In situ micromechanical investigation of solids under extreme conditions (*INSTINCT*)

Context:

Micro- and nanomechanical testing is a novel emerging field in materials science. Countless research groups have already thoroughly investigated deformation of bulk materials, where the smooth deformation is enabled by the presence of dislocations in large numbers inside the samples. Materials' response to external deformation fundamentally differs from bulk as the sample size is reduced. During compression of these so-called micro- and nanopillars, deformation happens in localized, distinctive slip bands that create inhomogeneous surface features [1]. These features (also referred to as slip bands) appear as a result of intermittent, sudden strain bursts, that produce jerky, stochastic stress-strain curves. It has been determined that the yield strength exhibits inverse dependency on the size when micropillars were examined in this regime (also referred to as "size effect").

If hydrogen is present in the solid (i.e. by wet electrochemical processes, introduced during manufacturing / environmental exposure or after contact with high pressure gaseous H), 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 [2]. To gain knowledge on how the presence of hydrogen modifies dislocation nucleation and mobility, interactions between phase and grain boundaries, *in operando* experimental investigations require high spatial resolution.

The objective of this post-doctoral work is to develop new methodologies for measuring the micromechanical response of solids under extreme conditions by exploiting FIB micromachining for the manufacture of dedicated specimens. The fellow will aims at paving the way towards mechanical characterization of materials subjected to extreme environmental conditions at small scales. These extremities include high strain rates (HSR, up to 10^4 s^{-1}) and temperatures varying between cryogenic (down to -150°C) up to medium ranges (room temperature to ~400°C). In particular, the work aims to study materials' characteristics in the hydrogen context.

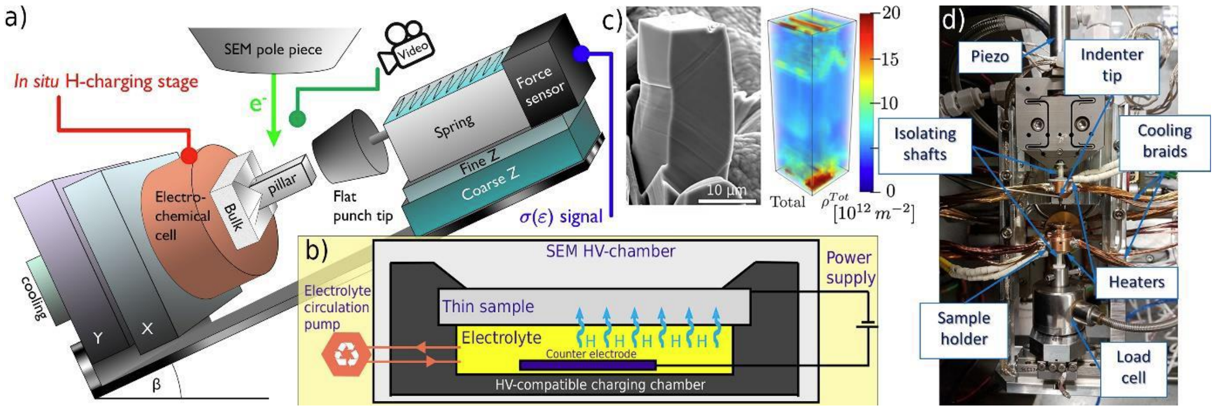

Figure 1. a) Experimental setup for the H-charging cell coupled with the HSR nanodeformation stage. The sketch shows disproportionately large pillar size (≤10 μm) compared to the size of the whole setup (~15 cm). **b)** Design of the *in situ* H-charging cell. **c)** Experiment / simulation comparison of a micropillar [3]. **d)** Cryogenic stage.

Summary of work:

1. Participation in the development of the H-charging cell. The fellow will contribute to the rigorous testing of the developed *in situ* H-charging cell, that can be operated under ambient and vacuum conditions, simultaneously with the nanodeformation stage.
2. Development of micromechanical tests of the alternating tension/compression and fatigue type on the micron scale to study surface layers (i.e. thin films). This work will require the micromechanical

device of Mines Saint-Etienne, where current developments allow the deployment of high-frequency tests compatible with cyclic stresses of compression/traction/fatigue types.

