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Nanomechanical testing of ODS steels irradiated with 1 MeV/amu heavy ions

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Work motivation

ODS steels, reinforced by oxide nanoparticles, are considered to be the most perspective materials for fuel cladding in Generation IV nuclear reactors due to their enhanced radiation resistance and high temperature creep resistance comparably to the conventional reactor steels. One of the key question is the study of fission fragment impact on the structure of the nanoparticles and mechanical properties of ODS steels in general.

Nanoindentation techniques are the only possible method for investigation the mechanical properties of materials after exposure to the high energy heavy ions irradiation simulating fission fragment impact due to specific character of the damage layer of irradiated material.

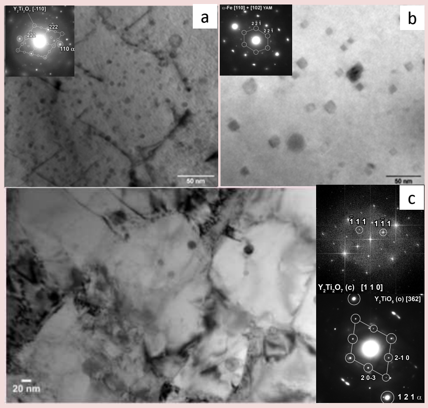

Fig.1 TEM bright field images and SAD patterns from oxide particles of EP-450 (a), KP4 (b) and Cr16 (c) ODS steels.

Damage profile produced by heavy ion irradiation is inhomogeneous. The most damaged region (several microns under the surface), often called straggling zone, lies at the end of the damage profile. In order to estimate the radiation hardening, one can apply the special procedures, e.g. 'unfolding' the damage profile on the sample surface via special irradiation modes.

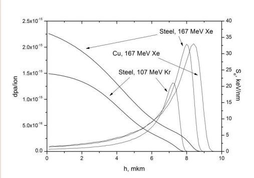

Fig.2 Damage and electronic stopping power profiles of Xe and Kr ions in ODS alloy and Cu.

Materials under study:

- *ODS alloys EP-450, Cr16 (VNIINM, Moscow),*
- *KP4 (Kyoto University)*
- *copper single crystal*

Irradiation parameters:

- 167 MeV Xe irradiation,
- 107 MeV Kr irradiation
- fluence: $1\cdot10^{12}$-$4.5\cdot10^{15}$ cm^{-2}
- dose ~0.01 dpa
- irradiation mode:

- direct irradiation,
- irradiation through the Al filters of variable thickness
- irradiation through bent filter

Fig. 3 IC100 accelerator (FLNR, JINR).

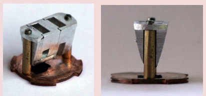

Fig. 4 Bent filter using for 'unfolding' the damage profile.

TEM observations

YAM particles in KP4 are more stable against the dense ionization induced by heavy ion irradiation comparably to $Y_2Ti_2O_7$ oxides in EP-450 ODS steel. With high fluence (>10^{14} cm^{-2}) irradiation YAM particles become amorphous and can't be identified as (Y, Al, O) compounds.

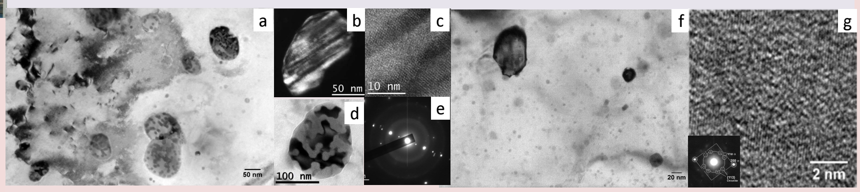

Fig.5 Microstructure of EP450 irradiated with $2\cdot10^{12}$ cm^{-2} 167 MeV Xe ions (a), latent tracks in $Y_2Ti_2O_7$: DF (b), HRTEM (c), BF (d), SAD image (e); Microstructure of KP4 irradiated with $2\cdot10^{12}$ cm^{-2} 167 MeV Xe ions (f), HRTEM of KP4 irradiated with $2\cdot10^{14}$ cm^{-2} Xe ions and SAD from chromites (g).

