter wire, and 20 s for the 0.5 mm diameter wire, to achieve similar conditions concerning heat transfer characteristic times to the ambient. Strain aging done with help of specifically designed devices.

Synchrotron radiation from ALBA was used to perform micro-diffraction experiments at 25ºC (Cerdanyola del Valles, Spain; beam-line BL04, 2013110801 experiment on MSPD@ALBA). Wavelength was 0.4246 A, the beam was of near 10 μm2 (FWHM), and a CCD detector of 2048 sections was used, each element ("pixel") of the detector was of 79 by 79 μm. Detector was situated at 260.5 mm of the samples with angular resolution between measured points corresponding to around 0.01º in two-theta. 4 rings (peaks) registered, corresponding to beta phase NiTi. All the samples produced only the cubic beta phase peaks. The raw data was processed with the software FIT2D, to obtain the diffraction along a given direction, with a +/-5º integrating margin in angle along the "cake" of data.

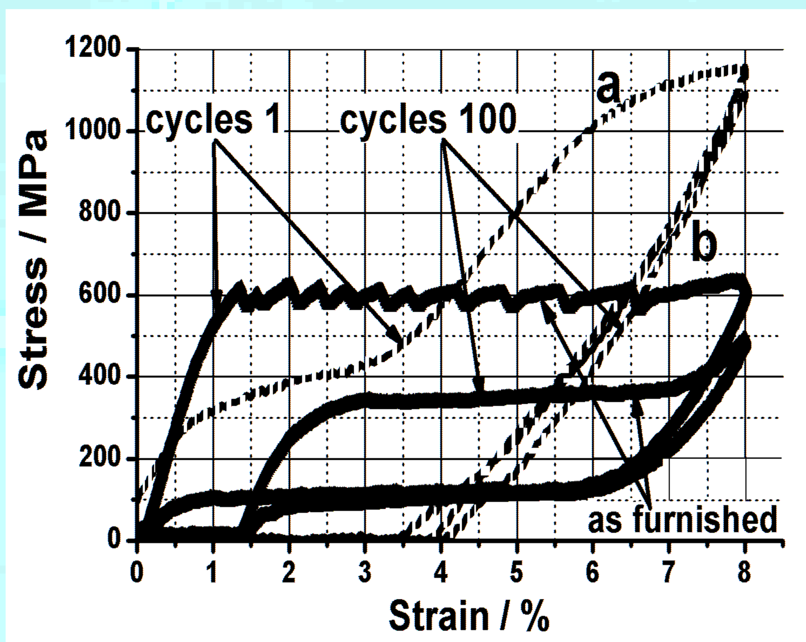
Mechanical Results:

Mechanical: 2.46 mm diameter



Full symbols; as furnished wire. Open symbols: aged at 7.8% strain, 100ºC 1 month

