Mechanical properties of ion irradiated steel EK-181 investigated by dynamic nanoindentation

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Abstract. Heavy ion irradiation is a unique tool that can be used to simulate and study radiation processes in materials. It can reproduce in a small near the surface layer various microstructural features (dislocation loops, precipitates, voids, etc.) observed in neutron-irradiated materials. Because the irradiation damage is restricted by a few micrometres near surface layer, mechanical properties investigation of the samples was carried out by dynamic nanoindentation technique. This method allows obtaining continuously the hardness and elastic modulus depth profile by one indent. Nanoindentation results acquired in this work showed increase in irradiated layer hardness caused by the increase in irradiation temperature and damage dose.

1. Introduction
Material research is an important task for development of energetics and other technical spheres. The most frequent characteristics under review are surface geometry and mechanical properties. Topography could be tested by scanning probe microscopy. Mechanical properties analysis, which is critically important for structural materials, could be carried out via nanoindentation. Current work demonstrates opportunities of nanoindentation to investigate radiation induced processes.

Most materials used at nuclear stations in the reactor pressure vessel constructions are exposed to neutron radiation. Such impact leads to induced radioactivity, heating, and damage of the material lattice structure. It is well established that irradiation with heavy ions can be used to simulate neutron irradiation. Ion irradiation can reproduce in a small near surface layer various microstructural features (dislocation loops, precipitates, voids, etc.) observed in neutron-irradiated materials. As shown in Ref. [1], there is a clear influence of radiation-induced features on material hardening.

SRIM (formerly TRIM) code based on Monte Carlo simulations is used to compute a number of parameters relevant to the ion beam implantation and modification of the materials. It can be used to calculate atomic displacements per atom (dpa), which is a common radiation damage exposure unit. In the work [2], the use of SRIM for calculation of dpa was evaluated and matching with an internationally recognized standard definition of dpa was made.

An austenitic alloy Fe-15Cr-20Ni after ion irradiation and helium implantation was studied in Ref. [3]. Influence on microstructural evolution, hardness, and behaviour of plastic deformation in
combined application of ion irradiation was estimated using technique of continuous nanosurface profiling. Austenitic alloy showed a strong (130%) hardening by single-beam irradiation.

In work [4], mechanical nominal hardness of ferritic alloys after irradiation by Fe ions was investigated. Experiments on ion irradiation were conducted at 290°C with 6.4 MeV Fe3+ ions. A depth profile of hardness was obtained via the constant stiffness measurement (CSM), being similar to dynamic nanoindentation technique. The results were considered on the basis of the Nix-Gao model and the widened film-substrate hardness model.

2. Material and methods
We investigated properties of promising ferritic-martensitic steel EK-181 after various irradiation modes. Irradiation temperature and damage doses are represented in Table 1. Steel samples were aged for 30 minutes in air at 980°C and tempered for 90 minutes in air at 760°C. Specimens used in irradiation experiments were prepared by the electric spark cutting in the shape of a disk with a diameter of 3 mm. The surface of the samples was subjected to multistage mechanical polishing using SiC abrasive sheets with a consequent reduction in the grain size. To remove mechanical deformations in the near-surface layer, electrochemical polishing was carried out using a solution of 5% HClO4 in ethanol. Heavy ion irradiation with Fe ions was carried out on a linear radio-frequency quadrupole (RFQ) accelerator HIPr-1 at NRC «Kurchatov Institute» - ITEP. Due to the special design of accelerating structure, it is possible to accelerate the ions up to energies of 101 KeV/nucleon. The total energy of Fe ions was 5.6 MeV. The irradiation was carried out under controlled heating of the target to 250, 300, 350, and 400°C. SRIM code was used to calculate damage dose according to recommendations provided in Ref. [2]. Ion implantation and displacement damage profiles are shown in Figure 1.

Nanoindentation is the process of recording loading-unloading curve, while indenter penetrates into the investigated material. One additional mode of the technique is dynamic instrumental indentation. It consists of monotonous indenter penetration at the same time with its harmonic oscillation. Such technique gives an opportunity to obtain depth dependent hardness and elastic modulus in a single experiment. The main disadvantage of this method is the local destruction of the material. For carrying out the tests, it is necessary to have a plastic imprint on the surface of the object under study. However, this feature makes it possible to obtain data on the hardness of the sample.

