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Cryo-FIB and Transmission Electron Microscopy of Cryoformed Metals

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Cryogenic forming has been extensively studied due to its particular deformation behavior. When it comes to FCC metals, the cryogenic deformation may result in improvements in mechanical properties, such as strength and/or ductility [1]. The enhancement occurs because of the partial suppression of dynamic recovery, which induces microstructural refining due to an increase of internal defect density [1,2]. Therefore, most deformation cryo-studies conduct the material characterization at room temperature, providing sufficient driving force to initiate static recovery mechanisms [3]. Some authors utilize *in situ* X-Ray Diffraction (XRD) to analyze the cryogenic forming [2,4,5]. It keeps the sample at cryogenic temperature, which avoids the recovery mechanisms. In this context, the cryogenic TEM analysis is a powerful technique that will complement XRD on explaining the phenomena behind cryogenic deformation.

This work is an extension of the previous work carried out by Namur, et al [6]. Silver was the material chosen for this experiment due to its low SFE (22 mJ/m^2) [2]. As well described in recent works [2,5], silver shows great improvement in mechanical properties - higher ductility and strength - when deformed at cryogenic temperature. This is a result of partial suppression of dynamic recovery proven by microstructural refinement [2,5]. A rectangular specimen with dimensions 3.5 x 3.5 x 8.0 mm was cryogenically deformed by compression and reduced in thickness to 1.44 mm. The sample was prepred by immersion in liquid nitrogen during deformation, for which a thermal box was adapted in a hydraulic press. In order to avoid warming the sample and recovery mechanisms, the sample was kept in liquid nitrogen after deformation. The Quorum PPT 3010 cryo preparation system was used to transfer the sample to the Helios NanoLab 600 DualBeam, FIB-SEM. The specimen was mechanically attached to a cryo sample holder (Figure 1 – A). In one side the deformed silver sample was attached using copper tape (white arrow); and in the other side, a modified TEM grids was mounted in a Cryo TEM Autoloader ring clip (red arrows). The microscope was equipped with the OmniProbe AutoProbe™ 200 in-situ sample lift-out system for foil preparation. The method for cooling the needle was developed by the authors in a previous work [6]. The temperature for the silver foil (Figure 1 – B) preparation on FIB-SEM was stabilized in -140º C.

On completion of the lamella preparation in the FIB, the grid was transferred to an autoloader cassette. The autoloader was moved to the Thermo Scientific Krios G3i Cryo-TEM, which has a 300 kV x-FEG source. This microscope has high-resolution performance due the system's stability (reduced thermal drift) during data acquisition sessions. The cryo-TEM image shows high dislocation density, pile up dislocations and deformation cells (Figure 2). The sample was at cryogenic temperature for the duration of the experiment, from deformation step until the TEM imaging acquisition.

The result of this study opens a new front of opportunities to analyze cryogenically deformed metal samples without thermal influence. The FIB preparation and the TEM analysis were successful. The next step is to conduct exploratory experiments to understand the mechanisms involved in the microstructural evolution of these materials before and after heating. This preparation methodology also can be used in other science areas, e.g., biological science.