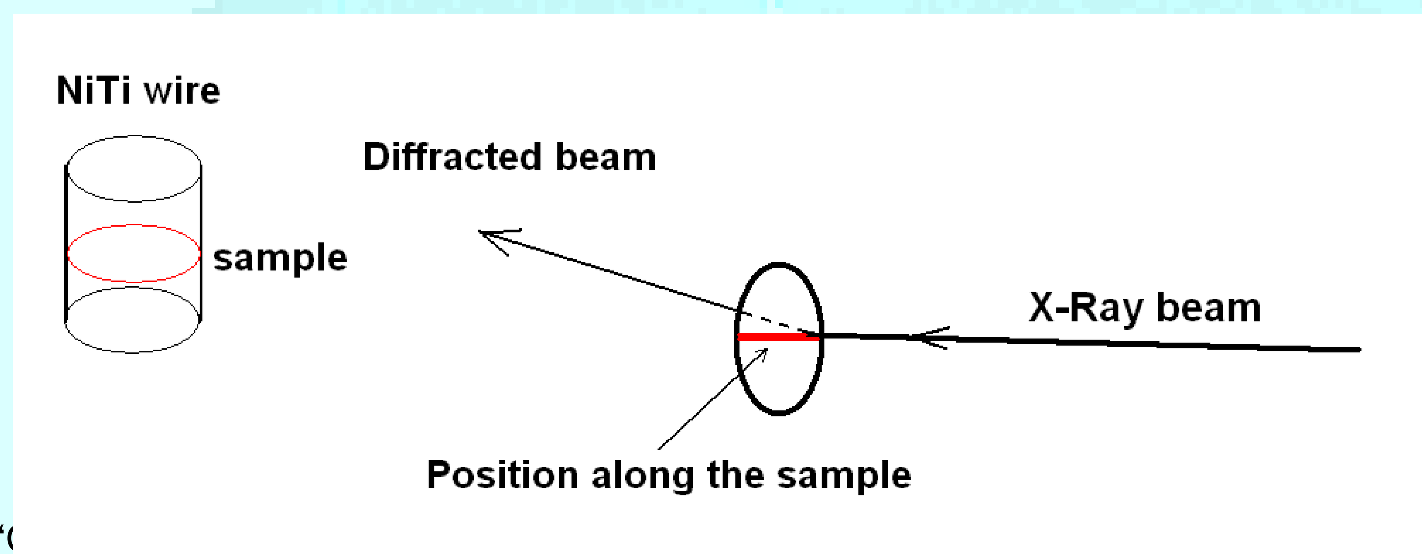
0.5 mm diameter

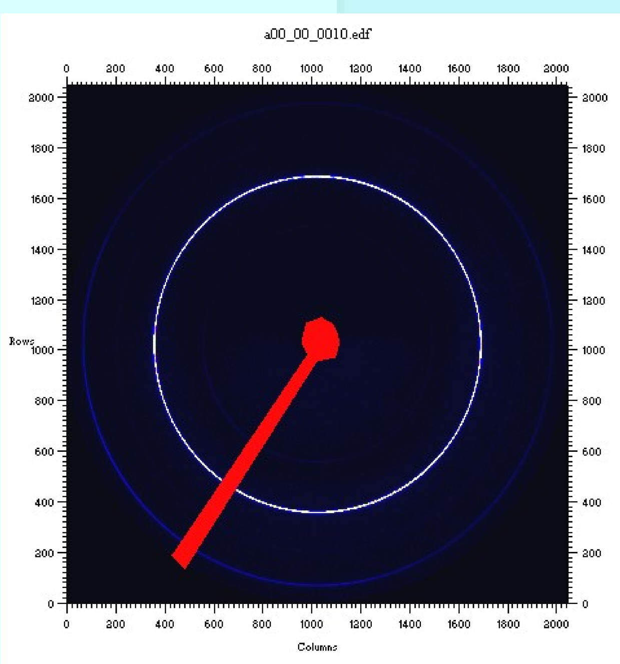


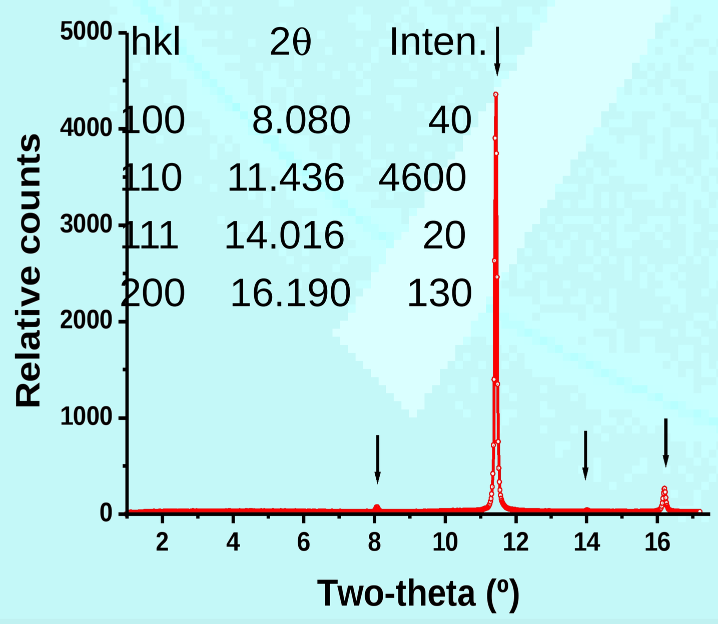
Thick line: as furnished. Dashed: aged at 100ºC

