



Centre of Excellence in Multifunctional Materials  
for Industrial and Medical Applications

A close-up, high-angle photograph of a microscope's internal components, showing a circular lens assembly and various mechanical parts. The image is overlaid with a semi-transparent blue circular graphic.

RESEARCH BROCHURE 2023

**FUNCTIONAL PROPERTIES  
OF MATERIALS**



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# GENERAL SUMMARY BY PROF. ŁUKASZ KURPASKA

## RESEARCH GROUP LEADER: FUNCTIONAL PROPERTIES OF MATERIALS

The functional Properties group comprises 6 Ph.D. students, two postdocs, and one senior scientist responsible for conducting X-ray diffraction experiments and data analysis. The work performed in the frame of the Functional Properties Group focuses on understanding and explaining the processes taking place in construction materials under the influence of radiation and high temperature. The essential features of the planned research strategy are the following:

- Conducting research on materials subjected to high temperatures and radiation using ion beam sputtering as a surrogate for neutron radiation
- Integration of the results from the tests conducted using structural and mechanical methods, which allows a precise description of the functional properties of the materials
- Multi-scale mechanical tests, including extrapolations of the obtained results from the nano to the micro-scale, contribute to the prediction of the material properties in the real industrial installations
- A multi-material approach that covers the study of structural elements used in the nuclear reactor (e.g., fuel claddings, materials for the pressure vessel in the primary cooling circuit), chemistry sector, and radiopharmaceutical business
- Development of competencies in conducting standardized tests so expertise for SMB or large industries can be offered
- Performing high-temperature in-situ studies (mechanical, structural, and thermal) with the aiming of establishing CoE as a pioneer in this subject in Poland

To assure the group's sustainability and develop multinational collaboration with other leading institutions, each member participates in preparing at least one proposal per year. By doing so, we are gaining competencies in project drafting and, later, its management. These activities resulted in receiving several grants and awards. Among many, one can name the Polonez Bis grant received by dr Wenyi Huo, the Prelludium funding received by MSc Edyta Wyszowska, or the Fulbright stipend received by dr Lukasz Kurpaska. We are also active in acquiring time on large infrastructures. Recently a proposal to RADIATE and EMIRa platforms was prepared and submitted. At the same time, dr Sri Tapasvi Nori has been granted two weeks on advanced TEM microscopes in the Norwegian Centre for Transmission Electron Microscopy via the ESTEEM3 platform.

The samples' characterization using an SEM microscope equipped with EDS and EBSD detector is crucial for the group. This equipment is also used to prepare TEM lamellas, which are later observed at the Warsaw University of Technology or Physics Institute. In addition to the structural characterizations, the group has continued assessing the materials' mechanical properties using the nanoindentation technique. This tool has significantly helped to increase the amount of information related to the hardening effect, plasticity, and yield point of pristine and ion-irradiated material. First High-Temperature in-situ XRD and Raman tests have been performed. These tests were done at temperatures up to 1000 °C, which is relevant to the operational conditions of the next-generation nuclear reactors. Finally, significant effort has been made to strengthen collaboration with numerical groups of NOMATEN CoE (headed by prof. Alava and Papanikolaou). This collaboration resulted in the publication of two articles in prestigious journals: *Materials* and *Design and Physics Review Materials*. In conclusion, the group published 10+ papers, and many more are under evaluation. It is expected that by the end of the project, we will be able to compete for external funding successfully.

## SCIENTIFIC DEVELOPMENTS

Prof. Kurpaska's group managed to develop and establish precise research subjects and is gaining competencies centered around several different areas: i) investigation of mechanical and structural properties of PLD manufactured amorphous alumina coatings (Ph.D. student: Agata Zaborowska), ii) manufacturing of high entropy alloys via powder metallurgy techniques combined with Spark Plasma Sintering (SPS) and Arc Melting (AM) technique (Ph.D. student: Artur Olejarz) and iii) studying the impact of radiation damage on functional properties of High Entropy Alloys (HEAs) (Post-doc: Wenyi Huo), iv) studying and understanding how specific features of Fe-Cr alloys - model alloys for ferritic/martensitic (F/M) steels - influence the microstructural changes induced by neutron surrogate - ion irradiation, to identify the features that mainly cause low-temperature hardening and subsequent embrittlement in F/M steels. This will be done by combining a microstructural examination of ion-irradiated materials with nanomechanical data (Ph.D. student: Katarzyna Mulewska, v) to understand the impact of radiation damage on mechanical and structural properties of newly developed HEA-ODS alloys (post-doc Sri Tapaswi Nori), vi) the understanding impact of temperature



Figure: Arc melting device at NOMATEN. It will allow melting samples of up to approx. 200 g at temperatures up to 3500°C





Figure: XRD diffractometer operated by senior scientist Maciej Zieliński PhD

and radiation damage on corrosion kinetics (under high-temperature water vapor conditions) of model cladding material - pure zirconium. The goal of this work is to investigate the behavior of commercially used in nuclear reactors zirconium-based alloys (E110, Zircaloy 2 and 4) (Ph.D. student: Kinga Suchorab), vii) and investigate the impact of the nanoprecipitate chemical composition on structural and mechanical properties of Oxide Dispersion Strengthened (ODS) steels (Ph.D. student: Małgorzata Frelek-Kozak) and finally viii) study the impact of radiation damage on functional properties of NiFe single crystals (Ph.D. student: Edyta Wyszowska).