Table 1. Hardness values of samples under investigation.

| Sample number | Temperature (°C) | Doze (ion/cm²) | Hardness at 500 nm (GPa) | Nix-Gao: |
|---------------|-----------------|----------------|--------------------------|---------|
|               |                 |                |                          | H0(0°r) | h*(0°r) | h*(unirr) |
| 1             | initial         | -              | 3.85±0.14                | 3.70±0.12 | 62 | 100 |
| 2             | 250            | 3·10^{15}      | 4.32±0.21                | 4.27±0.17 | 63 | 236 |
| 3             | 250            | 6·10^{15}      | 3.98±0.16                | 3.82±0.09 | 67 | 100 |
| 4             | 250            | 1·10^{16}      | 4.03±0.12                | 4.06±0.13 | 41 | 168 |
| 5             | 300            | 3·10^{15}      | 4.11±0.22                | 4.06±0.12 | 54 | 190 |
| 6             | 300            | 5.6·10^{15}    | 3.93±0.14                | 3.93±0.14 | 52 | 179 |
| 7             | 300            | 1·10^{16}      | 4.07±0.19                | 4.00±0.03 | 49 | 162 |
| 8             | 350            | 6·10^{15}      | 4.05±0.17                | 3.98±0.14 | 55 | 160 |
| 9             | 400            | 3·10^{15}      | 4.04±0.18                | 3.93±0.13 | 63 | 152 |
| 10            | 400            | 5.6·10^{15}    | 4.12±0.14                | 4.05±0.12 | 76 | 216 |
| 11            | 400            | 1·10^{16}      | 4.29±0.22                | 4.26±0.21 | 84 | 274 |
Figure 1. Depth profile of displacement damage (solid line) and implanted ions concentration (dots) in the EK-181 steel irradiated by 5.6 MeV Fe ions.

Figure 2. Elastic module of irradiated sample (grey line) and initial sample (black line).

A study of the mechanical properties of the samples was carried out on the NanoScan-4D nanohardness tester [5] (FSBI TISNCM, Russia) with a Berkovich type indentation tip using the dynamic nanoindentation. Calibration of the indentation tip shape and hardness calculation are based on the Oliver-Pharr method. We used the following test parameters: indentation depth 2000 nm, oscillation amplitude 10 nm, frequency 10 Hz, and loading rate 200 nm/min. A series of 30 measurements was carried out for each sample.

3. Results and discussion
Figure 2 demonstrates depth profiles of elastic modulus in the sample before and after irradiation. Black line shows data obtained on the unirradiated sample and grey line refers to the sample, which was exposed to 5.6 MeV Fe\(^+\) ions with total fluence of \(1 \times 10^{16}\) ion/cm\(^2\) at 350°C. The observed difference is rather small (less than 5\%) and only slightly exceeds the measurement error. Measurement of the rest irradiated samples demonstrated similar values of elastic modulus. The nearness of the elastic properties of the irradiated layer and the substrate allows calculating the true contact area and true hardness [6], irrespective of the effects of pile-up or sink-in.
Figure 3. Indentation-depth profiles of the averaged hardness at 10 dpa damage dose and heating to 250°C or 400°C comparing to hardness of initial sample.

Figure 4. Plot of $H^2$ versus $1/h$ for initial sample and irradiated at 10 dpa damage dose and temperature 400°C material. The lines show approximation of data groups of near-surface layers (dashed lines) and deep layers (solid lines).

Figure 3 shows indentation-depth profile of hardness of EK-181 steel before and after irradiation at 10 dpa damage dose. Hardness dependence on irradiation temperature was clearly observed. Data up to 100 nm was ignored due to test artefacts inherited from the indenter-surface contact uncertainties.

The measured hardness is maximal in the near-surface layer and decreases uniformly with increasing depth, tending to the hardness of the non-irradiated substrate material. The similar behaviour can be seen in the initial sample, which can be explained by the well-known indentation size effect [7-8]. In order to explain the ISE, Nix and Gao suggested a model based on a concept of geometrically necessary dislocation. The Nix–Gao model states that the hardness depends on the indentation depth as follows:
\[ H = H^0 \left( 1 + \frac{h^*}{h} \right)^{0.5} \]  

(1)

where \( H^0 \) is the bulk hardness of the sample, \( h^* \) is a specific length, which relies on the material and the shape of indenter tip. To calculate the \( H^0 \) and \( h^* \), data was plotted as \( H^2 \) versus \( 1/h \).

Figure 4 illustrates an example of such plot. Table 1 shows \( H^0 \) for irradiated layers of the samples and \( h^* \) for both regions. Average hardness value of unirradiated layers of all the samples is 3.50±0.07, which is close to hardness of initial sample. A border between irradiated and bulk material layers was estimated at a depth of 300 nm. This choice is due to the softer substrate effect (SSE), which can be seen in the various systems of hard thin film on soft substrate [9]. Besides, the table includes results of hardness at a depth of 500 nm, the values obtained are similar to results of Nix-Gao model.

Obtained data show that with the increase in damage, dose hardness of irradiated layer increases. This may be explained by formation of radiation induced defects. Number density of these features strongly correlates with the level of radiation damage. Maximum increase in hardness (0.76 GPa) was observed for the steel sample irradiated up to 10 dpa at 400°C.

4. Conclusions
Dynamic nanoindentation technique was used to investigate influence of Fe⁺ ion irradiation on mechanical properties of f/m steel EK-181. Depth dependences for the hardness and elasticity modulus are constructed. The elasticity modulus of the samples does not change after irradiation. Gradient of hardness can be explained not only by the indentation size effect, but also a softer substrate effect. Strong dependence of irradiated layer hardness on the damage dose and irradiation temperature is observed.

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