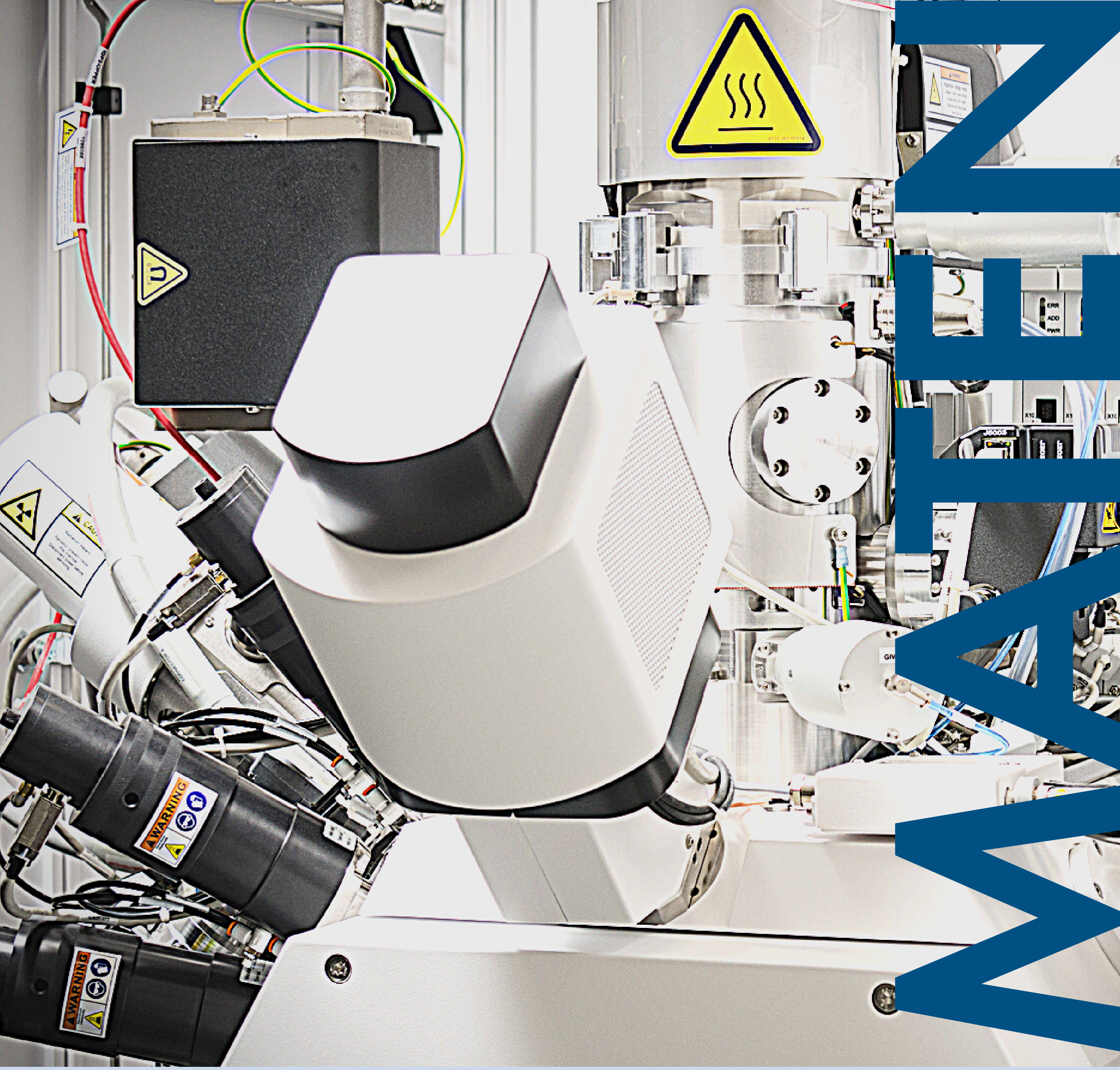

NOMATEN

RESEARCH BROCHURE 2023

MATERIALS CHARACTERIZATION

NOMATEN

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GENERAL SUMMARY

IWONA JÓŹWIK PhD

RESEARCH GROUP LEADER: MATERIALS CHARACTERIZATION

The main goal of the group is to design and execute experiments focused on advanced characterization, analysis and development of advanced multifunctional materials in close collaboration to other RGs and Project Partners using a wide range of techniques, including SEM/ FIB/EBSD/EDX tools, TEM (Transmission Electron Microscopy) analysis, as well as advanced and in-situ X-ray diffraction (XRD) and Raman spectroscopy. Significant part of the research conducted is realized in close collaboration with Functional Properties and numerical modelling Groups of NOMATEN CoE.

