ESRF	**Experiment title:** Coupling of plastic deformation and magnetism in Heusler alloys: an in situ microLaue experiment during compression of micropillars	**Experiment number**: A32-2-844
Beamline: BM32	**Date of experiment**: June 8th to 13th, 2022	**Date of report**: 9 January 2023
Shifts: 15	**Local contact(s)**: MICHA Jean-Sebastien, ROBACH Odile, PURUSHOTTAM RAJ PUROHIT Ravi Raj Purohit	*Received at ESRF:*

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Report:

Introduction

Magnetic ordering is intimately related to local atomic environment (nature and distances of neighboring atoms) and mechanical deformation can lead to a wide range of interesting phenomena. The coupling between mechanics and magnetism is particularly evident in ordered intermetallics that undergo plastic deformation, where dislocations may place atoms on new atomic sites, and therefore create new chemical environments and interatomic distances. In a recent study [1] the group at UCSB has investigated the influence of plastic deformation on the magnetic behavior of the ferromagnetic Heusler alloy $MnAu_2Al$. A decrease in net magnetization after plastic deformation of compressed ingots or ground powders of $MnAu_2Al$ [1] has been observed, which shows the role of plastic deformation on magnetic properties in this intermetallic alloy; the converse could also be a potent effect wherein long-range magnetic order could influence the energy landscape for dislocations and their mobility. The relative simplicity and large magnitude of this mechanism makes the further exploration of plasticity in ordered intermetallics a promising direction for obtaining mechanical control over magnetic behavior. It is the aim of the present experiment to investigate in detail the plasticity of the simple room temperature ferromagnet $MnCu_2Al$ ($L2_1$ structure with space group $Fm\bar{3}m$) Heusler alloy. To achieve this goal, we have compressed $MnCu_2Al$ single crystal micropillars and followed *in situ* elastic deformation as well as defect initiation by using micro-Laue diffraction.

Preparation of samples

Sample preparation is decribed in figure 1. Wedges have been first prepared fom bulk sintered polycrystals. The orienetation of the grains at the top of the wedge have been determined by EBSD (Electron back-scattered diffraction). Then 50 microns wide pedestals are etched from the top of the wedge with a fs laser. Finally micron-sized pillars are etched from the pedestals with FIB (Focused Ion Beam).

