ESOMAT 2009, 02003 (2009)
DOI:10.1051/esomat/200902003
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Intragranular austenite orientation evolution of a Cu-Al-Be SMA during an in-situ tensile test

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Abstract. We used synchrotron radiation to study the local behaviour of individual grains embedded in a polycrystal. First the existing methods have been extended for the first time to the case of SMA by taking into account the phase transformation. Then it was applied to determine the rotation of different grains during an in-situ tensile test. Results are discussed in comparison with the occurrence of the martensitic transformation in the grain and with respect to their mean orientation.

1. Introduction

Martensitic transformation in SMA can be induced and controlled by external stress. The transition is accompanied by the reversible evolution of large high successive inelastic strains which leads to the superelasticity in thermomechanical loads. Studies on single crystals studies report a lots of experimental data to understand the rules which provoke the motion of the austenite/martensite interfaces during mechanical tensile loading [1-4]. However if these rules are applied to grains inside a polycrystal it gives a crude simplification. The strain compatibility in the grain boundaries and the redistribution of stress among grains of the transforming polycrystal affects significantly its overall macroscopic stress-strain-temperature behavior and the superelasticity decreases from 10% for a single crystal to 5% for polycrystalline alloys.

Multi-scale micromechanical [5, 6] and numerical [7] models were developed but too few multi-scale experimental values were measured to validate these models. The aim of the paper is to report on two in-situ experimental synchrotron X-ray diffraction studies of the superelastic behaviour associated with the stress induced martensitic transformation in a CuAlBe alloy. These diffraction methods were adapted to transforming materials in order to determine orientation evolution of the austenite at the grain scale.

2. Experimental technique

2.1 Experimental setup

Measurements were performed using the three dimensional X-Ray diffraction method (3DXRD) [8] developed on the ID11 beamline at the European Synchrotron Radiation Facility (ESRF) at Grenoble, France. The 3DXRD method is based on diffraction of high energy X-Rays and allows fast and non-destructive three dimensional characterization of the local distribution of crystallographic orientations in the bulk. The 3DXRD method is a volume characterization using monochromatic beam coupled with an analysis procedure treating each grain through the volume as a single crystal. Beam size is 200*200μm^2. Figure 1 presents a sketch of the 3DXRD installation. For 3DXRD analysis, the sample is rotated around the omega axis and for each position, diffraction spots are recorded on a CDD-2D detector. This gives the list of coordinates of diffraction spots (ω, θ, η) for a given volume analysis.

Generally, in a polycrystalline sample, many grains diffract simultaneously. Hence diffraction spots originated from different grains appear within the same detector image. We considered only the diffraction patterns coming from the grain located at the intersection between the incident beam and the omega axe. Knowledge of crystal lattice parameters and space group symmetry are used to simulate the diffraction pattern that should be obtained for all possible orientations in Euler space.

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Article available at http://www.esomat.org or http://dx.doi.org/10.1051/esomat/200902003

Comparing the simulated scattering vectors with the observed one allows grain indexation. Two indexing software packages: ImageD11 [9] and Grainspotter [10] are used to determine grain orientations during superelastic cycle. The occurrence of a stress-induced martensitic transformation upon loading is detected when characteristic peaks associated to the martensite phase appear on the recorded diffraction pattern. It is both an advantage and an inconvenient. Theoretically, informations on both austenite and martensite phases can then be obtained but peak analysis rapidly becomes rather complex as diffraction spots from the two crystallographical lattices interfere. In the following we only focus on austenite peaks analysis which allows following the microstructural evolutions within the austenite phase during the transformation.