Part of the research studies is being conducted using Helios 5 UX (Thermo Fisher Scientific) (Fig. 1) – a fully digital, Extreme High Resolution (XHR) Field Emission Scanning Electron Microscope (FE SEM) equipped with Focused Ion Beam (FIB) technology, EDS (Energy Dispersive X-ray Spectroscopy) and EBSD (Electron Backscatter Diffraction). It allows fast characterization of nanometer-sized details and analysis in 2D, elemental analysis (qualitative and quantitative), crystallographic analysis and best in class sample preparation and flexible nanoprototyping by focused ion beam (Fig. 2).

Fig.1. Helios G5 UX by ThermoFisher Scientific at the NOMATEN laboratory

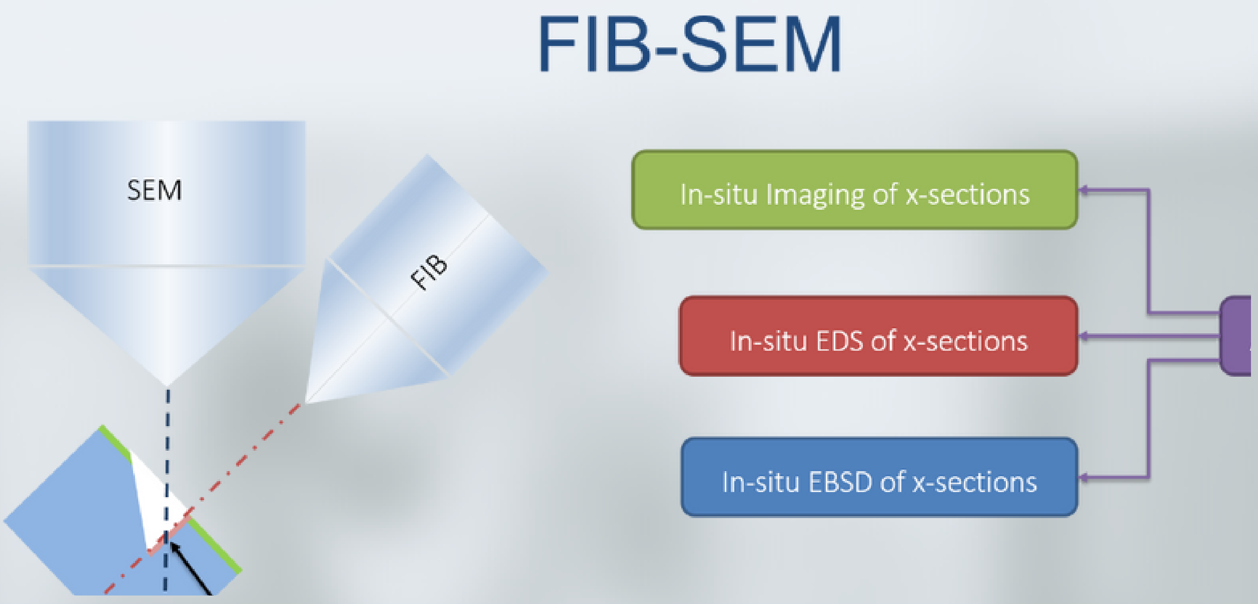


Fig. 2. Schematic representation of the dual-beam concept (FIB-SEM)

The topics scientific research activities of the Group are as follows:

- mechanical and electrical properties of polymer materials exposed to ionizing radiation;
- detailed characterization with Raman imaging of purity and degree of order of various types of graphitic materials intended for application in HTGR technology; in-situ high temperature studies of structural evolution of graphite, surface and in-depth distribution of defects;
- mechanically driven structural changes in model materials and impact of radiation damage;
- in-situ Raman spectroscopy study of the phase transformations in materials in broad range of temperature;
- XRD study of the stress/strain and phase transformations in materials in broad range of temperature.
- structural studies of irradiated Fe, alumina coatings, HEA (NiFe binary systems) – SEM, EBSD, TEM, XRD, Raman to understand accumulation of radiation damage;
- SEM/TEM study of the tensile/stress influence on the irradiation defect formation in materials;
- in-situ SEM/EBDS studies of materials deformation;
- study of plastic deformation in Ni-based alloys with application of SEM-FIB-EBSD 3D reconstruction.