Diffraction Results:

Geometry:





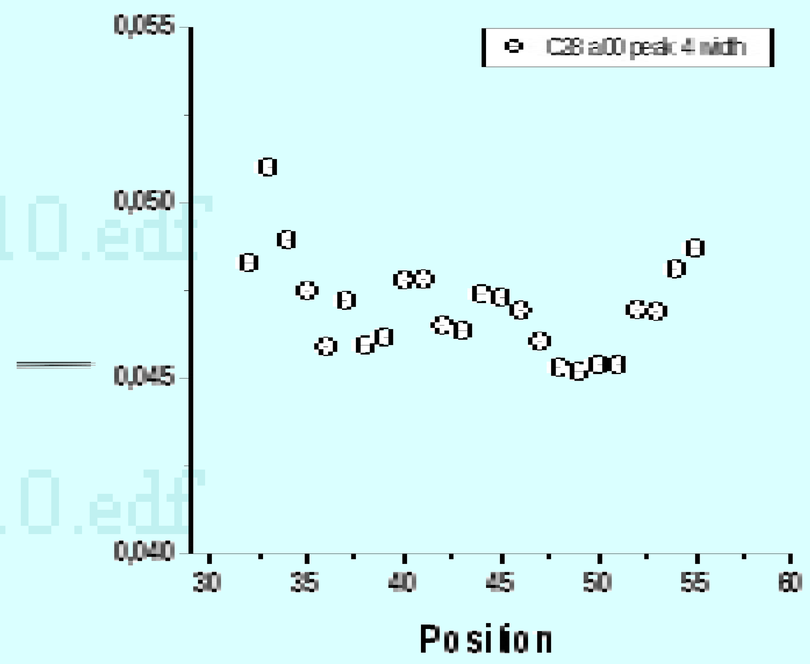


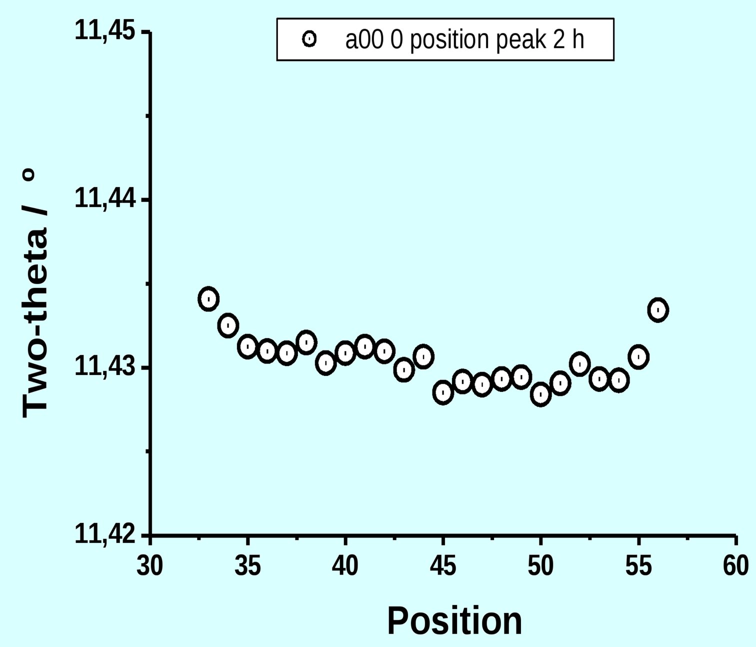
Low number of peaks, texture, resolution, statistics, prevented using full profile fit to strained samples.

Then we fit individual peaks to Pseudo-Voight functions.