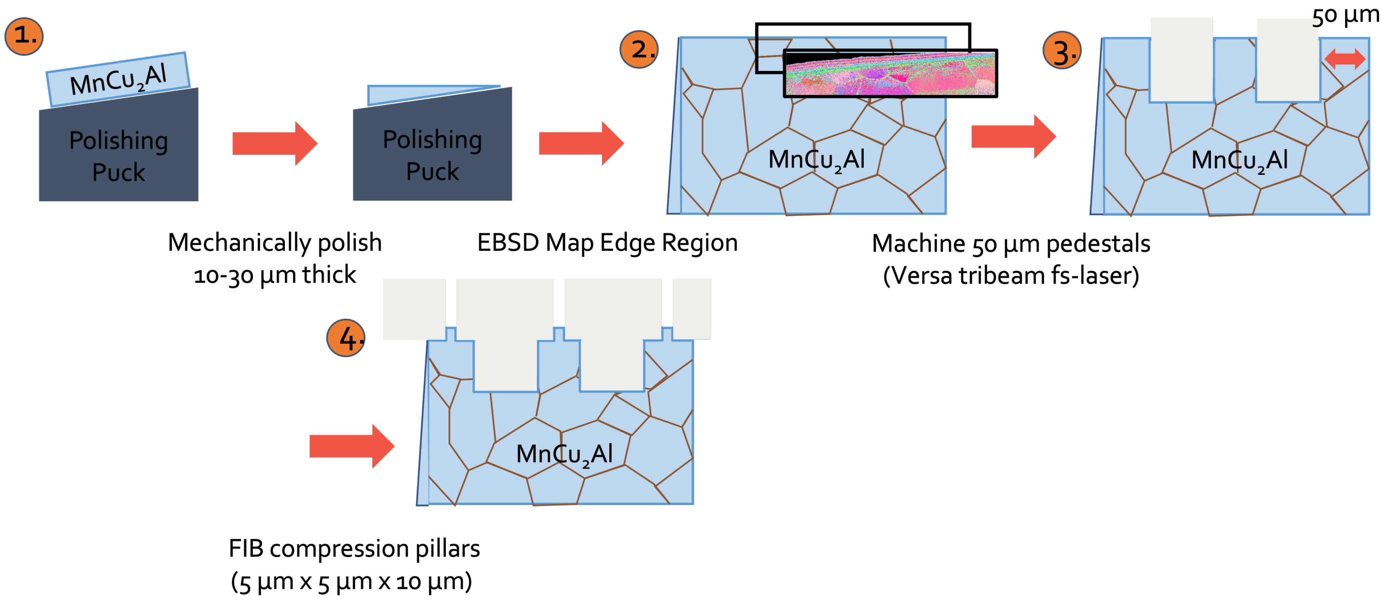


Figure 1 : Schematic of the fabrication workflow for micropillars.

This procedure allows getting micropillars that are fully detached from the bulk and thus easy to reach with the X-ray microbeam. An example of as-prepared sample is shown in figure 2.

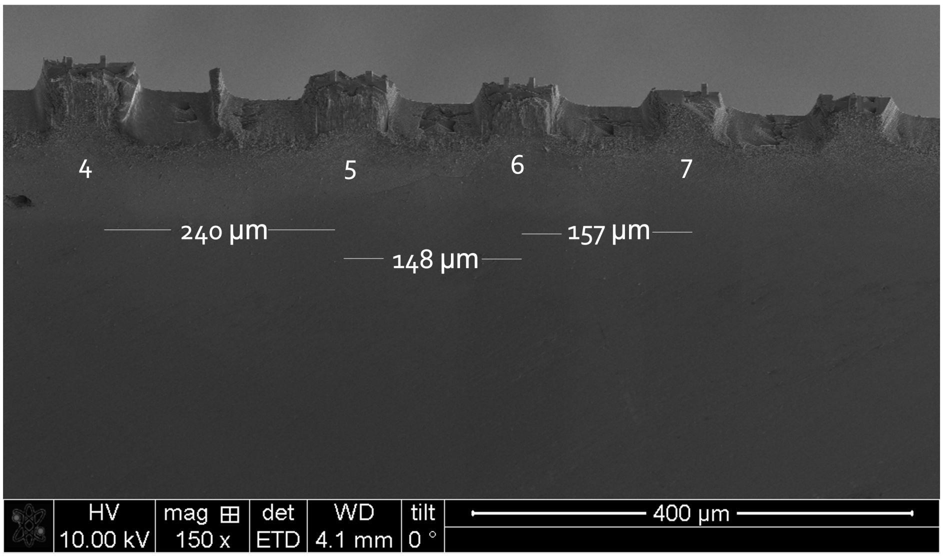
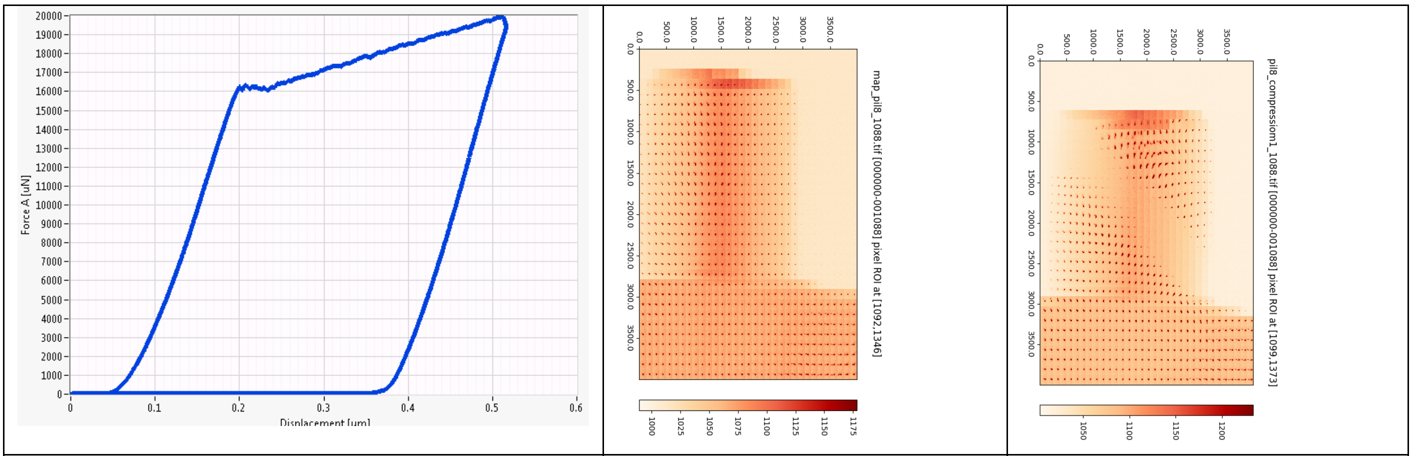