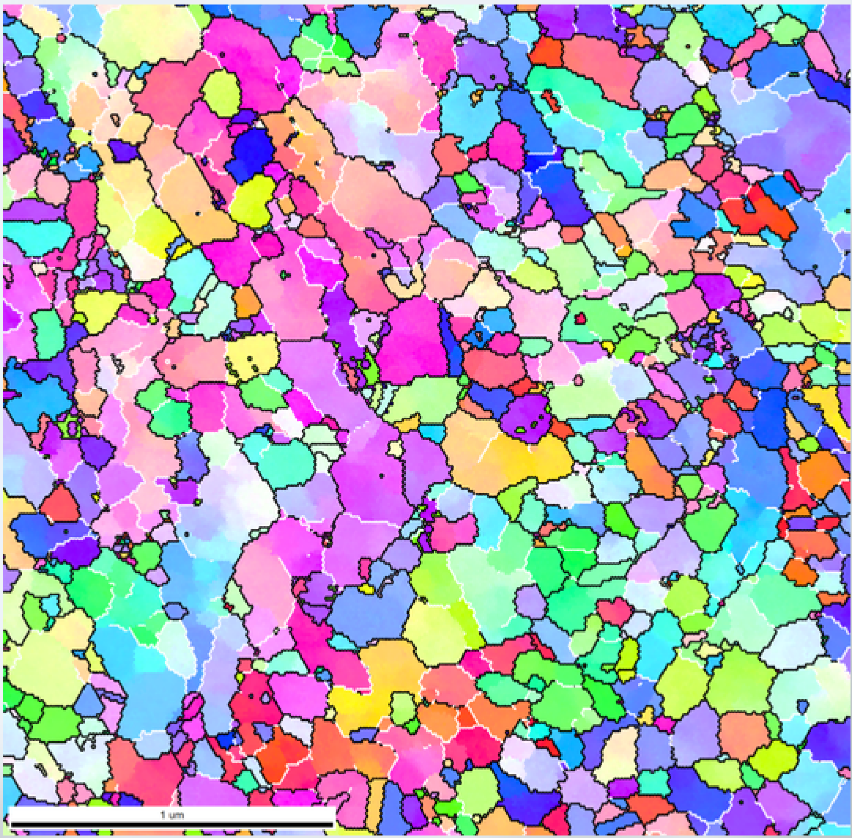


Fig. 3. EBSD orientation imaging microscopy map of HEA obtained in transmission mode (Transmission Kikuchi Diffraction - TKD).

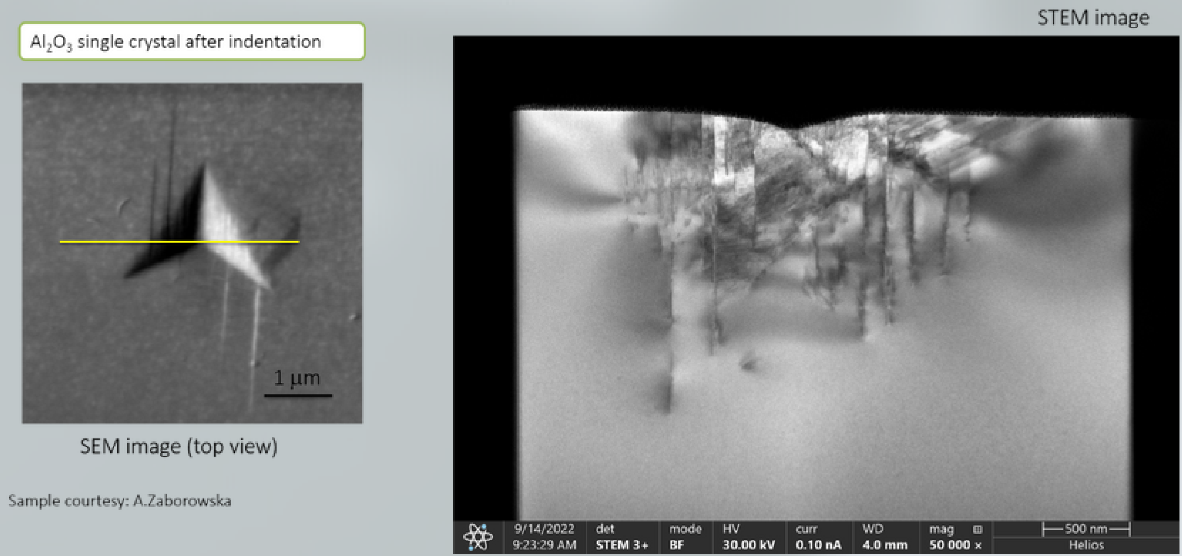
The aforementioned include, among others, the characterization of the microstructure of pure Fe, steels, high entropy alloys (HEA), oxide-dispersed strengthen (ODS) steels and Ni-based alloys, polymers, ceramics and coatings using scanning electron microscopy (SEM) together with the study of the crystal orientation using electron backscatter diffraction (EBSD) and chemical composition by energy-dispersive X-ray spectroscopy (EDS).

