RADIATION RESISTANCE OF MULTICOMPONENT ALLOYS REINFORCED WITH OXIDE NANOPARTICLES UNDER HIGH-ENERGY HEAVY ION IRRADIATION

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Conventional structural alloys, such as ferritic steels, cannot meet the requirements for Gen IV reactor components regarding high operational performance at elevated temperatures and radiation doses [1]. Nowadays multicomponent alloys, specifically medium- and highentropy alloys, are of interest in radiation materials science due to their unique properties, particularly radiation stability, high strength, and corrosion resistance [2, 3]. It is believed that the high level of radiation resistance in multicomponent alloys is achieved due to the high energy barriers for the migration of radiation-induced point defects, resulting from the strong lattice distortions inherent to these alloys. Another area of research is the study of oxide dispersion strengthened (ODS) multicomponent alloys, as this strengthening can significantly enhance mechanical properties [4]. Radiation stability in ODS alloys is improved through the suppression of dislocation motion due to barrier strengthening and better annihilation of point defects at the matrix-oxide particle interfaces. Since dispersed particles determine the high strength characteristics of ODS alloys, the stability of their structure under irradiation is a key issue when considering these alloys as potential reactor materials. One important concern in this regard is the structural stability of dielectric inclusions under exposure to fission fragments. Fuel cladding materials are exposed to fission fragment irradiation, and high ionization energy losses during this process can significantly alter the structure of dielectric materials. Currently, there are no systematic data on the effects of fission fragment irradiation on the structure and properties of multicomponent alloys.

In the present study, the multicomponent ODS alloy CoCrFeNi was irradiated with Xe and Bi ions at energies > 1 MeV/nucleon to induce amorphous oxide particles in the structure. The evolution of the microstructure of the irradiated material, depending on the level of electronic energy loss, was investigated using transmission electron microscopy throughout the entire depth of the damaged layer, employing a specialized layer-by-layer electropolishing

technique. The level of radiation hardening in the damaged layer regions, with varying contributions of radiation defects produced via dense ionization and elastic collisions, was determined.

CoCrFeNi alloys were synthesized using the spark plasma sintering (SPS) method at 980°C at the Institute of Materials Science, Vietnam Academy of Science and Technology. During the synthesis, nanosized yttria oxide particles were added to some of the alloys. The alloys were irradiated at room temperature with 150 and 475 MeV Xe ions and 670 MeV Bi ions to fluences up to 10¹³ cm⁻² using the U400 and IC-100 cyclotrons at FLNR JINR. Foils from different regions of the damaged layer for TEM analysis were prepared using an experimental setup with one-sided electropolishing, which allowed for the removal of the specified thickness of the damaged region from the sample. Thus, foils were prepared from those regions of the irradiated layer where either ionization effects or elastic collisions dominated.

Damage profiles and electronic energy loss for Xe and Bi ions in CoCrFeNi and the regions from which TEM foils were prepared are in fig.1.

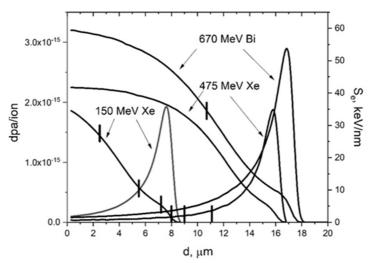


Fig.1. Damage profiles and electronic energy loss for 150 MeV Xe, 475 MeV Xe, and 670 MeV Bi in FCC CoCrFeNi, calculated using SRIM-2013 software. The vertical lines indicate the regions from which TEM foils were prepared

Structural investigations were carried out using TalosTM F200i S/TEM transmission electron microscope with accelerating voltage of 200 kV. The XRD structural analysis was performed at room temperature using an EMPYREAN (PANalytical) powder diffractometer with Cu-Kα radiation. Nanoindentation measurements were conducted using a NanoScan-4D scanning nano-hardness tester (FSBI TISNCM, Russia) with controlled indentation depth.

Typical radiation-induced defects in irradiated materials include dislocation loops, voids, radiation-induced segregation (RIS), and, for FCC materials, stacking fault defects, particularly stacking fault tetrahedra (SFTs), in the presence of an excess vacancy concentration. Previous

studies have shown that in FCC alloys irradiated with neutrons and low-energy ions, the size of stacking fault tetrahedra does not exceed a critical size of a few nanometers [5, 6], with irradiation temperature being the key parameter influencing their formation.

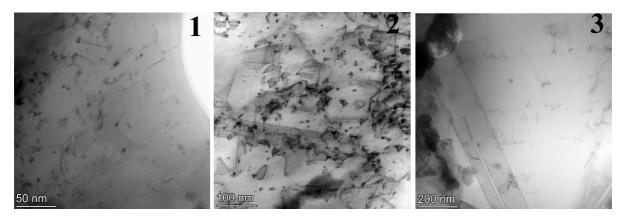


Fig. 2. Microstructure of the metallic matrix of FCC CoCrFeNi at the surface (1) and at depths of 2.5 μ m (2) and 5.5 μ m (4) after irradiation with 150 MeV Xe ions at a fluence of 10^{13} cm⁻²

In the present study, the primary radiation-induced defects in CoCrFeNi alloys after irradiation with high-energy heavy ions were identified as stacking fault tetrahedra (SFTs). Fig. 2 shows the microstructure of the metallic matrix of FCC CoCrFeNi at the surface (1) and at depths of 2.5 μm (2) and 5.5 μm (3) after irradiation with 150 MeV Xe ions to a fluence of 10¹³ cm⁻². It can be seen that stacking fault tetrahedra (SFTs) with sizes reaching several tens of nanometers dominate in defect structure. A rough estimate suggests that their average size correlates with electronic stopping power as their sizes decreases with depth. It is likely that, under high-energy heavy ion irradiation, SF formation are formed due to local thermal heating during ion-target interactions in the presence of an excess vacancy concentration. When an ion impacts oxide particles, this localized heating leads to the formation of latent tracks. Fig. 3 presents TEM images of oxide particle. Latent tracks, with sizes below 10 nm, can be observed within the particle structure.

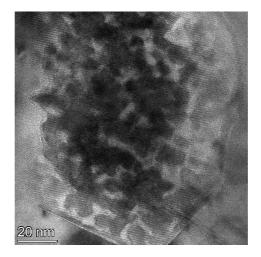


Fig. 3. Oxide particle after irradiation with Xe ions at a fluence of 10¹³ cm⁻²

Radiation-induced defects are the primary cause of radiation hardening and embrittlement of materials. For unirradiated materials, the addition of yttria oxide particles during synthesis increases the alloy's strength by 8 %. Both alloys exhibit radiation hardening, with ODS CoCrFeNi showing greater hardening compared to the alloy without oxide particles. Radiation hardening increases with fluence for both alloys, reaching 16 % for ODS CoCrFeNi, corresponding to a hardness of 5.78 ± 0.23 GPa. Measurements of mechanical properties using nanoindentation were also conducted at various depths from the irradiated surface.

In conclusion, we studied the effects of swift heavy ions on the structure and properties of oxide-strengthened multicomponent CoCrFeNi alloys. It was shown that irradiation leads to the formation of stacking fault tetrahedra, with a correlation observed between defect parameters and the electronic stopping power. Irradiation also results in the formation of tracks in oxide particles and their complete amorphization in the subsurface layer at a fluence of 10^{13} cm⁻². Radiation hardening after irradiation reaches up to 16 %.

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