Experimental report

Proposal	**MA-4757**	Date	**11-13 June 2021**
Proposal Title	**In-situ 3DXRD/DCT and Topotomography methods for the martensite M18R phase characterization**		
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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 [1]. There are very few results for individual grain behavior in a polycrystal [2-4] and the mechanical behavior of the martensite M18R phase has never been understood at the grain scale. This proposal consists in an *in-situ* loading experiment of a polycrystalline Cu-Al-Be SMA, using Diffraction Contrast Tomography and topotomography.

Experimental method:

The studied alloy is a 87.9%Cu-11.5%Al-0.6%Be alloy. It was provided by Nimesis Technology© and was obtained by hot drawing. Specimen for nanox tensile rig was cut from a rectangular bar (3*1 mm^2 section); final dimensions are presented figure 1.
At room temperature, the alloy is fully austenitic; its grain size was measured by optical microscopy after polishing and chemical etching with a Mi14 solution (figure 2). It is around 80 ± 5 µm.

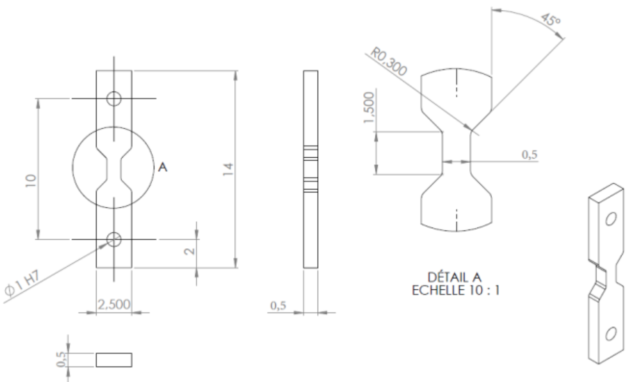


Figure 1 : Nanox sample

Figure2: Initial microstructure

An in-situ loading test was performed and was stopped at different loading point to acquire DCT and TT patterns. 12 measurement points were recorded during loading and 2 during unloading.

Results

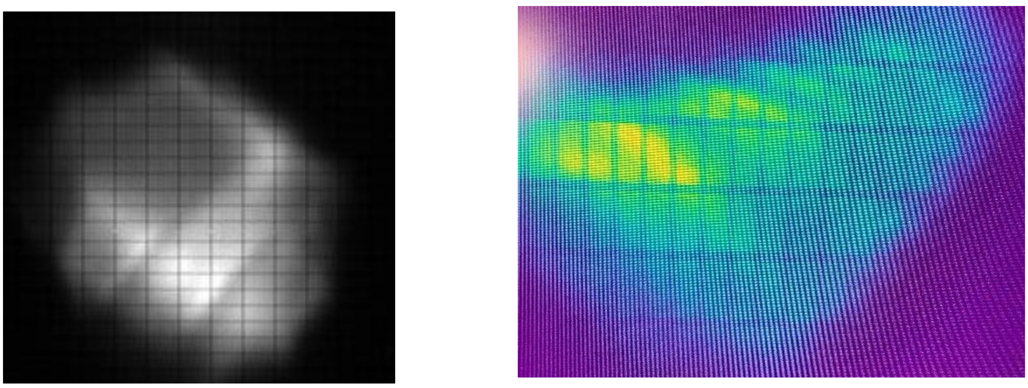
A first DCT analysis was performed before loading; the initial microstructure was then reconstructed. It was used to select particular grain for further topotomography: we consider different orientations, locations (surface/volume of the sample) and/or grain sizes. Table 1 summarizes selected grains.

Grain number	Orientation	Position	Used diffraction vector	Theoretical relative intensity
3	[110]	Bulk	220	100
9		3’ Neighbor	311	**3,6**
10		Surface/ large grain	311	**3,6**
15	[110]	25’ neighbor	220	100
18	[001]	3’ neighbor	200	**4,1**
25	[111]	Bulk	111	7,8
47	[110]	Surface / large	220	100
81	[001]	Small	200	**4,1**

Table 1: selected grain for topotomography

At each loading step, DCT and TT were done. Unfortunately, we could not measure the sample strain; only the force was accessible.

In the selected grains, the first variant was observed at the 10^{th} loading point, corresponding to a macroscopic applied stress of 340 MPa. The grain 18 was the first one to transform (figure 3a): one variant appears in the grain. This could be expected as it is [001]-oriented in the tensile direction, which is known as an easy direction to transform.



(a) (b)

Figure 3: (a) first variant observed in grain 18 at 340 MPa. (b) Grain 10 at 360 MPa.

At the next loading point (applied macroscopic stress of 360 MPa), all the studied grains have started to transform and several martensite plates are visible (figure 3b). Upon unloading, all these stacking plates have disappeared: this confirms that they are due to martensite and not to another inelastic phenomenon.

Further works:

The first key factor of this experiment is that we were able to observe martensite formation in a CuAlBe alloy at the grain scale, which has never been done up to now.

All the studied grains will be reconstructed in 3D to identifiy the number of variants activated in each grain, their development. Their orientations well be determined first using crystallographic approach, given the initial orientation of the austenitic grains and the habit planes. All these processing are currently under works.