# Experimental report

16/09/2019

**Proposal:** **1-02-260**

**Council:** 10/2018

**Title:** Study of twinning using in-situ neutron diffraction experiments and acoustic emission during compressiontest in Mg single crystal.

**Research area:** Engineering

**This proposal is a new proposal**

**Main proposer:** **Gerardo GARCES**

**Experimental team:** Kristian MATHIS
Pavol SIMKO
Gerardo GARCES

**Local contacts:** Sandra CABEZA

**Samples:** Mg

| Instrument | Requested days | Allocated days | From | To |
|---|---|---|---|---|
| SALSA | 3 | 3 | 29/07/2019 | 01/08/2019 |

**Abstract:**

Twinning is an important deformation mechanism in magnesium. Twinning process has three stages: nucleation, propagation and growth. The proposal will study twinning process using neutron diffraction and acoustic emission during in-situ compression magneisum monocrystal oriented in different lattice orientations.

Two single crystals with two sample orientations (A and B) where prepared with the [0002] and [1010] directions parallel to the compression axis. Compression samples were prisms of section of 4x4 mm$^2$ and length of 6 mm. In the first orientation (A), basal slip and tensile twinning is forbidden. In the second orientation (B), tensile twinning is activated.

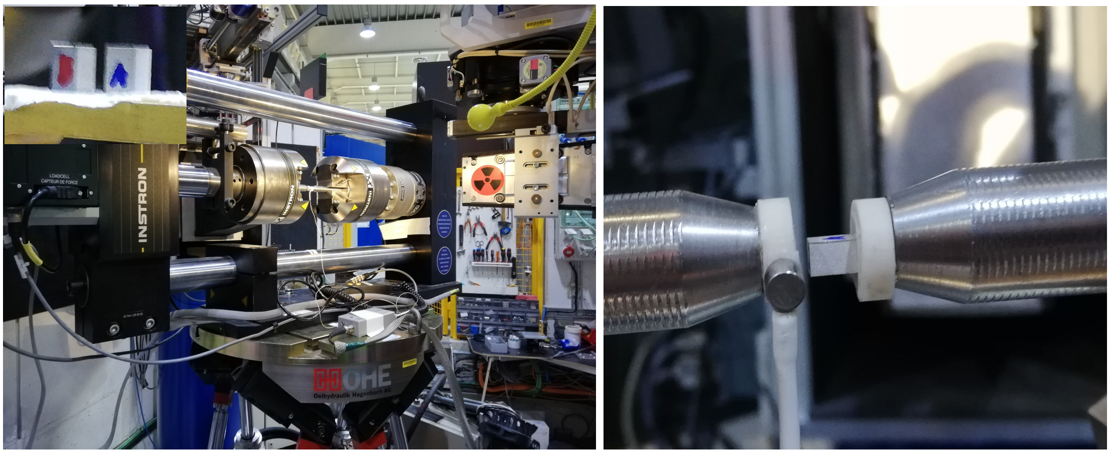


Fig 1 (a-c). Set up of the compression test of single crystal in the beamline SALSA

The gauge volume defined by the primary and secondary collimators was 2x2x2 mm$^3$ and was positioned in the center of the compression sample. The diffraction peaks corresponding to the (1010) y (0002) planes parallel to the compression axis were recorded simultaneously centering the area detector at 2θ=37º. Ceramic plates were located between the compression sample and the clamps. The Acoustic Emission detector is located in the ceramic plates as it is shown in Figure 1c.

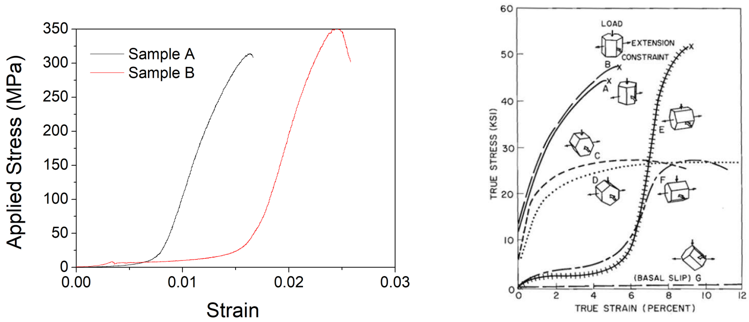


Fig 2 (a,b). Compressive curve obtained during in-situ experiment and published in the paper of Kelley and Hosford.

Figure 2 shows the compressive test obtained for sample A and B. Results are in good agreement with the study of Kelley and Hosford (Sample A = Sample A in figure 2b, Sample B is Sample E in figure 2b. The measurement was carried out in a continuous mode (list mode). In this acquisition mode, the first data reduction consists in the data binning parameters selection: a moving average of time and overlapping positions. From a first iteration (see Figure 3) the initial parameters have been selected to continue reducing all data. Figure 3a shows the moving average distribution for sample B: binning of 60s acquisition time, divided into 20pt for the first 300s of the experiment analyzed. In Fig3b the stacking of detector signal for those 300s can be observed, where at a certain time the peak is disclosed in Figure 3c. The final comparison of binning parameters in order to select the final ones is given in Figure 3d. However, deeper analysis and correlation to acoustic emission is needed and on-going.

