APPLICATION OF IN SITU HYDROGEN CHARGING DURING MICROMECHANICAL TESTING

Szilvia Kalácska, Mines Saint-Etienne, Univ Lyon, CNRS, UMR 5307 LGF, France
szilvia.kalacska@cnrs.fr
Vincent Barnier, Mines Saint-Etienne, France
Guillaume Kermouche, Mines Saint-Etienne, France
Frederic Christien, Mines Saint-Etienne, France

Understanding mechanisms of deformation at the sub-micron scale is the key for designing new materials and alloys for industrial applications, as the mechanical behavior of materials at such small scale differs strongly from macroscale. It requires the determination of strains/stresses [1], dislocation distribution [2, 3] and the overall microstructure evolution, which is often extremely challenging. Microstructural processes during external mechanical loading are hard to observe due to the complex multiscale nature of the phenomenon.

If hydrogen is present in the solid (i.e. by wet electrochemical processes, introduced during manufacturing and environmental exposure), it can cause embrittlement or enhanced cracking, when the material is subjected to stress. This would eventually lead to the reduced lifetime or critical failure of the component. Although it is known for a long time that hydrogen causes degradation of mechanical performance in metals, the microscale mechanisms remain a subject of debate. Direct H-detection within the lattice is an extremely challenging task, while one has to deal with continuous diffusion and outgassing issues from the studied samples. Microstructure observations are still mostly performed post mortem on bulk samples. Due to the mobility of H in metals it is critical to have continuous hydrogen charging on the test piece.

In situ H-charging is therefore essential for future experiments. Samples can be loaded electrochemically through the back surface [4], using a cell compatible with high-vacuum (HV) scanning electron microscopes (SEM). This way, H diffuses into the lattice from the back, avoiding contamination to the surface of interest. The developed system will be discussed in details during the talk, focusing on the coupling of the cell with the nanodeformation stage by performing nanoindentation experiments on H-charged metallic samples. Examples of micropillars deformed by compression will also be presented (Figure 1). High (angular) resolution electron backscatter diffraction (HR-EBSD) [1,2] is used to map the evolved geometrically necessary dislocation (GND) densities along with stresses and strains in the deformed samples. Scanning Kelvin probe force microscopy (SKPFM) is applied to investigate the efficiency of the charging process thanks to the design of the H-cell that offers compatibility with this analytical method.

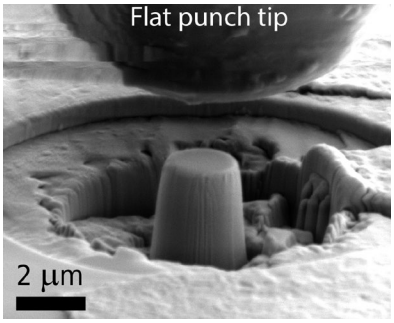

Figure 1 Micropillar before compression.

References:
[1] S. Wang, S. Kalácska, X. Maeder, J. Michler, F. Giuliani, T. B. Britton, The effect of δ-hydride on the micromechanical deformation of a Zr alloy studied by in situ high angular resolution electron backscatter diffraction, Scripta Materialia 173 (2019) 101-105. doi: 10.1016/j.scriptamat.2019.08.006
[2] S. Kalácska, Z. Dankházi, G. Zilahi, X. Maeder, J. Michler, P. D. Ispánovity, I. Groma, Investigation of geometrically necessary dislocation structures in compressed Cu micropillars by 3-dimensional HR-EBSD, Materials Science and Engineering A 770 (2020) 138499. doi: 10.1016/j.msea.2019.138499
[3] P. D. Ispánovity, D. Ugi, G. Péterffy, M. Knapek, S. Kalácska, D. Tüzes, Z. Dankházi, K. Máthis, F. Chmelík, I. Groma, Dislocation avalanches are like earthquakes on the micron scale, Nature Communications 13 (2022) 1975. doi: 10.1038/s41467-022-29044-7
[4] J. Kim, C. C. Tasan, Microstructural and micro-mechanical characterization during hydrogen charging: An in situ scanning electron microscopy study, International Journal of Hydrogen Energy 44 (12) (2019) 6333-6343. doi: 10.1016/j.ijhydene.2018.10.128

Acknowledgement:
This research was funded by the French National Research Agency (ANR) under the project No. "ANR-22-CE08-0012-01" (INSTINCT).