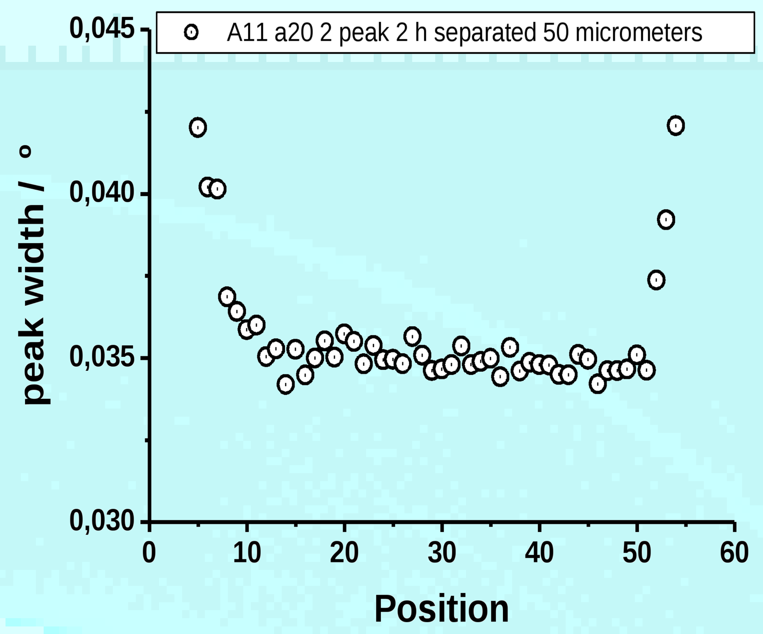
Results:

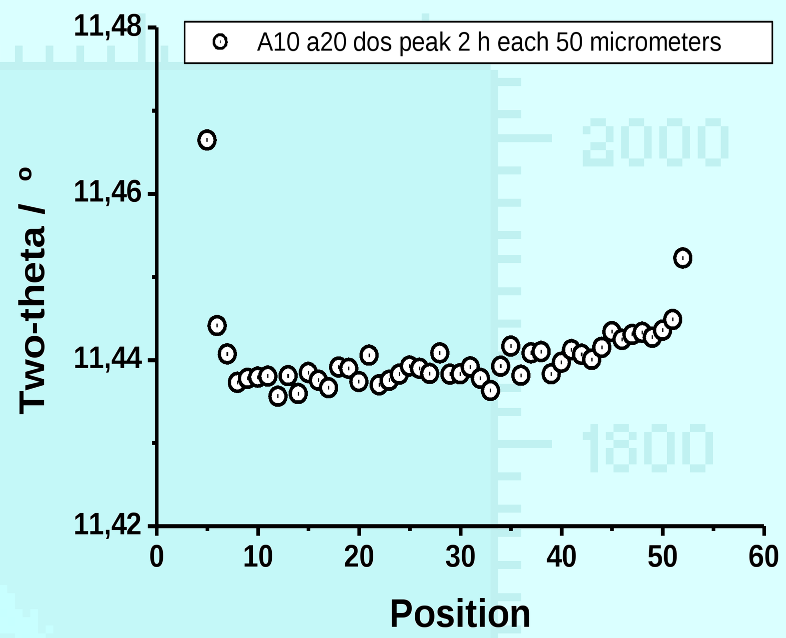
2.46 mm diameter wire (Position in 0.1 mm units, arbitrary origin, as furnished wire, peak 110):





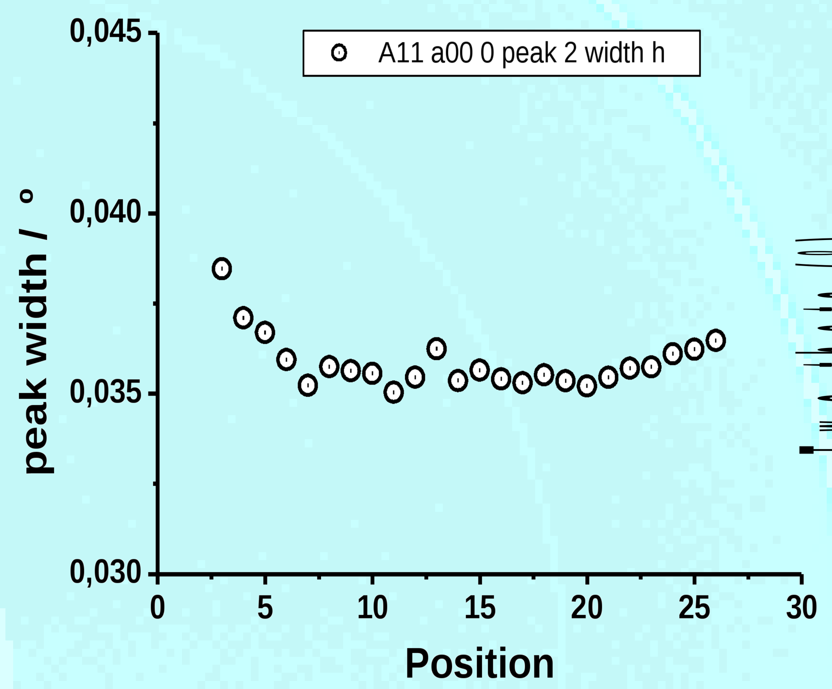
2.46 mm diameter wire (Position in 0.05 mm units, arbitrary origin), aged 1month at 7.8% strain, 100ºC :

