EUROPEAN SYNCHROTRON RADIATION FACILITY

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Experiment Report Form

	Coupling 3DXRD and DCT-Topotomography methods for the martensite M18R phase characterization during in-situ tensile tests in shape memory alloys	**Experiment number**: MA5178
Beamline: ID11	**Date of experiment**: from: 2022/03/30 to: 2022/04/04	**Date of report**:
Shifts: 15	**Local contact(s)**: Wolfgang LUDWIG	*Received at ESRF:*

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Report

Introduction

Shape memory alloys (SMA) undergo a martensitic transformation during mechanical loading which is reversible; this gives rise to their superelastic behavior. From an experimental point of view, the study of SMA requires the use of diffraction to separately identify austenite and martensite behavior. Most of the works follow either the microstructural evolution under load, or the average behavior of each phase. There are very few results for individual grain behavior in a polycrystal and the mechanical behavior of the martensite M18R phase has never been understood at the grain scale. A first attempt had been done in MA4757 using Diffraction Contrast Tomography and topotomography proposal to index martensite in a polycristal. It did not succeed due to a too high number of grains in the diffracting volume. This proposal consists in an *in-situ* loading experiment of a single crystal of Cu-Al-Be SMA and a coarsed grain polycrystal.

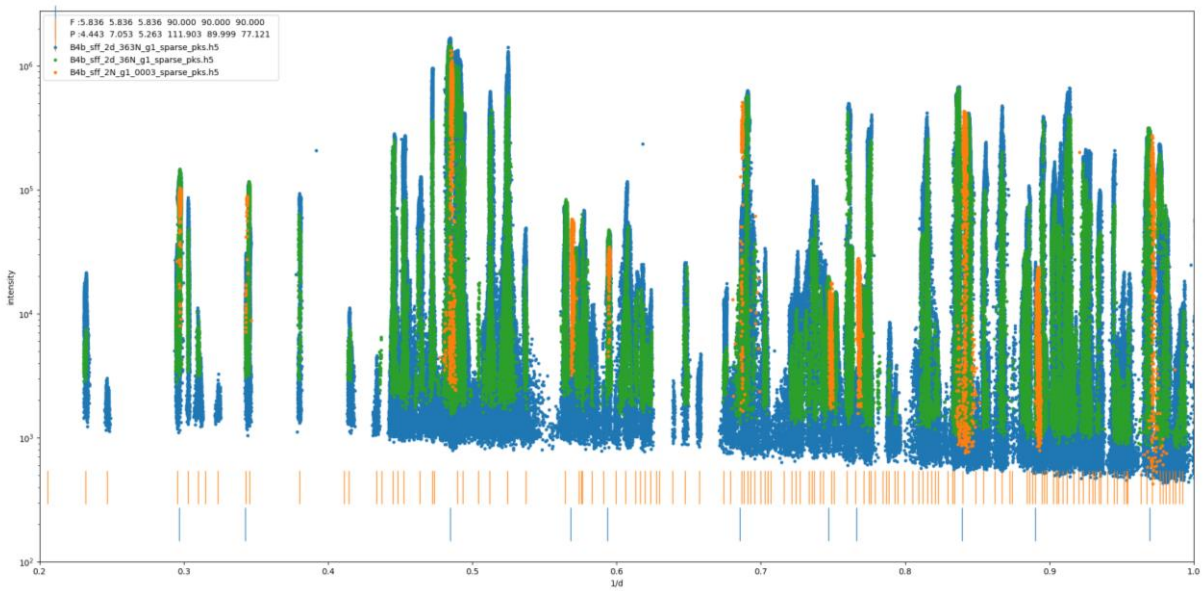
Experimental method:

The studied alloy was a 87.9%Cu-11.5%Al-0.6%Be alloy. It was provided by Nimesis Technology©. Single crystal was in the form of a cyclindrical wire with a 3.5 mm diameter; preliminary measurements (IHMA242) have shown that this ‘industrial single’ crystal was in fact composed of two grains misoriented by 6°. One orientation was located at the surface and the other one in the center. Two ? specimens for nanox tensile rig were machined from the wire center to ensure one single orientaion. The polycrystal was obtained by hot drawing, in the form of a rectangular bar (3*1 mm^2 section). Specimens for nanox tensile rig were prepared from it. An in-situ loading test was performed and was stopped at different loading point to acquire DCT and TT patterns. To access strain/stress state, 3DXRD was also performed; a peculiar grain was chosen in the polycrystal for Scanning-3DXRD measurement . 6 measurement points were recorded during loading and 5

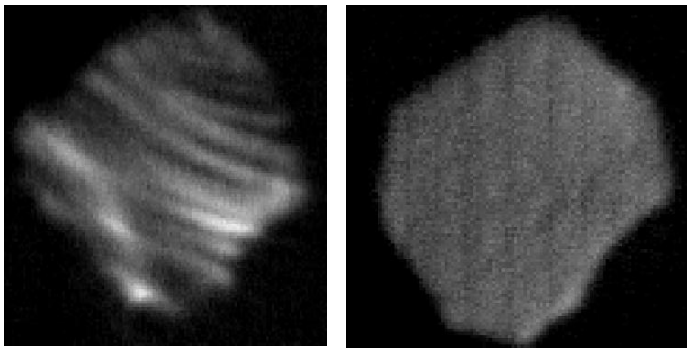
during unloading for the polycrystal. For the single crystal, two measurement points were done in the elastic domain and 3 on the transformation plateau.

First analysis

- **Single crystal :** Figure below indicates fitted position of austenite (blue line) and martensite (orange lines) from literature data. If most of positions correspond, some were missing while extra-positions were observed. So there would be different atoms position in the unit cell not expected and/or different symmetry groups that are not yet taken into account. Works are in progress to improve this first step of indexing.



- **Polycrystal :** in a fisrt step, 3D DCT reconstruction have been made using the classical algorithm SIRT. A 6D reconstruction algorithm, developed recently [6] takes into account the grain strain state; up to now, it has been validated only on theoretical microstrucutres. It will be applied to the SMA; results will be compared between both algorithms. Last an algorithm to process topotomography reconstruction has been developed. Mapping of the 10 studied grains (see below) will be compared to the DCT microstructure obtained with the 6D reconstruction.



Topotomography reconstruction of 1 grain :
Left: during transformation (martensite plates are visible)
Right : after unloading, martensite has reversed.

Further works

Besides the continuation of preliminary analysis presented before, scripts have also to be develeped to process scanning-3DXRD data. Expected results are the orientation, strain and stress fields inside the grain in the initail state, the elastic domain and as it has started to transform: the final goal is to get these data both in autenite and martensite phase. Internal Stress field can be mapped in austenite first, as the stifness tensor of monoclinic martensite is unkown up to now. The experimental results will also be used for / compared to mechanical modeling.