INVESTIGATION OF HELIUM-INDUCED EMBRITTLEMENT

Veronika Sabelová[1]*, Vladimír Kršjak*[2]*, Vladimír Slugeň*[1]

[1] *Faculty of Electrical Engineering and Information Technology, Slovak University of Technology in Bratislava, Ilkovičova 3, 81219 Bratislava, Slovakia,*

[2]*Paul Scherrer Institut, 5232 Villigen PSI, Switzerland*

E-mail: sabelova.veronika@stuba.sk

Received 12 May 2014; accepted 24 May 2014

Abstract

In this work, the hardness of Fe-9%(wt.)Cr binary alloy implanted by helium ions up to 1000 nm was investigated. The implantations were performed using linear accelerator at temperatures below 80°C. Isochronal annealing up to 700°C with the step of 100°C was applied on the helium implanted samples in order to investigate helium induced embrittlement of material. Obtained results were compared with theoretical calculations of dpa profiles. Due to the results, the nanohardness technique results to be an appropriate approach to the hardness determination of thin layers of implanted alloys. Both, experimental and theoretical calculation techniques (SRIM) show significant correlation of measured results of induced defects.

1. Introduction

The consequences of radiation in structural materials are one of the most studied effects in the field of nuclear research. Irradiation effect includes complex combination of various types of damage in various dimensional and time scales. Irradiation of materials in real conditions of nuclear facility for many investigations purposes would be complicated task. Therefore the simulation of radiation damage using implantation facilities is often used today. With increasing burn-up of the nuclear fuel various in-core components may suffer from the high production rate of helium. Helium embrittlement is also an important lifetime-limiting factor of plasma facing components in fusion devices. Despite the strong research efforts aimed at the understanding of the phenomenon [1-12] a unified helium embrittlement mechanism has not emerged due to discrepancies between experiment and theory [13].

1.1 Helium embrittlement

Helium is formed in construction materials by well know nuclear reaction (n,α). High energy α (2 - 9 MeV) created in the nuclear reaction produce displacement damage by series of cascade collisions. After energy transmission, helium is settled down in open volume defects, where particle density is lower. Immobile dislocations and vacancies containing helium get worse thermal expansion and internal stress grows. Helium embrittlement is typical feature of material ageing [14].

The transmutation helium is quite insoluble in irradiated materials. It has tendency to diffuses in to the matrix and form a bubbles at microstructural trapping sites at higher temperature range. These bubbles can cause the grooving of voids and creep cavities at grain boundaries [15], and thus they change the total value of He-to-dpa ratio. These values give reasonable information about the helium behaviour during thermal treatment of irradiated material.

2. Experimental

The experimental investigation of the helium behaviour in structural materials is limited by the resolution of the instruments available. Even the high-resolution microscopy techniques cannot distinguish helium-filled defects in the early stages of embrittlement and only large bubbles of few nm sizes are visible to them.

2.1 Investigated material and implantation performance

The material used in this work is Fe – Cr based model alloy with 9%Cr (8.39 %wt.Cr) obtained by furnace melting of industrial pure Fe and Cr (detailed chemical composition is reported in [14]). After casting, the obtained ingots were cold worked under protective atmosphere to fabricate plates of 9 mm in thickness. Fe – Cr model alloys were treated at 1050 °C, for 1 h in high vacuum for stabilization. Thereafter, the tempering was done at 730 °C for 4 h, followed by air cooling.

Linear ions accelerator (it used energies to 900 keV) at Slovak University of Technology in Bratislava was used for specimen implantation by helium ions (He^+) and thus simulation of high nuclear damage. Two levels of fluencies and energies were used for that purpose. Temperature during implantation was below 80°C.

2.2 1. Hardness indentation of surface layers

Hardness testing is a very convenient tool for evaluation of material properties, quality control of manufacturing and development processes. The nanoindentation technique is powerful method for ultra-small scale material testing of mechanical properties changes due to the ion beam irradiation. Due to the complicated geometry involves (tip, sample, surface preparation, etc.) it not a simple method for quantitative numbers [16]. The measurements were performed using Nano Indenter G200 system. Continuous stiffness measurement is nanoindentation mode based on constantly increasing load on tip in order to obtaining the load as function of indent depth. Hardness is defined as the load divided by contact area of plastic displacement.

