



Mechanical and structural properties of ODS RAF steels submitted to low-energy ions irradiation

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ARTICLE INFO

Keywords:

ODS steel

Ion irradiation

Mechanical properties

Nanoindentation

ABSTRACT

The effect of ion implantation on mechanical and structural properties of 12%Cr ODS steel have been investigated. Specimens were submitted to single (He^+ or Fe^+) and dual ion beam (He^+ and Fe^+) implantation campaigns. Modification of three mechanical parameters: hardness, Young modulus and yield strength were characterized by means of nanoindentation technique. Structural properties were measured by using Grazing Incidence X-ray Diffraction (GIXRD) method. Correlation between obtained mechanical and structural results have been found. Recorded results show that ion-irradiation causes hardening effect which may be associated with creation of radiation defects and dislocation loops. The weakest effects have been observed for single He-ion implantation, whereas the dual beam irradiation led to significant change of all mechanical parameters. During structural analysis, small shifts of α -Fe peaks have been observed. Reported structural changes can be attributed to the effect of local stress introduced during ion irradiation.

1. Introduction

Materials considered for structural components of future nuclear applications are expected to meet number of requirements, for example: (i) structural stability in intense irradiation and high temperature (400–700 °C), (ii) high swelling resistance and (iii) excellent mechanical properties. Reduced Activation Ferritic Oxide Dispersion Strengthened (ODS RAF) steels are one of the most promising candidates for such harsh work environment. Due to thermal stability of the nanometer oxides particles (Y_2O_3 , TiO_2 and Al_2O_3) dispersed in ferrite matrix, ODS steels exhibit improved mechanical properties at high temperature, excellent creep strength and swelling resistance maintaining at the same time good corrosion resistance [1–4].

Study of the irradiation effects is one of essential issues in applicability of ODS steels in nuclear industry. Transmutation products such as (n, α) and (n, p) play a crucial role in deterioration of materials' properties. Generation of vacancies, interstitial atoms, dislocation loops, recombination of α parts and growth of He bubbles are major irradiation-induced structural changes. All these factors are reported as sources of embrittlement [5,6] and hardening effects [1,7,8] in metallic alloys. In order to study influence of irradiation on above-mentioned properties, one must either place promising materials into the reactor core or simulate reactor environment by ion implantation. However,

direct neutron irradiation produce highly radioactive materials and is time – consuming methodology. For this reason ion-implantation has been found as a safe (no sample activation), fast (high dpa level can be obtained in hours or days instead of years) and fully – controlled method, which allows to imitate true reactor conditions. Currently, the most advance techniques include dual or triple beam irradiation in simultaneous [9,10] or sequential mode [6,9]. The most common technique is implantation of He^+ ions which imitates α particles [11,12], and self-irradiation which induces cascade effect [12,13].

Although, there are many experimental works devoted to this subject [6–13], ODS steels are still under intensive investigation including different, complex regimes of irradiation modes. The aim of this study was to correlate nanomechanical and nanostructural properties of 12%Cr ODS alloy submitted to sequential and dual beam low energy ion implantation of He^+ / Fe^+ . Since ion implantation modifies only thin layer of material, bulk of the studies were conducted by using nano-scale method (nanoindentation [14–17] and GIXRD [7]). In addition to above mentioned goal of this study an issue of reliability of obtained results and possibility of estimating mechanical parameters by probing very small volume have been addressed. For this reason, nanomechanical results have been confronted with literature data and micromechanical tests. Micro-hardness – HV0.1 tests have been conducted on the virgin material. Reported micromechanical investigation

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Table 1
Chemical composition of research material.

C	Cr	W	Ti	Y ₂ O ₃	Ni	Si	Ni
0.02	12.2	1.94	0.25	0.22	< 0.01	0.03	< 0.01

have been found to be in full agreement with literature results [18,19].

2. Materials and methods

2.1. Material preparation

An ODS steel samples were cut from a 6.5 mm-thick plate manufactured by KOBELCO-Japan (chemical composition of the specimens can be found in Table 1). Studied material were previously used in GETMAT project [20]. In order to produce investigated samples, elemental powders were mechanically alloyed (220 rpm/48 h) in Ar atmosphere and consolidated in 1150 °C by Hot Extrusion (HE) technique which resulted in obtaining a bar with diameter $D = 30$ mm. Afterwards, cast material was submitted to hot forging process with inter-annealing treatment (1150 °C/1 h) until thickness of 6.5 mm were obtained. Fully described production details of the cast material can be found in [19].

Afterwards, rectangular shape (8.0 × 6.5 mm) samples were cut as 0.5 mm-thick specimens by using Wire Electric Discharge Machining (WEDM) method (Fig. 1). Subsequently, they were grounded with abrasive papers with gradation from 220 to 1200× and then mirror-polished using diamond-suspensions with grain size from 9 μm to 1 μm. Final step of polishing was performed with silica suspension (0.1 μm grain size).