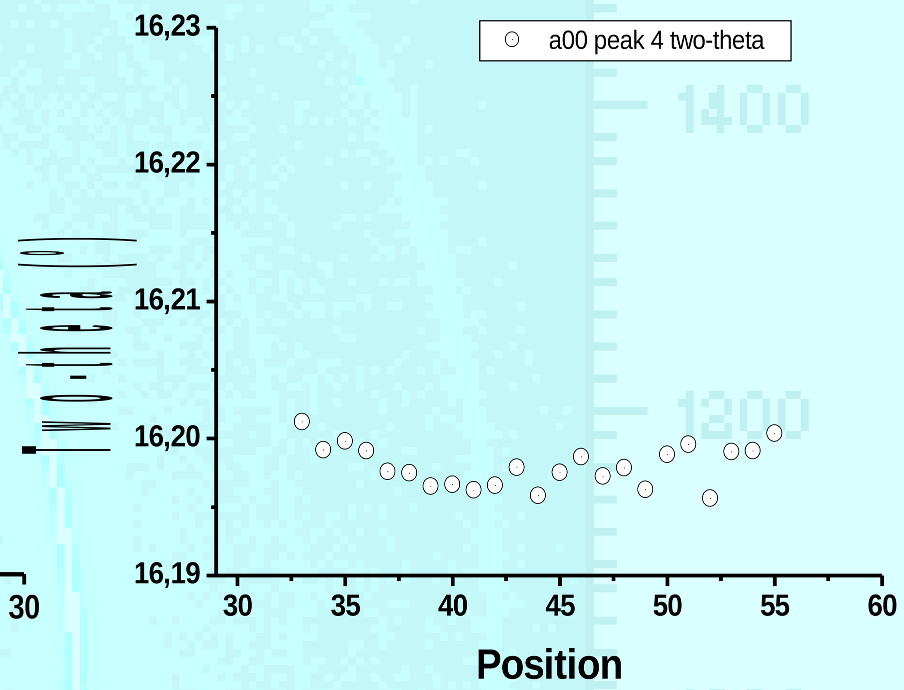


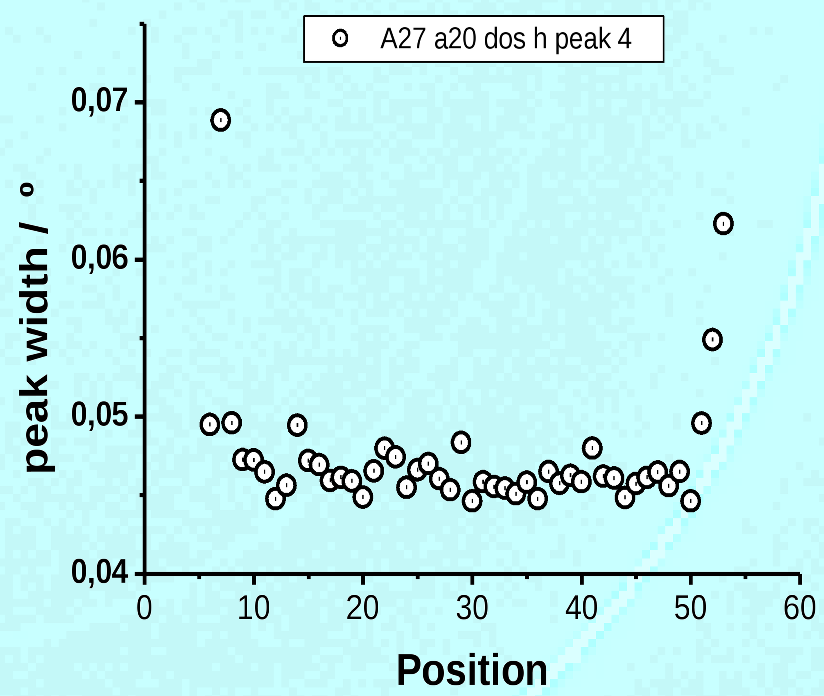


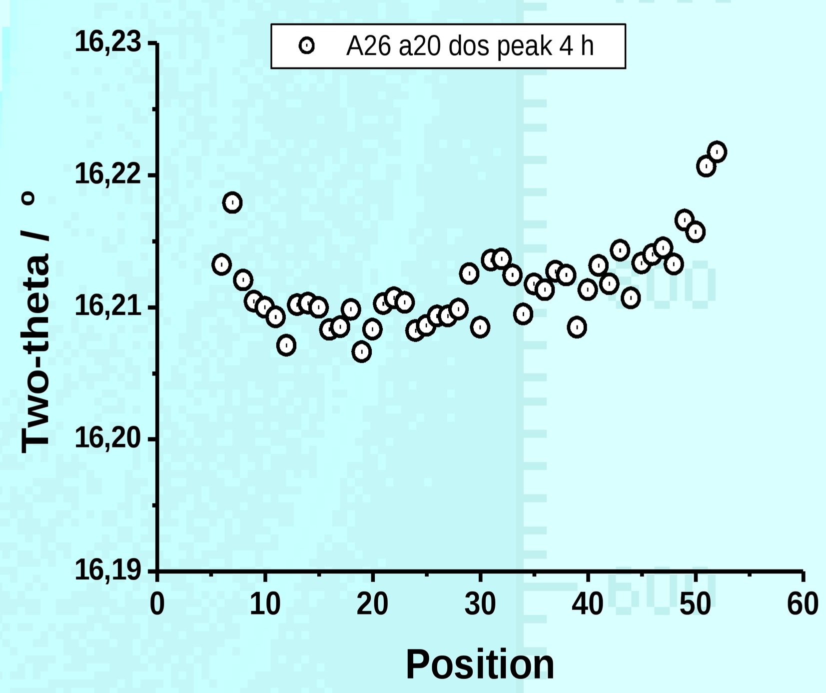
All peaks resulted similarly, values at the end of the cross-section (near surface of the wire) have an increase in width. There is also a slight increase in two-theta after strain aging.

Results for peak (200) of 2.46 mm diameter wire, first row as furnished wire, bottom aged wire (left: Width of peaks; right: Two-theta of peaks (as function of Position in the cross section of the wire, step 0.1 mm, arbitrary origin):









Experiments were done with the as furnished 0.5 mm dia. wire, with position steps of 50 μm. Results showed also a slight increase of line width and two theta angles near the lateral surface of the sample.

Then, it can be concluded that a "surface" effect exist on the microstructure of the wires (due to drawing, thermomechanical treatments, and surface oxidation on the wire). The effect is enhanced by strain aging, so it explains different mechanical behaviour (after strain aging) of wires with different diameters.

Conclusions: Microstructural effects of strain aging at 100ºC, on NiTi pseudoelastic wires, have been looked for by micro-diffraction performed at ALBA-CELLS synchrotron radiation utility. The results of the measurements show that, on strain aged sample, the width of the diffraction peaks increase appreciably near the surface of the wires. This effect might be understood as the result of the high stresses and some plastic deformation during the strain aging, leaving a wider distribution of residual stresses near the surface of the wires. This wider stress distribution near the surfaces exists after the strain aging because the asymmetry of the surface, the existence of imperfections (mechanical defects) and oxide on the surface, which act as a source of non-symmetric stresses on transforming.

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