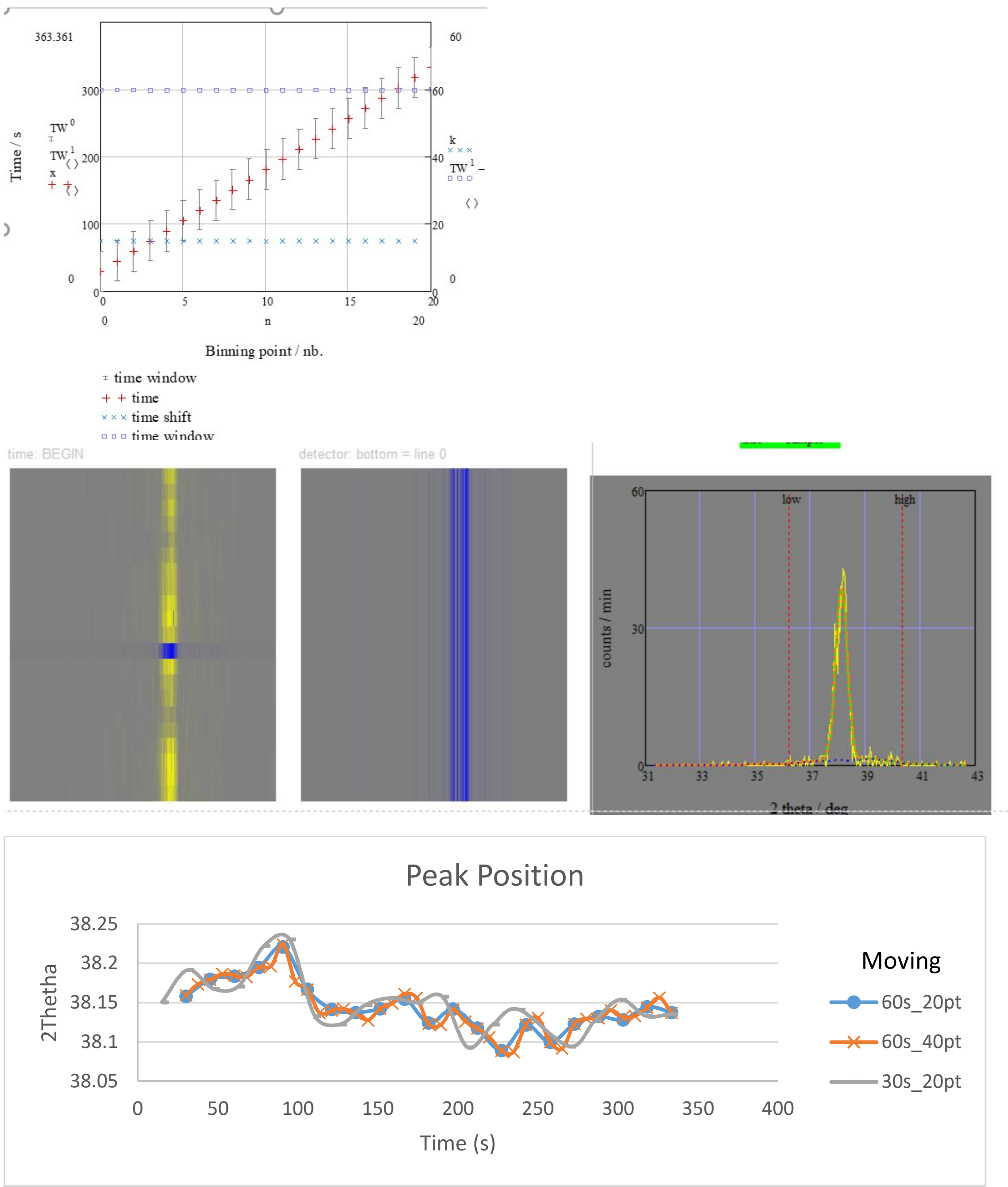


Figure 3 First data reduction parameters for Sample A (a) moving average partitioning in 60sec for 20 points (b) stack of detectors for the first 5min and in blue (c) 2D detector image at that time with (d) peak fit associated.

In conclusion, we have been able to disclose the twinning activation and growth. On one hand, the resolution of the diffraction peak of the single crystal was achieved. On the other hand, the continuous acquisition mode at SALSA has proved essential for the study of the plastic deformation in single crystal magnesium alloys. Further and detailed analysis of peak shift and acoustic emission signal are in process with ILL scientists. Final results will be implement in a draft for a peer-reviewed journal.