During ion-implantation, specimens were irradiated up to ~150 nm thickness (the highest damage of the material occurs up to 100 nm depth). Hence, micro-hardness test is not sensitive enough to display changes of mechanical properties. Therefore, in order to evaluate results obtained by micro- and nano-scale methods, studies of the non-irradiated ODS material were performed and compared with literature data [18,19]. However, it should be pointed out that the goal of this study is to confront nanomechanical and nanostructural characteristics of the material and address subject which is still poorly understood in the literature.

2.2. Microstructure characterization

Studied specimens were examined in terms of microstructure by using Scanning Electron Microscopy (SEM) equipped with secondary electron (SE) detector, Energy selective Backscattered Electron (EsB, low-loss BSE) detector and EBSD system provided by Bruker. Special design of lens and positioning of detectors provide separation of SE and BSE signals in real time. Images of inverse pole figure maps (IPF) were presented in orthogonal direction respectively to the sample.

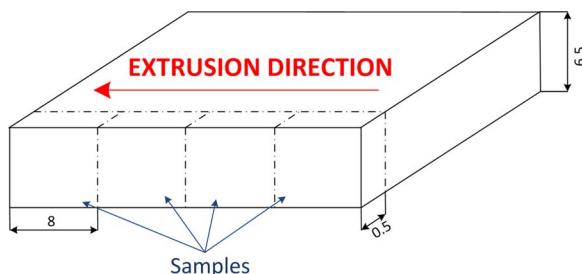


Fig. 1. Scheme of the material sampling by using Wire Electric Discharge Machining (WEDM) method – dimensions in mm.

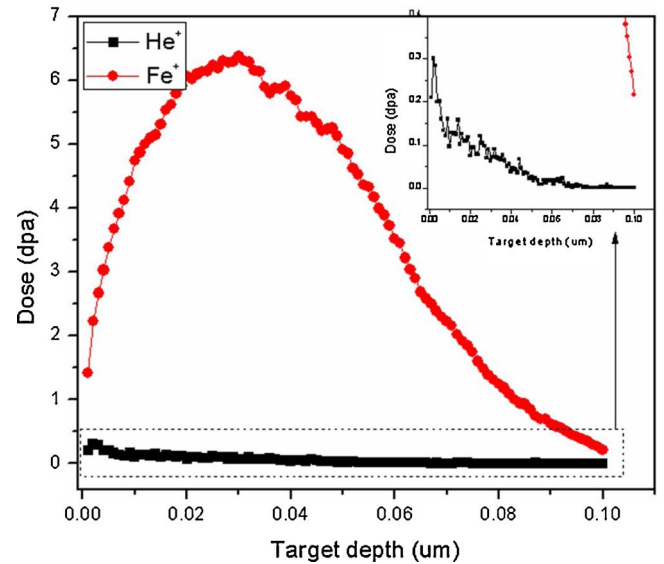


Fig. 2. The SRIM [21] damage profile for 30 keV/75° He⁺ and 150 keV Fe⁺ ions in 12%Cr ODS steel. The calculations were carried out for a dose of 1×10^{17} ions/cm² using the threshold energy of 40 eV and the Kinchin-Pease option.

2.3. Ion irradiation

Rectangular samples were submitted to sequential dual beam ion irradiation. The parameters of ion implantation (energy and specimen angle) were determined by using SRIM code [21]. In order to maximize material damage at similar depths, energy of He⁺ and Fe⁺ ions were set as 30 keV and 150 keV, respectively. During He⁺ implantation specimens were positioned at an angle of 75° to the beam. These parameters corresponded to modification of ~150 nm thick layer. However highest damage of the material occurs until 100 nm depth, see Fig. 2 (calculated within use of SRIM code [21]). During implantation the ion beam current was limited to 0.1 μA cm⁻² to avoid significant heating of the samples. The temperature of the specimens were controlled continuously using a thermocouple. Full irradiation campaign is presented in Table 2.

2.4. Mechanical measurements

Nanomechanical properties were investigated by using NanoTest Vantage system provided by Micro Materials Laboratory. Measurements were performed with Berkovich-shaped indenter using 1 mN load, which provides indentation depths from 50 to 70 nm (depending from the specimen hardness). This corresponds to approx. 50% thickness of modified layer. According to Dolph et al. [22], in order to obtain valuable mechanical data, nanoindentation depth should be set in the range of 20–50% of the total thickness of the tested layer. Implementation at these conditions guarantees collecting of the data only from the studied layer (without influence of the bulk material). However, in our case some influence of unmodified substrate material is expected. Each measurement was repeated at least 8 times. Oliver and Pharr method [23] was applied for calculation of nanomechanical parameters: nanohardness and Young modulus. Yield strength σ_{ys} was determined using relationship for b.c.c. Fe-Cr alloys [22,24]:

$$\sigma_{ys} [\text{MPa}] = 3.06 H_V \left[\frac{\text{kg}}{\text{mm}^2} \right] \quad (1)$$

where H_V is the Vickers hardness. Correlation between Vickers hardness H_V and Berkovich nanohardness H_B is defined by Fischer-Cripps equation and can be found in [25]:

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