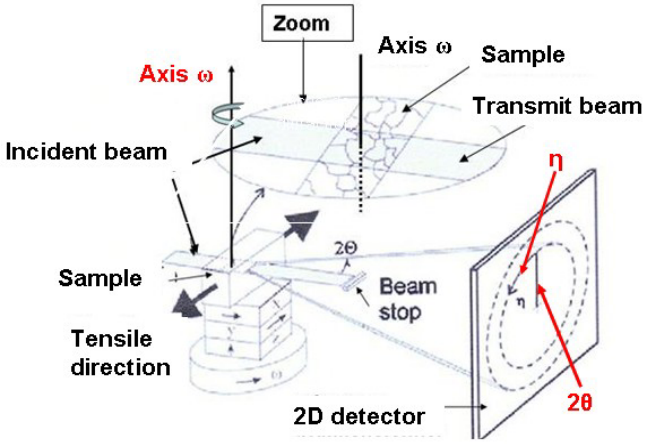
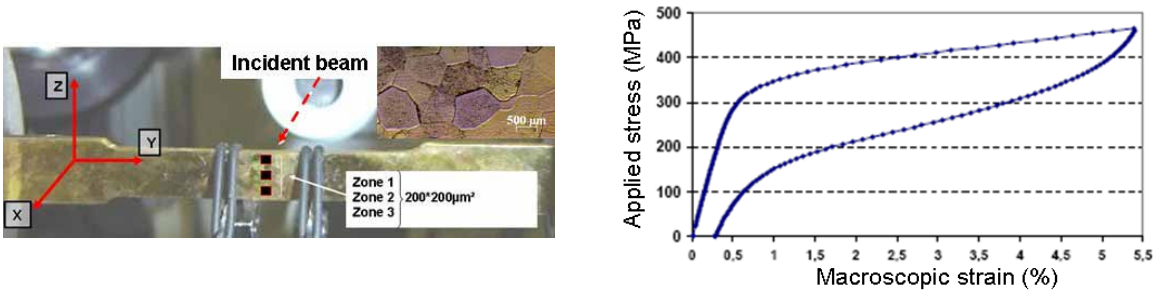


Fig. 1. Sketch of the 3DXRD installation

2.2. Tensile samples

We studied a Cu-Al-Be shape memory alloy in order to have sample with coarse grain size to reduce the number of grains which diffract simultaneously. We chose a chemical composition presenting a very low martensitic transformation temperature to obtain a fully austenitic microstructural state at room temperature and to reduce the occurrence of stabilized martensite. This chemical composition is near Cu-11.6%Al-0.6%Be (wt.%) ; this corresponds to a martensitic transformation start temperature at -115°C. The alloy was homogenized in the β-phase above 650°C and water-quenched. In situ dog-bone tensile specimens were then machined with a width of 8mm and a thickness of 2mm (Fig. 2a). Synchrotron measurements were performed during in-situ loading test; the macroscopic stress-strain curve obtained is plotted figure 2b. From this curve, one can estimate the critical transformation stress on that sample is about 300MPa.



a) b)

Fig. 2. a) Tensile sample in the tensile rig with the three analysis zones considered. In the upper-left corner: micrography of the central area showing the grain size. b) Macroscopic stress-strain curve.

3. Grains rotation during in-situ tensile test

The 3DXRD method was applied to determine the amount of rigid-body rotation experienced by the austenite crystallographycal lattice within grains during a superelastic loading. As the mean grain size is 500 μm that is larger than the synchrotron beam size we define 3 different analysis zones on the samples, labelled zones 1 to 3 (Fig. 2a) in order to be able to analyse several grains. These 3 zones allow isolating 4 grains that were followed for each loading step during loading and unloading sequences. We present successively the observations made during these two steps.

3.1 Upon loading.

In figure 3, the 4 grains orientation is plotted in an inverse pole figure in the tensile direction. The four grain analysed have different crystallographycal orientations. This orientation evolves on a rather large amount during the loading sequence: the different dots correspond to the crystallographycal orientation of the austenitic lattice at each loading steps (from 0 to 465MPa).

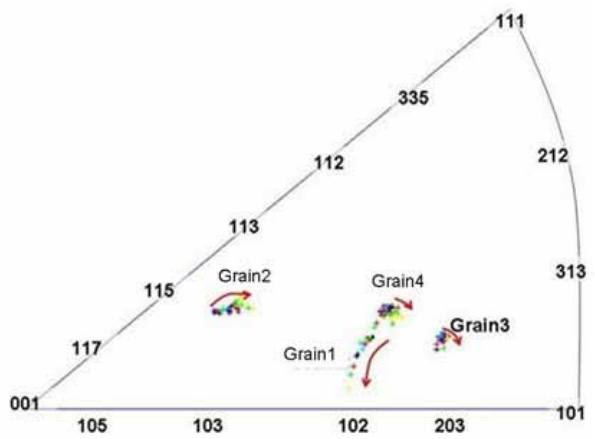


Fig. 3. Inverse pole figure in the tensile direction showing the orientation of the four grains during loading.

Thanks to the initial crystallographic orientation of the grains, we calculate the Schmid factor for phase transformation for each grain. Values are summarized table 1.

Table 1. "Schmid" factor for transformation of the 4 grains.