3. Application to surface layers. The objective will be to apply all the developed methods on materials of interest to project *INSTINCT* (i.e. materials subject to hydrogen embrittlement, coatings). From an academic point of view, the objective will be to study the correlation between the state of residual stress, the microstructure and behaviour of the material in compression, bending, traction and fatigue.

General informations :

Place: Saint-Étienne (Laboratoire Georges Friedel, CNRS UMR 5307, Mines St-Etienne)
Funding: ANR-22-CE08-0012-01 (*INSTINCT*)
Scientific manager: Szilvia KALACSKA
Type of contract: post-doctoral fellow (experience < 2 years)
Duration: 24 months
Start date: 1st November 2024
Salary (gross): ~2800 €/month

The coaching team: The experimental developments will be carried out at the LGF Laboratory (Mines Saint Etienne) under the direction of Szilvia Kalacska, Frederic Christien and Guillaume Kermouche. The machining work by FIB will be carried out in the premises of Manutech-USD (St. Etienne).

The Host Institution: The research groups at LGF (SURF and PMM) are experts in the fields of metallurgy, mechanics of materials and surface functionality (i.e. against corrosion). They are committed to designing the new generation of metallic materials in relation to the use of new manufacturing processes and surface treatments. The LGF laboratory is involved in the Manutech network, which aims to put the Lyon-St-Etienne region at the forefront of surface manufacturing and tribology.

What we offer: Cutting-edge training in mechanics and materials; a stimulating and enriching research and teaching program, a large international network with the best scientists in the field (EMPA-Thun, MPIE, KIT Karlsruhe, …). The fellow will have access to the latest micromechanical equipment (high/low temperature modules, ultra-highspeed deformation) coupled with versatile characterization (*in situ* high (angular) resolution EBSD [3-5], *in situ* Acoustic Emission [1]) techniques, and a dual beam FIB/SEM system dedicated to micro-sample preparation. In case of an early start, the fellow can participate in a large-scale facility measurement at the ESRF ID11 (Feb. 2024) to study hydrogen-related effects in porous stainless steel samples.

Requirements:

- PhD degree, or (be close to acquiring it) in mechanics/materials
- Skills in mechanics of materials and mechanical testing, electron microscopy characterization
- Affinity for numerical simulation and programming
- Good level of English
- Ability to work in a team
- Spirit of synthesis / editorial quality

The candidate must provide a **detailed CV, a motivation letter, a PhD certificate** (or the expected defence date)**, the PhD Thesis with a list of publications, a letter of recommendation and a "research sketch"** (proposing your ideas on the desired research within the framework of *INSTINCT*, in max. 1 paragraph)**.**

Contacts :

Szilvia KALACSKA, CNRS, LGF St-Etienne, szilvia.kalacska@emse.fr, www.micromechanics.fr
Frederic CHRISTIEN, LGF Mines St-Etienne, frederic.christien@emse.fr
Guillaume KERMOUCHE, LGF Mines St-Etienne, kermouche@emse.fr

References :

[1] P.D. Ispánovity, D. Ugi, G. Péterffy, D. Tüzes, S. Kalácska, Z. Dankházi, M. Knapek, 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

[2] J. Bestautte, S. Kalácska, D. Béchet, Z. Obadia, F. Christien, Investigation of quasi-cleavage in a hydrogen charged maraging stainless steel. *Corrosion Science*, 218 (2023) 111163. DOI : 10.1016/j.corsci.2023.111163

[3] K. Zoller, S. Kalácska, P. D. Ispánovity, K. Schulz, Microstructure evolution of compressed micropillars investigated by in situ HR-EBSD analysis and dislocation density simulations. *Compter Rendus Physique*, 22:S3 (2021) 267-293. DOI: 10.5802/crphys.55

[4] S. Kalácska, J. Ast, P.D. Ispánovity, J. Michler, X. Maeder, 3D characterization of the plastic zone below the crack tip in tungsten at various temperatures by HR-EBSD. *Acta Materialia* 200 (2020) 211-222. DOI: 10.1016/j.actamat.2020.09.009

[5] D. Ugi, K. Zoller, K. Lukács, Z. Fogarassy, I. Groma, S. Kalácska, K. Schulz, P. D. Ispánovity, Irreversible evolution of dislocation pile-ups during cyclic microcantilever bending. ArXiv preprint: https://doi.org/10.48550/arXiv.2306.08262