Part of the research activities is devoted to the development of electron microscopy techniques in the study the influence of ion irradiation on the mechanical properties of abovementioned materials and also electrical properties in case of polymers (studies of degradation of cable insulations at nuclear power plant environment for the future application of various materials in the electronics industry or in nuclear reactors). The group is also involved in extensive preparation of materials for a transmission electron microscopy (TEM) using the focused ion beam (FIB) technique for subsequent analysis of damage accumulation mechanism in irradiated materials as well as materials deformation processes with the use of TEM techniques.

Case study 1: Al2O3

The example of Al2O3 after nanoindentation test is shown below (Fig. 4). Thanks to the analysis of EBSD patterns line of cut during FIB lift-out was oriented along specified direction so that the TEM analysis visibility criterion has been fulfilled. That assures the advanced TEM analysis of the individual dislocations formed under the indentation mark. The subsequent STEM imaging allowed to visualise the slip traces related to the material deformation under the strain of nanoindenter and also defects emerging in the result of material deformation. Further TEM analysis is needed to recognize the specific defects formed under the nanoindent, however the first step of analysis under SEM provides with the overall information on the damage mechanism and the full-size view of the area affected by deformation which is way smaller during TEM analysis. The technique described above allows one to select the individual nanoindentation marks at specific orientations which makes the TEM analysis much more efficient. It also has unrivalled advantage of site-specific and angle-specific FIB preparation of the TEM foils for subsequent analysis.

Fig. 4. S-TEM (scanning electron microscopy in transmission mode) image of crystalline Al2O3 after nanoindentation test. The site-specific FIB lift-out preparation of the thin foil allowed to obtain the transparent film of the sample at defined line of the cross section. The position of the line of cut was established by SEM imaging of the sample surface and nanoindentation mark (pile-ups and slip traces related to the material deformation are visible in both images).



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Case study 2: graphite

The surface modification of graphite by ion irradiation and in-situ high-temperature studies of phase transformation in various materials in a broad range of temperature by means of Raman spectroscopy are another topics of scientific interests of the Group activities. The studies are focused on Raman analysis of three types of nuclear graphite intended for application in high-temperature gas-cooled reactors (HTGR) – pristine and irradiated with helium and argon ions with various fluence, structural analysis of high-temperature corrosion products of silicon oxycarbide-based materials, phase analysis of TiO2 hybrid (anatase/rutile) coatings on titanium, preliminary study of defects induced in GaN by irradiation and high-temperature Raman analysis of amorphous alumina coatings.

Case study 3: refractory HEA

The development of fusion reactors creates an exceptionally harsh environment for the structural materials of reactors due to higher neutron fluxes, a more corrosive environment, and higher operating temperatures. Further restrictions are imposed by using materials containing only low activation elements, allowing for fast and safe recycling of parts after the removal from the reactor Thus, the aim of the work was to characterize the novel low activation W-Ta-Cr-V refractory high entropy alloy (HEA) fabricated using the magnetron sputtering and to determine the effect of the He irradiation on the microstructure of this material.

