ESRF	**Experiment title:** Fatigue damage at ultrasonic speed in NiTiNOL for medical applications – Are superelastic phase tranformations fast enough?	**Experiment number**: MA3628
Beamline: ID15	**Date of experiment**: from: 17. 2. 2018 to: 20. 2. 2018	**Date of report**: 27. 02. 2018
Shifts:	**Local contact(s)**: Stephano Checchia	*Received at ESRF:*

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Report:

The experiment aimed at the in-situ observation of phase transformation in the shape-memory alloy NiTiNOL and the elucidation of the respective speed during ultrasonic fatigue cycling, using high energy X-ray diffraction.

A self constructed tensile stage including an ultrasonic fatigue (USF) testing loadtrain was mounted onto the sample stage of ID15 (Fig 1a). 250 µm thick dog-bone shaped specimen were mounted into the tensile device and low and high frequency loading cycles were applied with 0.02-0.2 Hz or 18.3 kHz, respectively. The X-ray measurements were carried out with a 130 x 170 µm beam and a 100 x 100 µm beam (in order to average enough of the grains in the sample) at 60 keV X-ray energy and the distance to the Dectris Cd Te Pilatus 2M detector was chosen in a way that q-space up to 5 nm^{-1} was recorded.

Data acquisition of the detector was externaly enabled. A gating signal from the PC also controlling the ultrasound device allowed to resolve elastic and plastic strains, along with phase identification during one full ultrasonic cycle (loading – unlaoding) at 50 discrete phase positions, 1.09 µs wide each. Every 9174 individual images were added to gain a total exposure time of 10 ms to obtain a high quality diffraction pattern at each phase step.

The USF tests were complemented by low frequency cycles to gain reference data for the comparison with conventional fatigue (CF) tests. USF and CF were conducted at different amplitudes superimposed on static mean loads. At certain static loading levels the center of the specimen is transformed from Austenite (A) to Martensite (M) with a intermediate phase R involved. Thus a strained sample shows two transition zones (TZ) which were investigated in detail.

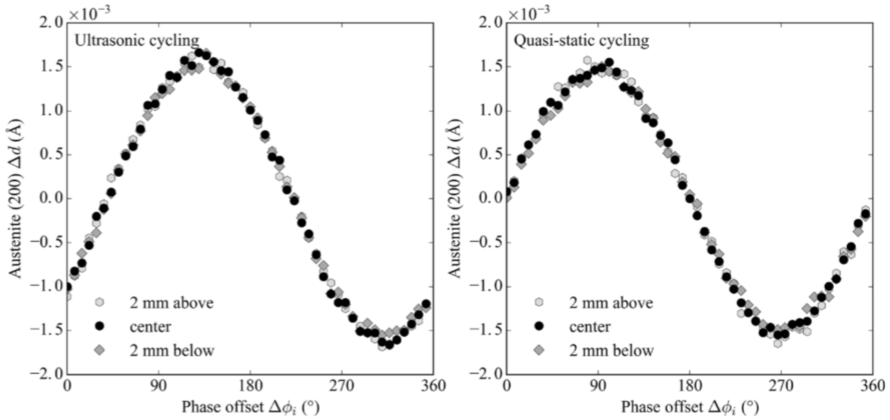
Main objectives addressed during the experiment were:

1) Monitoring strain along the specimen during USF and CF cycling
2) Mapping and behaviour of the TZ during USF cycling
3) Checking for occurrence of phase transformation (A, R, M) during an USF cycle at different positions

Outcome:

We were able to map the strain distribution within the sample during USF at maximum and minimum load within one ultrasonic load cycle. We could compare strains obtained in USF and CF cycling for the A phase. Furthermore we mapped the TZ in detail in order to gain deeper insight into the transformations occuring during cyclic loading.

