**Acronym: OXIMAS**
**Title: OXIdation resistant high-entropy films obtained by MAgnetron Sputtering for energy applications**

**Directors:**
Mohamed EL GARAH, Research professor at UTT, mohamed.el_garah@utt.fr
Frederic SANCHETTE, Full Professor at UTT, frederic.sanchette@utt.fr
University of Technology of Troyes (UTT), LRC CEA-LASMIS, Nogent International Centre for Coating Innovation (NICCI), Pôle Technologique de Sud – Champagne, 52800 Nogent, France.

**Subject:**
High-temperature oxidation significantly affects many materials, resulting in high economic losses. The development of materials resistant to high-temperature oxidation can therefore improve component performance and lifetime. In this context, the development of high-entropy thin-film alloys with superior properties is of a great interest and should be pursued in many applications, especially for applications in the field of low-carbon energy.

This project aims to develop innovative films capable of resisting oxidation in harsh environments. We have begun work focusing on how to improve the oxidation resistance of high-entropy refractory alloys at high temperatures (800°C) [1-3]. Encouraging results show that it is possible to attenuate oxygen diffusion in this type of material. Nevertheless, the oxidation mechanism needs to be studied in depth to gain a better understanding of the phenomenon and propose a solution with improved resistance ($T > 1100$°C). Oxidation measurements will be carried out at LASMIS, Nogent Antenna, using Thermogravimetric Analysis (TGA) under atmospheric conditions.

To address these challenges, we adopt an experimental approach that first aims to control the architectural, micro- and nano-structural properties of new high-entropy coatings. In this study, new alloys based on chromium and silicon will be proposed. The first step will be to synthetize high-entropy thin-film by magnetron sputtering technique (conventional as well as HiPIMS). The aim is to optimize processing parameters and control composition. We are therefore interested in the synthesis of nanostructured materials (2D and 3D architectures). With regard to nanostructured films, other materials in addition to high-entropy alloys will be developed. The resulting materials will then undergo physicochemical characterization and functional performance evaluation.

- Material processing

Materials will be developed using Physical Vapor Deposition (PVD) techniques, in particular magnetron sputtering (conventional and HiPIMS). Deposition parameters will be optimized and relationships will be established between structural and micromechanical properties. Magnetron sputtering reactors at LASMIS, Nogent antenna, will be used to develop these materials.

- Characterization and Resources (LASMIS)

Several skills and resources will be used to achieve the objectives of **OXIMAS** project. LASMIS is specialized in mechanics and materials engineering. It has several technological resources (4 PVD machines, high-temperature furnaces and characterization tools such as MEB-FEG, ATG/DSC, DRX, nano-indentation, etc.). The nanostructure will be studied using transmission electron microscopy (TEM) at the *Institut Jean Lamour*, which is specialized in materials science: materials, metallurgy, plasmas, surfaces, electronics and nanomaterials.

- Methodology and techniques

This work presents a study of new generations of potential materials for applications that can operate under extreme environments. Coatings will be obtained by magnetron sputtering and the deposition parameters will be linked to the physicochemical characteristics (structure, microstructure, morphology) and functional properties, in particular resistance to high temperatures. The influence of architecture (2D and 3D nanostructuring) will be particularly studied. Understanding the phenomena must be the subject of particular attention, particularly with regard to the oxidation mechanisms depending on the coating architecture.

- Scientific and technological objectives

There are mainly two objectives. The first is to develop process-material couples in order to obtain coatings which can provide solutions for resistance to harsh environments in terms of temperature and aggressive environments in the fields of energy (nuclear, hydrogen production).
The second is the understanding of the degradation phenomena of complex alloys depending on the architecture (2D and 3D nanostructuring), based on multiscale analyses. This project is part of the accelerated development of materials for energy initiated within the CEA-UTT NICCI (Nogent International Center for Coating Innovation) Corresponding Research Laboratory set up in 2012.

- Results and potential scientific impact

This work can lead to the publication of the results as articles and patents. Presentations will be given in international and national conferences. Moreover, the optimization of deposition processes should enable the development of robust techniques for the production of high-entropy thin films with advanced functional properties. In this context, a technology transfer could be envisaged. Beyond the technological spin-offs, the potential results are likely to be of interest to the scientific community. Generic knowledge of high-entropy materials and their performance in extreme environments could be established.

- Next steps

The aim here is to deposit high performed films on large size substrates. To achieve this objective, an industrial facility is needed, and the Nogent lab aims to acquire an industrial PVD/PECVD hybrid reactor. This reactor would be installed in the branch's premises in 2024 or 2025. This tool should enable us to carry out process up-scaling under optimum conditions.

**References**

[1] M. El Garah, D. Soubane, F. Sanchette, Review on mechanical and functional properties of refractory highentropy alloy films by magnetron sputtering. emergent mater., 2023. https://doi.org/10.1007/s42247-023-00607-8

[2] D. E. Touaibia, S. Achache, A. Bouissil, J. Ghanbaja, S. Migot, M. A. Pour Yazdi, F. Schuster, B. Panicaud, F.Sanchette, M. El Garah, Oxidation resistance and mechanical properties of AlTiZrHfTa(-N) high entropy films deposited by reactive magnetron sputtering.Journal of Alloys and Compounds, 2023, 969, 172397

[3] A. Bouissil, S. Achache, D. E. Touaibia, B. Panicaud, M. A. Pour Yazdi, F. Sanchette, M. El Garah. Properties of a new TiTaZrHfW(-N) refractory high entropy film deposited by reactive DC pulsed magnetron sputtering, Surf. Coat. Technol., 2023, 462, 129503.