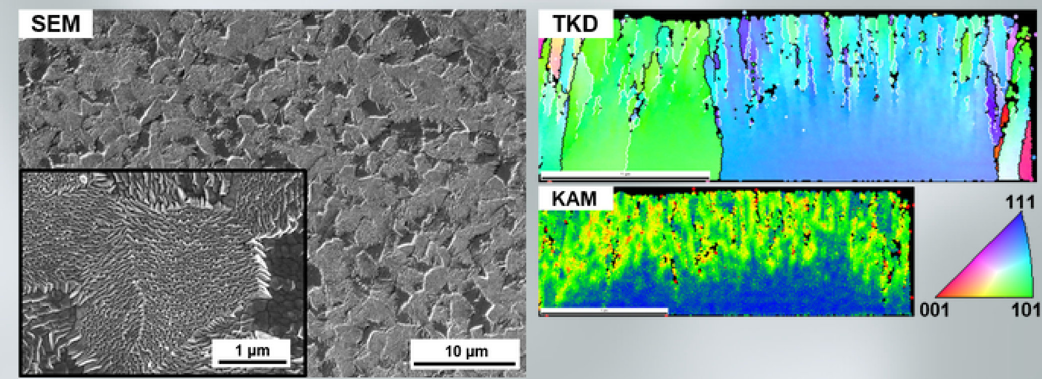

Fig. 5. SEM topography image (left) of the W-Ta-Cr-V HEA surface and Transmission Kikuchi Diffraction (TKD) and corresponding KAM maps collected from the cross sectional sample. The layer initially growth in the form of bigger grains, which change into a columnar nanostructure, as a result of the increasing stress in the material.

The as-deposited W-Ta-Cr-V HEA exhibited a fine microstructure containing the nano-columns with a thickness in the range of 10–50 nm. The orientation map acquired using Transmission Kikuchi Diffraction (TKD) method showed that the layer initially grows in the form of bigger grains however, after the initial stage the refinement of the microstructure occur, probably as a result of substrate-layer structure mismatch and increasing stress in the W-Ta-Cr-V alloy during its growth.

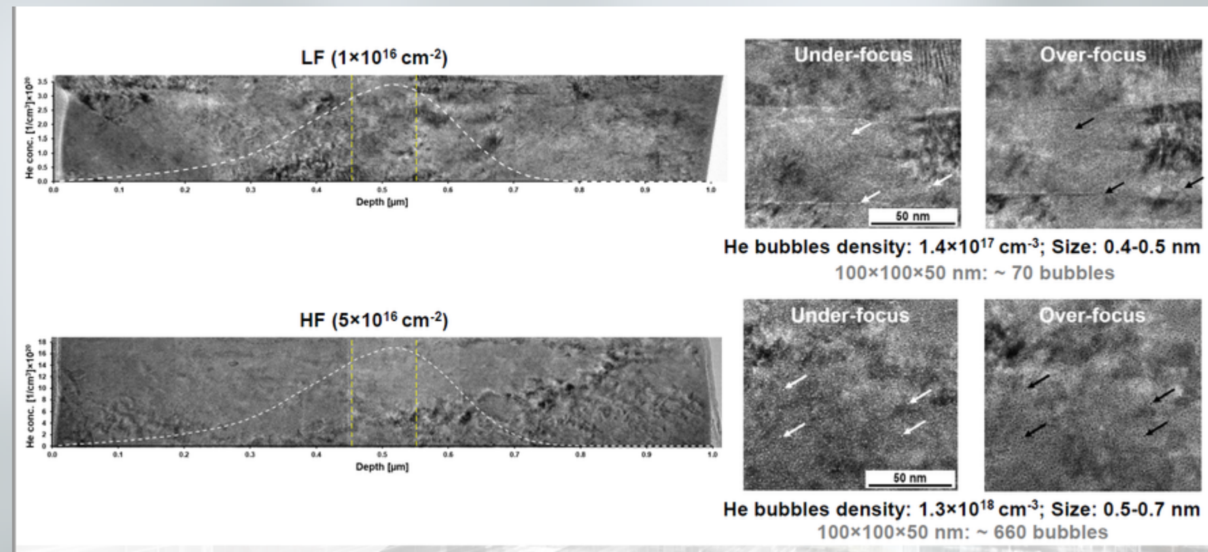

Fig. 6. TEM images of He+ irradiated samples. He bubbles are formed at a fluence of 1e16 cm-2.

The layers of HEA were irradiated with He+ ions of energy of 200 keV. The TEM analysis revealed the presence of the He bubbles of a size of 0.4 – 0.5 nm formed even in the sample irradiated with the fluence of 1×1016 cm-2. The increase the He fluence to 5×1016 cm-2 resulted in the increase of the bubbles size and density. Their maximum density was found at the depth 400 – 600 nm, which is in good agreement with SRIM calculations. To our knowledge the effect of He is unknown as no study is available in the literature devoted to the behavior of He and its coupling with the mechanical properties in these material systems. Therefore, our work aims to couple mechanical and structural properties of the material and address effect of He diffusion in W-based HEA devote to fusion technologies.