Fig. 6 Phase-resolved shift in B2 (200) lattice spacing $d = 2\pi/Q$ in the fully austenitic specimen with respect to d at tensile mean strain in the center of the gauge section, and 2 mm above and below, respectively, during one full ultrasonic cycle ($f_{res} \approx 18.3$ kHz, $\Delta u/2 = 15$ µm, $\dot{\varepsilon} \approx 7 \times 10^1$ s^{-1}) (left) and during quasi-static cycling ($f = 0.1$ Hz, $\Delta u/2 = 15$ µm, $\dot{\varepsilon} \approx 4 \times 10^{-4}$ s^{-1}) (right)



We were able to follow the phase transition A → R → A during one full ultrasonic cycle. Cycling at higher ultrasonic amplitudes revealed M phase at maximum load during a cycle, and M phase vanishing during minimum load, indicating a transformation from A to M phase and back (Fig 8) at ultrasonic frequency.
Additional, during a partial fatigue test, the evolution of the TZ was monitored for potential fatigue damage.

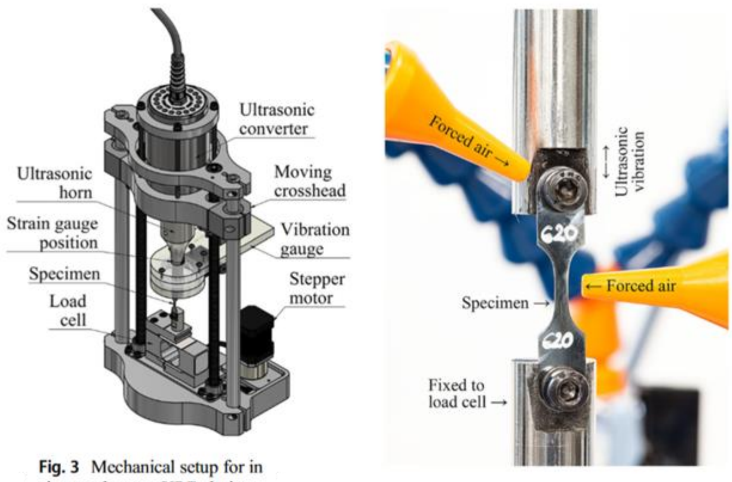


Fig. 3 Mechanical setup for in situ synchrotron XRD during cycling at ultrasonic frequency; schematic (left) and detail view with mounted specimen (right)

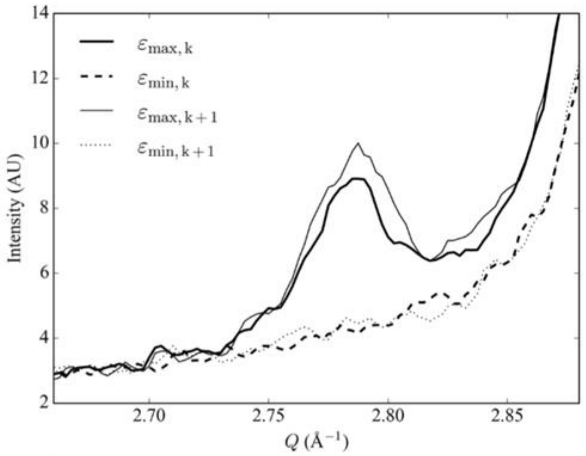


Fig. 8 Austenite - martensite forward and reverse transformation shown by the appearance of the martensitic (101) peak (plotted versus scattering vector Q) during maximum tensile strain ε_{max} and its disappearance during minimum tensile strain ε_{min} for subsequent exposures $_k$ and $_{k+1}$; $\Delta u/2 = 35$ µm, $\dot{\varepsilon} \approx 2 \times 10^2$ s^{-1}

Summarizing:

For the first time we were able to follow a single ultrasonic cycle with X-Ray diffractograms in a time resolved manner at 50 discrete phase postions.

We were able to follow elastic and plastic deformation, as well as phase transformation during 18.3 kHz cycling.

Thus we could demonstrate that phase transformation between A, R and M phase occurs at ultrasonic frequency, which was previously entirely unkown.

We could furthermore show the movement of the TZ and map the strain distribution and phase distribution in the sample during cycling.

The results were published in the scientific journal:

M. Fitzka, et.al. (2019) High Speed In Situ Synchrotron Observation of Cyclic Deformation and Phase Transformation of Superelastic Nitinol at Ultrasonic Frequency. *Experimental Mechanics*, DOI: 10.1007/s11340-019-00562-8.