Size effects on the mechanical properties of polycrystalline materials in oxidative or inert atmospheres

PhD offer : **ERC-SG_HT-S$_4$DefOx#7**

Context :

Structural metallic/intermetallic materials operating at **high temperatures** (650°C-1200°C) in severe environments are commonly subjected to in-service **surface reactivity, *i.e.* oxidation, corrosion**. This issue is encountered in several industrial applications, especially when high temperatures, mechanical stresses, and highly corrosive atmospheres gather (power plants, aeronautic turbines, etc.)[1]. Environment-assisted degradation alters both the surface of the materials and their bulk properties due to a progressive selective consumption of elements involved in the surface degradation process and/or diffusion of oxidising elements (*e.g.* depleted sub-surface layer for Ni-based superalloys due to Al consumption to form Al_2O_3 [2, 3], brittle oxygen/nitrogen-enriched layer in Ti and TiAl alloys due to O and N solubility [4, 5], etc.). The material in a shallow region beneath the reactive surface subsequently presents a gradient of chemistry, microstructure and physical properties. **This gradient of microstructure and properties evolves with the time due to the oxide growth and diffusion processes.**

Each family of metallic/intermetallic materials reacts differently to the so-called "stress-corrosion", but also to the corrosive/oxidative-deformation. However, all the materials are potentially affected by these mechanisms due to the concomitant effects of surface reactivity, microstructure evolution and deformation. **Despite the negligible scale of the physical, chemical, metallurgical gradients (from 0.1 to 100 micrometres beneath the surface) in comparison with the dimensions of the structural components, the variability in mechanical behaviour within the gradient often drives premature damage and the progressive rupture of the component** [6]. Such material evolution and degradation could be included in the so-called "***stress-corrosion cracking***", investigated for decades for all the structural materials used at high temperature. **HOWEVER**, industrial and ecological motivations to use structural materials in ever-more extreme, severe, harsh conditions push them to their performance limits. **The synergy operating between inter- versus intragranular strain localisation and surface reactivity/diffusion processes promotes unexpected damage and high variability in lifespan of structural components exposed at "too high temperatures – too much (cyclic and/or steady) stresses"** [7]. A better understanding of the thermo-mechano-chemical elementary mechanisms responsible of early damage at the microscale is needed. Advanced characterisation techniques such as high resolution digital image correlation techniques [8–11] and topotomography [12–14] have recently demonstrated a great potential to study plastic slip localization at the length scale of the crystallographic grain and can be used to tackle this topic.

Project motivations :

To address this point, HT-S$_4$DefOx, a project funded by the European Research Council (ERC - Starting Grant), intends :

- To assess the mechanical behaviour within the time-evolving gradient of microstructure and properties, *i.e.* within the "sub-surface" material (micro- and mesoscale approach);
- To assess the variability in sub-grain mechanical behaviour of the metallic material at the metal/oxide interface (microscale approach). This interface, considered as the "extreme surface"; is on the front line for the thermo-mechano-chemical coupling;
- To model and simulate thermo-mechano-chemical coupling on time-evolving microstructures and properties of the "sub-surface" material with boundary conditions on the "extreme surface".

References :

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14. Stinville JC, Ludwig W, Callahan PG, et al (2022) Observation of bulk plasticity in a polycrystalline titanium alloy by diffraction contrast tomography and topotomography. Mater Charact 188:111891. doi:10.1016/j.matchar.2022.111891

Description of the PhD project :

The present PhD project will focus on the experimental investigation of size effects in miniaturized mechanical specimen affected or not by oxidation and confrontation with numerical simulation, in collaboration with another PhD student in this project. « Size effect » in mechanics at the microstructure scale is fundamental to well understand the mesoscopic response of ultrathin product materials, i.e. the mesoscale response of « surface grains » versus « core grains ». The mechanistic understanding of surface effects is crucial for thermo-mechano-chemical coupling. The mechanical response of small-sized materials in the presence of a free-surface versus an oxidised surface (dense, continuous and adhesive oxide) is expected to behave differently due to change in confinement for dislocations emergence or dislocations pile-up at the metal/oxide interface, respectively. Further investigations are needed to dissociate the different alterations due to surface reactivity (oxide and “sub-surface” affected material) compared to materials evolving in inert atmospheres (free surface and homogeneous material). In the presence of a free-surface, microtensile testing performed on specimens with various thicknesses shows a softening of the mechanical strength of the material with the thickness decrease. This transition in mechanical strength is denoted the “polycrystalline to multicrystalline transition”. Surface effects, i.e. the loss of confinement of the “surface grains” versus “core grains”, is attributed to a lower work-hardening component due to dislocation escape from the free-surface, thus limiting dislocation dynamics and activation of additional slip systems; Therefore, the mechanical behaviour of the “surface grains” must be different during materials identification. In oxidative atmospheres, the oxide could act as an interface, thus as a barrier to dislocation escape/motion. However, change in sub-surface microstructure and properties was generally found responsible of size-dependent mechanical response. In addition to “visible/verifiable” materials evolution such as depleted-zone in precipitation strengthened materials, gradient of hardness due to oxygen-insertion, “invisible/unverifiable” materials evolution such as vacancies injection/annihilation, condensation need to be considered due to coupling effect with plasticity and diffusion processes. The goal of the present PhD project is to identify the mechanical behaviour of “surface grains” versus “core grains” in the presence of a free-surface or a metal/oxide adherent interface and zone affected by oxidation.

In the present project, the PhD student will :

- Perform mechanical tests on miniaturised specimens combined with different apparatus, i.e. conventional tensile and fatigue tests, in-situ scanning electron microscope or laser scanning confocal microscope, in-situ synchrotron , ex-situ synchrotron;
- Identify the mechanical behaviour of miniaturised specimens and integrate size effect based on microstructural parameters in a crystal plasticity model ;
- Characterise intragranular slip events and intergranular deformation using high resolution digital image correlation techniques ;
- Characterise intragranular slip events and intergranular deformation using topotomography techniques or nanotomography under synchrotron radiation;
- Characterise dislocation structures in the oxidation-affected region and non-affected region using post-mortem TEM analyses and/or in-situ TEM testing to test the validity of the crystal plasticity model integrating size effects ;
- Characterise strain-localisation- assisted oxides using HR-DIC and nanotomography under synchrotron radiation;
- Develop and identify a model describing the formation and growth of strain-localisation- assisted oxides.

Expected skills and/or know-how :

The PhD student should have the following skills and/or know-how :

- Mechanical engineering (and more particularly at the microscale) ;
- Material sciences and/or computational solid mechanics ;
- Surface reactivity ;
- Metallographic characterisation skills ;
- Image analyses ;
- Scientific computing (Matlab and/or Python language, etc.).

Administrative details :

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PhD locations :

> **INSTITUT CLEMENT ADER**
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> *(there is a shuttle bus between Paris Sud and the laboratory)*

AND

> ***Experiments at SOLEIL and ESRF***