	Grain 1	Grain 2	Grain 3	Grain 4
Schmid factor	0.31	0.49	0.40	0.41

Grain 2 has the highest Schmid factor whereas grain 1 is the less favourably oriented grain. During loading, we observe that the 4 grains rotates in various directions even if grains 2, 3, 4 seems to rotate towards the same direction. Table 2 summarizes the averaged rotation of the four grains.

Table 2. Rotation of the tensile direction in comparison to the crystal axis.

Macroscopic loading		**Rotation for grain label**			
Stress (MPa)	**Strain (%)**	**1**	**2**	**3**	**4**
300	0.6	0.5°	0.5°	0.3°	0.4°
360	1.3	1°	1°	0.5°	0.5°
410	3	1.9°	1.5°	0.6°	0.6°
430	3.7	2.9°	1.8°	1°	0.8°
445	4.5	3.8°	2.0°	1.8°	1.3°
465	5.4	4.9°	2.5°	2°	1.4°

In the elastic domain (applied stress lower than 300MPa), the grains rotate since the first loading step; the rotation angle is quite the same for all orientations. Then the martensitic transformation starts in grain 2: its rotation increases faster than in grains 3 and 4. Almost at the same time, grain 1 starts to transform too though it has the weakest Schmid factor. This behavior has already been reported by Kaouache et al. [11]. They have

shown that the correlation between the order of transformation and the Schmid factor is not always true. This result comes from the stress heterogeneity inside a grain and the fact the martensitic transformation is a localized phenomena. A stress concentration due to intergranular interactions can produce a local stress state superior to the average stress in the grain which contributes to transform the grain before another grains better oriented. In our study the evolutions of the averaged rotation during the loading do not always fit with the increase of the Schmidt factors as the grains 1 and 4. In fact for the grains 2 and 3 the strain accommodation of the neighboring grains should counteract with the expected increase of the Schmid factor.

3.2 Upon unloading.

The austenite rotations during unloading are presented Fig. 4 for the 4 grains.

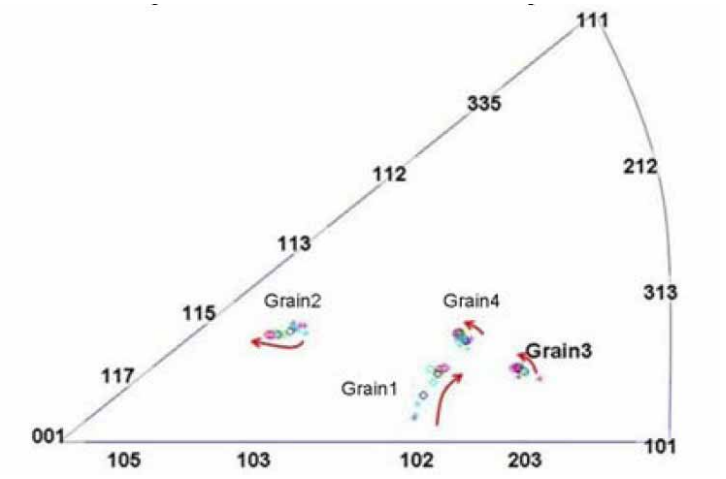


Fig. 4. Inverse pole figure in the tensile direction for the 4 grains.

We observe that the grains rotate towards their initial position. But, the reverse rotation is not complete: a residual misorientation remains; its magnitude varies between 0.2° and 0.5°. The values are summarized in table 3.

Table 3. Residual rotation of the 4 grains

Grain	1	2	3	4
Residual Rotation (°)	0.3	0.4	0.5	0.2

Therefore one can conclude there are an elastic part and a "plastic" part in the grain rotation. The residual misorientation (by reference to the initial state) may be due to internal stresses or retained martensite [12]. If the elastic rotation varies a lot as a function of the grain orientation, the plastic residual orientation is much less sensitive to the crystallographic orientation.

4. Conclusion

For the first time, individual grains rotations have been measured in the bulk material during a phase transformation using synchrotron radiation and in-situ tensile tests. During loading, rotation can be divided in two steps: before transformation, the austenite grains rotate slowly and when martensite starts to appear, the rotation increases. Upon unloading, a reverse rotation is observed; the final misorientation is less than 0.5° by reference to the initial orientation.

The authors are grateful to J. Wright and T. Buslaps ((ESRF Grenoble) for useful help in experiments.

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