Nanomechanical testing: ODS steels

In order to find the valid region of radiation hardening estimation, measurements were performed in continuous stiffness measurement (CSM) with maximum indentation depth of 2 µm.

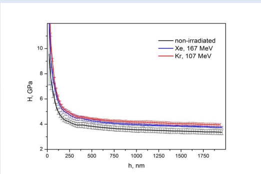

Fig. 6 Hardness profile of irradiated with Xe and Kr ions and non-irradiated KP4 ODS steel

According to the Nix-Gao model two size effects were observed: indentation size effect (ISE) and soft substrate effect (SSE).

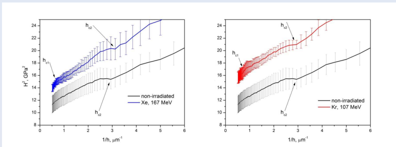

Fig.7 Reconstructed data in H^2 – 1/h coordinates of hardness profile of irradiated with Xe and Kr ions and non-irradiated KP4 ODS steel.

ISE: $h_{1c} = 0.3$ µm

SSE: $h_{2c} = 1.6$ µm for Xe irradiation

$h_{2c} = 1.3$ µm for Kr irradiation

Saturation of radiation hardening

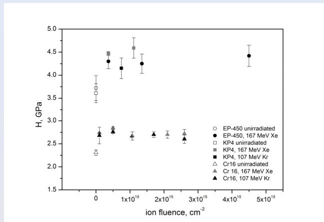

Fig.8 Hardness versus ion fluence for EP450, KP4 and Cr16 ODS steels

Nanomechanical testing: Cu single crystal

Irradiation through the flat filters

Irradiation of samples through filters of different thickness varying from 6 to 12 µm, allows estimating the proper depth range, where radiation hardening can be measured, according to the Nix-Gao model. This irradiation mode is more preferable comparably to the bent filter due to reduced ion scattering and related dispersion of the energy loss when passing through the foil.

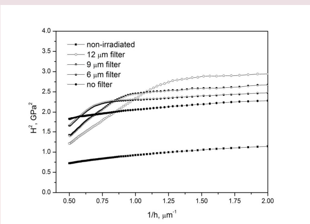

Fig.8 Reconstructed data in H^2 – 1/h coordinates of hardness profile of irradiated with Xe ions through foils and non-irradiated Cu single crystal.

The critical depths of 1.4, 1,1 and 0.78 µm for irradiated single Cu crystals irradiated through 6, 9 and 12 µm foils respectively, were obtained.

For the sample irradiated without filtering, the critical depth exceeded 1.8 µm.

Irradiation through the bent filter

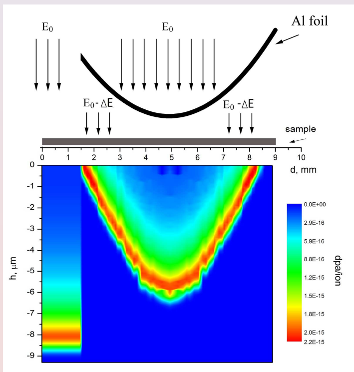

Fig.9 Schematic view of irradiation Cu with Xe ions through bent filter .

Radiation hardening of the sample, irradiated through the bent filter, was measured at different depths from 0.3 to 1.5 µm.

In case critical indentation depth exceeds the straggling zone, measured hardness approaches the initial value.

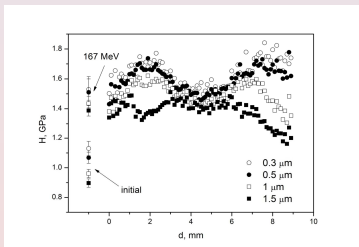

Fig.10 Hardness of copper single crystal: initial, irradiated with 167 MeV Xe ions and results from irradiation through bent filter.

Conclusions

Nanoindentation testing of the ODS alloys irradiated with 1.2 MeV/amu Xe and Kr ions strongly implies that radiation hardening level saturates at relatively low damage doses, around 0.01 dpa.

Similar dose behavior was found also for some pure metals irradiated with high energy heavy ions (Cu, Ni, Zr).

Detected radiation hardening is not associated with radiation-stimulated changes in the nanoparticle morphology.