Figure 2: SEM (Scanning Electron Microscopy) image of part of wedge 1.

<u>Description of the experiment at the beamline: methodology and results</u>

For *in situ* microcompression tests, the nanoindenter FT-NMT04 (FemtoTools) [2] equipped with a diamond flat punch was brought by IM2NP group and installed on the goniometer of the BM32 beamline. This was actually the first time this device was installed on BM32 beamline. During compression in displacement-controlled mode with a displacement rate of 30 nm/s, Laue microdiffraction patterns were recorded using a sCMOS camera installed at 90° with respect to the incident polychromatic X-ray beam. The beam size on the 40° inclined sample was 300 nm x 500 nm. Cyclic loading-unloading curves with increasing maximum load were applied to each micropillar until a clear sign of plastic deformation was visible in the load-displacement curve. During compression, in order to prevent any unwanted additional force in the sample under loading time scans at a fixed position on the pillar were acquired. 2D Laue microdiffraction maps were recorded of the micropillars both in their pristine state and after compression. An example of such an experiment is shown in figure 3 for pillar 8 on wedge 2. Plastic yielding occurs at a force of 16 mN, which translates in a yield stress of 520 MPa. The unloaded pillar exhibits a plastic strain of 3.7%. Figures 3 b and c show mosaics of a single

Laue spot from the pristine and unloaded pillar. A 45° inclined band where the Laue spot has gone outside the pre-defined box is most probably a sign of slip. A thorough analysis of crystal rotation using the LaueTools software is underway.



a	b	c

Figure 3 : In situ compression of Pillar 8 from wedge 2. (a) loading and unloading curve showing that pillar has undergone plastic deformation; (b) mosaic of one Laue spot from pristine pillar; (c) mosaic of one Laue spot from unloaded deformed pillar.

Table 1 is a list of analyzed samples together with their orientation, Young's modulus and maximum Schmid factors. The orientation of the pillars is more easily visualized in the inverse pole figure representation shown in figure 4. In total sixteen samples have been measured on the beamline (12 compressed *in situ* and 4 measured *postmortem*).

Wedge	Pillar	[hkl]	Young Modulus (GPa)	Max Schmid Factor	Compression
1	1	217	93	0.48	*UCSB*
1	2	116	86	0.45	*In situ ESRF*
1	4	225	127	0.43	*UCSB*
1	5	214	124	0.49	*In situ ESRF*
1	6	214	124	0.49	*In situ ESRF*
1	7	516	152	0.46	*In situ ESRF*
1	8	516	152	0.46	*In situ ESRF*
1	10	516	152	0.46	*In situ ESRF*
2	3	221	185	0.41	*UCSB*
2	4	105	83	0.47	*In situ ESRF*
2	5	416	138	0.49	*UCSB*
2	6	416	138	0.49	*In situ ESRF*
2	7	416	138	0.49	*In situ ESRF*
2	8	416	138	0.49	*In situ ESRF*
2	9	212	185	0.41	*In situ ESRF*
2	10	212	185	0.41	*In situ ESRF*

Table 1 : List of measured samples with their orientation. Four samples have been mechanically tested at UCSB and measured *postmortem* at ESRF. Twelve samples have been compressed *in situ* on BM32. Young's moduli are calculated from the constants of elasticity reported in [3] and maximum Schmid factors are calculated for the {110}<111> slip systems reported in [3].