Proposed experimental research topics are implemented in close collaboration with numerical groups. For example, understanding of nanoindentation is done with Alvarez, Papanikolaou, and Alava, HEA compositional search with Alava and Papanikolaou, while Raman studies

are performed in collaboration with Joźwik group. Close cooperation with local specialists from TEM (Chromiński) and XRD (Zieliński) is being managed throughout all experimental works. Some of the described Ph.D. and post-doc topics are conducted at PP facilities (for example, HR TEM at VTT or ion irradiation and mechanical tests at CEA). It is expected that this work will result in establishing robust and long-lasting collaboration in nuclear materials investigation with project partners and preparing project proposals for the new Horizon Europe perspective.

# SELECTED PAPERS ABSTRACTS

## Effect of milling time on the microstructure and mechanical properties CoCrFeNi High-Entropy Alloy

Paper accepted for publication in Journal of Alloys and Compounds, prepared by Artur Olejarz et al.

High-entropy alloys are extensively studied due to their very promising properties. However, nowadays, manufacturing methods used to prepare HEAs are complicated, costly, and likely non-industrially scalable. This limits their evolution and poses questions regarding the material's applicability in the future. Considering the abovementioned point, we developed a novel methodology for efficient HEA production using a low ball-to-powder ratio (BPR). Using different milling times, we manufactured four HEA powder precursors using a BPR of 5:1 that were later sintered via the Spark Plasma Sintering technique. Microstructural characterization was done by optical microscopy, Scanning Electron Microscopy equipped with EDS and EBSD detectors, and X-ray diffraction. Mechanical properties were measured using nano and microhardness techniques. In this work, we follow the structural evolution of the material and connect it with the strengthening effect as a function of milling time. Further, we discuss the impact of different sintering and annealing conditions, proving that HEA characterized by high mechanical properties may be manufactured using low BPR.

*Figure: an operating XRD diffractometer at NOMATEN*

## Absolute radiation tolerance of amorphous alumina coatings at room temperature

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This study assessed the structural and mechanical properties of a one  $\mu\text{m}$  thick  $\text{Al}_2\text{O}_3$  coating deposited on 316L stainless steel by Pulsed Laser Deposition (PLD), subjected to high energy ion irradiation. Mechanical properties of pristine and ion-modified specimens were investigated using the nanoindentation technique. A comprehensive characterization combining Transmission Electron Microscopy and Grazing-Incidence X-ray Diffraction provided deep insight into the structure of the tested material at the nano- and micro-scale. Variation in the local atomic ordering of the irradiated zone at different doses was investigated using a reduced distribution function analysis obtained from electron diffraction data. Nanoindentation measurements revealed a slight reduction in the hardness of all irradiated layers. At the same time, TEM examination indicated that the irradiated layer remained amorphous over the whole dpa range. No evidence of crystallization, void formation, or element segregation was observed up to the highest implanted dose. Reported mechanical and structural findings were critically compared with each other, concluding that alumina coatings exhibit excellent radiation resistance under given irradiation conditions over the whole range of doses used. Obtained data strongly suggest that investigated material may be a promising candidate for next-generation nuclear reactors, especially LFR-type, where high corrosion protection is one of the highest prerogatives to be met.





## FUTURE PLANS

In the long term, Functional Properties Group plans to establish itself as a leading unit in in-situ high-temperature studies. That includes mechanical properties evaluation using small-scale samples in the tensile test (elongation measured not by standard extensometer but DIC option), in-situ high-temperature Raman spectroscopy, and XRD analysis.

We also plan to develop unique competencies in high-temperature nanomechanical studies. This will allow us to understand the mechanical properties of ion-irradiated materials (devoted to nuclear applications) under their operating conditions. Finally, we are developing competencies in thermal properties evaluation, especially

TG/DTA or dilatometry. All this data should, of course, be confronted with the structural analysis done by SEM and TEM. This is done in collaboration with the Józwick group. For example, Eurofer 97, which is planned to be used for the next generation nuclear reactors, is poorly described at temperatures over 300 C. At the same time, there is almost no data on ion-irradiated mechanical properties of this alloy measured in-situ above this temperature. Some of the studies mentioned above have started already. We investigated the amorphous alumina phase transition and recorded changes in the early stages of the zirconium alloy corrosion process. Understanding this phenomenon may result in controlling this detrimental process, thus limiting its impact and extending the life of the cladding material (increasing burnup of the fuel = increasing efficiency of the energy production).



# FUNCTIONAL PROPERTIES OF MATERIALS