Industry collaboration

The Materials Characterization Group has been also involved in collaboration with industrial partners (profile: steel and automotive) to study the microstructure of the specific elements that have undergone production line tests. The data obtained using SEM, FIB-SEM, XRD and Raman spectroscopy techniques have been carefully analysed. The interpretation on the data was used in order to explain the mechanisms of tools degradation during the production processes.

The outcome findings of the studies were/will be implemented as the modification of the production procedures. The high level of satisfaction of the collaborating partners looks promising in terms of future collaboration, tasks and problems that will be undertaken by CoE in the future. The results not only have a direct impact on the project, but also possible impact into the society and economics by an increase of efficiency of the automotive elements production by process optimization and which follows – reduced costs and energy usage

SELECTED PAPERS ABSTRACTS

Structural and chemical changes in He+ bombarded polymers and related performance properties

Journal of Applied Physics 132, 074701 (2022); https://doi.org/10.1063/5.0099137

The paper "Structural and chemical changes in He+ bombarded polymers and related performance properties" presents the effect of He+ ion irradiation of selected polymeric materials: poly(tetrafloroethylene), poly(vinyl chloride), ethylene-propylene-diene monomer rubber, nitrile-butadiene rubber, styrene-butadiene rubber, and natural rubber, on their chemical composition, physical structure, and surface topography. The modification was studied by scanning electron microscopy, Fourier transform infrared spectroscopy, Raman spectroscopy, and differential scanning calorimetry. Irradiation with a high-energy ion beam leads to the release of significant amounts of hydrogen from the surface layer, resulting in an increase in cross-linking that manifests itself by shrinkage of the surface layer, which in turn causes significant stresses leading to the formation of a crack pattern on the polymer surface. The development of microroughness is combined with oxidation. Shallow range of the ions makes the modified layer "anchored" in the substrate via bulk macromolecules, assuring its good durability and adhesion to elasto-plastic substrates. Changes in the surface layer were manifested by the modification of functional properties of the polymers. The hardness of the layer subjected to the ion irradiation process increases even up to 10 times. After modification with the ion beam, a significant decrease in frictional forces was also observed, even up to 5–6 times. The microscopic analysis of wear traces confirmed that the wear resistance also significantly increased. However, ion bombardment of polymeric materials caused a reduction in their mechanical strength (despite the range limited to the surface layer of the order of micrometers) and electrical resistance, which has a negative impact on the possibility of using the materials in some applications

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Effects of Fe atoms on hardening of a nickel matrix: Nanoindentation experiments and atom-scale numerical modelling

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The paper "Effects of Fe atoms on hardening of a nickel matrix: Nanoindentation experiments and atom-scale numerical modelling" describes the significant hardening effect due to Fe concentrations in Ni-based alloys with face-centered-cubic structure that has been studied by using a combined experimental and atomistic-based computational approach via nanoindentation tests. The obtained experimental load–displacement data for the [0 0 1] crystal orientation reached a qualitative good agreement with molecular dynamics simulations results, leading to strong evidence that the main strengthening factors are associated to sluggish dislocation diffusion, reduced defect sizes and the nucleation of tetrahedral stacking faults. Here, interstitial type prismatic dislocation loops mainly formed by ⅙ 〈1 1 2〉 Shockley dislocations are nucleated during the loading process, where their interaction leads to the formation of pyramidal shaped stacking fault which are mainly created by ⅓ 〈1 0 0〉 Hirth dislocations lines. Observing both types of defects coexisting in the same plastic deformation zone by both approaches. Reported mechanical data, measured experimentally and interpreted numerically, are also in accordance with microstructural SEM and TEM investigations.

FUTURE LOOK

The main goal of the group is to conduct advanced characterization of novel multifunctional materials under extreme conditions at the atomistic level using state-of-the art equipment. The structural characterization of the studied materials fills the gap between simulations and functional properties of the material, by verification of the structural model, analysis of material response on various conditions occurring in real environments, analysis of mechanisms of damage accumulation and studies of microstructure influence on the mechanical properties.

An integration between the two already established theoretical/modeling research groups and the experiment focused groups has been achieved by a careful choice of research topics (for example radiation damage studies, nanoindentation studies of advanced materials, comparisons of Raman spectroscopy and XRD studies and associated MD/MC and larger scale models).

In the nearest future the group will focus on studies of damage accumulation in ion irradiated HEA’s and graphite as well as study the structural changes in metallic alloys under tensile/stress, trying to look at the plastic deformation in an atomic level to shed light on the mechanisms of defect production and dislocation nucleation due to external load.