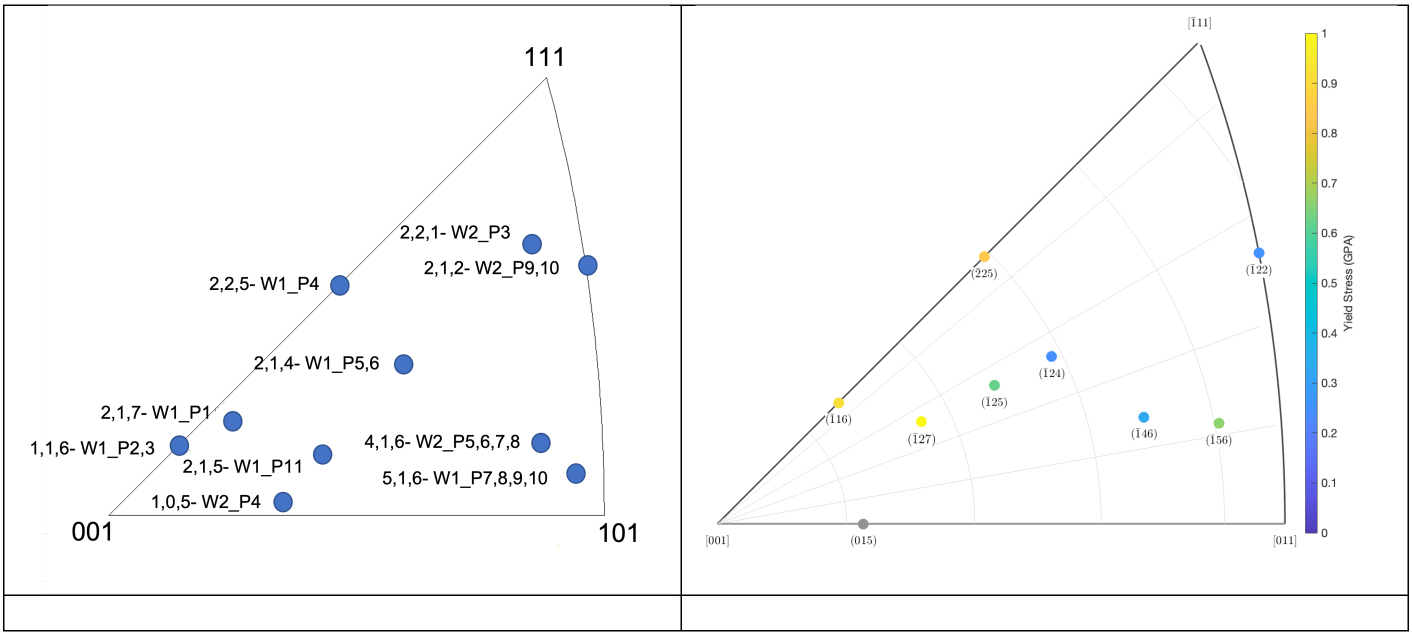


Figure 4 : Inverse pole figure representations of investigated Heusler pillars: (a) orientation ; (b) yield stress and orientation.

Conclusion and future work

For the first time the FT-NMT04 nanoindenter has been installed successfully on beamline BM32. Twelve single crystal $MnCu_2Al$ micro-pillars have been compressed *in situ* while recording Laue diffraction patterns. A detailed analysis of these patterns (in particular the rotation of the crystal lattice) is underway using the LaueTools software and the LaueTools neural network (newly developed by the BM32 beamline staff). In addition, the compressed pillars are being thoroughly characterized by transmission electron microscopy.
This successful experiment opens avenues for continuing using *in situ* Laue microdiffraction for the investigation of the coupling between plasticity and magnetism in Heusler alloys. In particular, it would be of high interest to investigate the plastic behavior of alloys such as $MnCu_2Al$, which is not ferromagnetic at room temperature.

References

[1] E. Levin *et al.*, Physical Review Materials 5, 014408 (2021).
[2] https://www.femtotools.com/products/ft-nmt04/description
[3] M. Green, G. Chin, J.B. Vander Sande, Metallurgical Transactions A 8A, 353 (1977).