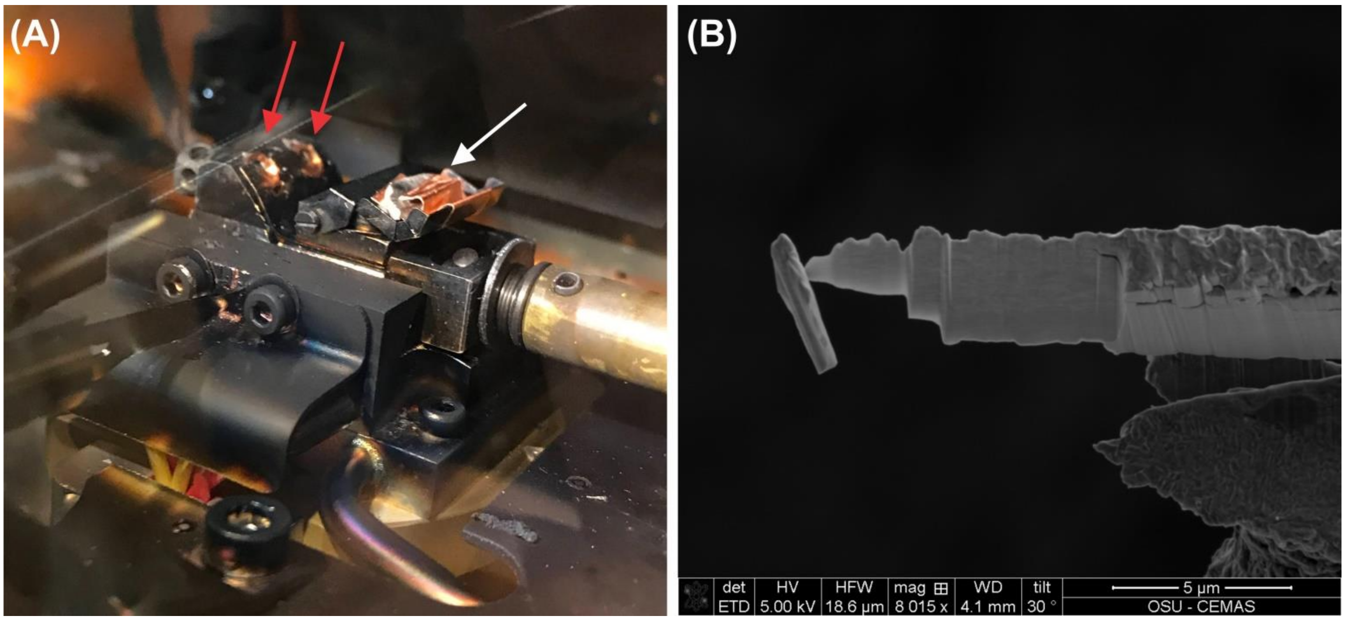

Figure 1. (A) Cryo-SEM transfer system with the deformed silver sample attached to the sample holder using a copper tape, and two TEM grids. (B) Silver foil obtained by Dual Beam FIB at cryogenic temperature.

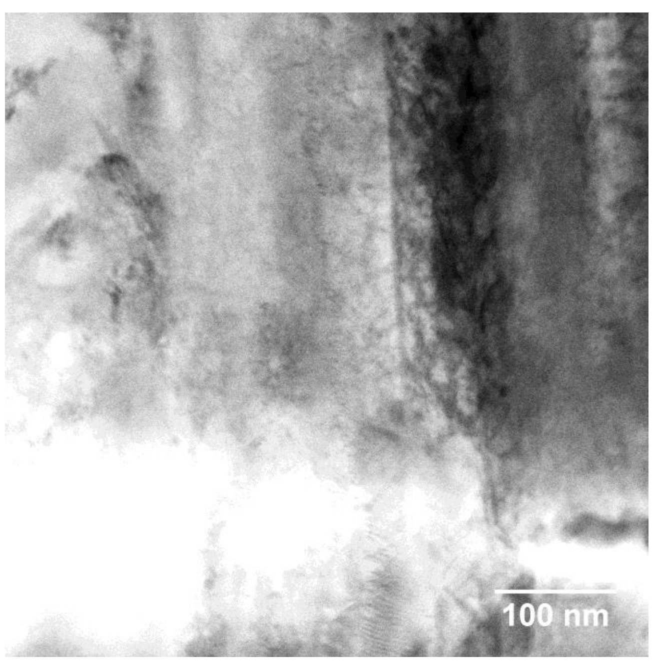

Figure 2. Silver sample cryoformed by Cryo-TEM.

References:

[1] DCC Magalhães et al., Journal of Materials Science **52** (2017), p. 7466. doi: 10.1007/s10853-017-0979-8
[2] MT Izumi et al., Journal of Materials Engineering and Performance [Online] **28** (2019). https://doi.org/10.1007/s11665-019-04226-5
[3] MY Maeda et al., Materials Research **20** (2017), p. 716. doi: 10.1590/1980-5373-MR-2017-0054
[4] PHF Oliveira et al., Materials Science and Engineering A [Online] **813** (2021). https://doi.org/10.1016/j.msea.2021.141154
[5] SV Navas et al., Cryogenics [Online] **120** (2021). https://doi.org/10.1016/j.cryogenics.2021.103384
[6] RS Namur et al., Microscopy and Microanalysis **25**(S2) (2019), p. 916. doi:10.1017/S1431927619005312

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