Performing of nanoindentation to various indentation depths leads to different hardness reading due to the small scale of testing technique. This effect is called indentation size effect [17]. Therefore indentation procedures with same indentation depth were realized using diamond Berkovich tip after precise focusing on the specimen´s surface. Ten indents for each measurement were performed to obtain a good statistical data. Nanoindentation of helium implanted material, affected by exfoliation of surface layers required a manual selection of location of each indent. Every test was performed on unblistered surface. The correct location of indent is shown in Fig. 1. The comparison between correct and incorrect indent are marked with black circles.

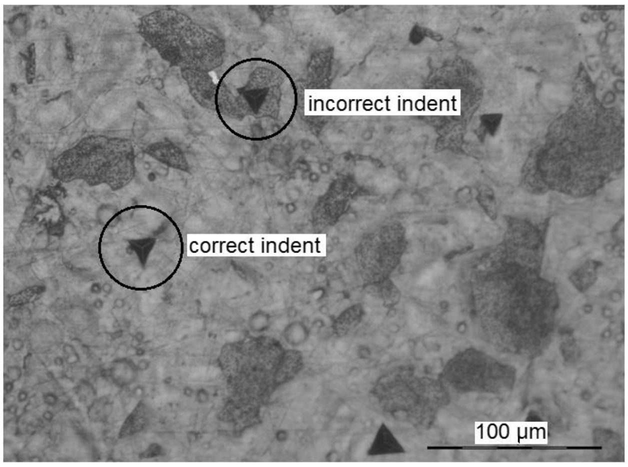

Fig.1: Berkovich indents of Fe-9%Cr alloy implanted by He$^+$ manually located, with correct and incorrect indent comparison.

For computing of radiation damage exposure the Kinchin – Peace model [18] was selected using SRIM 2013 (formerly TRIM) Monte Carlo simulation code interphase. This model assumes a linear relationship between the number of Frenkel pair produced and the initial energy of a primary knock-on atom. Below the threshold energy (40 keV), no new displacements would be produced. The energy above the high energy cut-off is dissipated in electronic excitation and ionization [19].

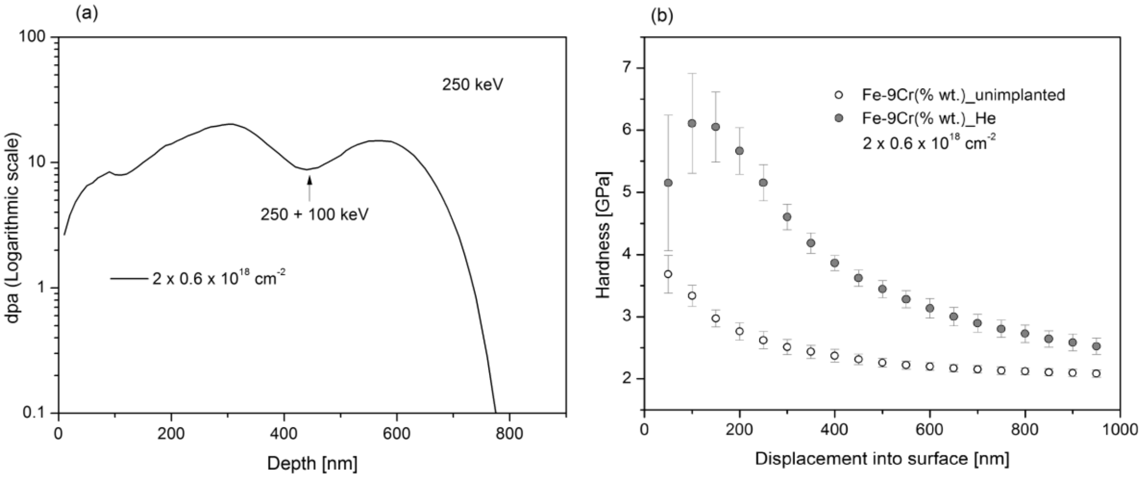

Fig.2: Calculated dpa profile of Fe-9%Cr alloy implanted by He$^+$ ions (a) and average hardness profiles of unimplanted and implanted Fe-9%Cr versus indenter displacement into the surface (b). Error bars represent standard deviation. Implantation was performed using He$^+$ ions with fluency 2 × 0.6 × 10^{18} cm^{-2} and energies 250 and 100 keV.

During indenting of the samples surface in the ion beam implantation direction, a wide dose range is sampled due to the not homogeneous dose profile. The average hardness depth profiles of unimplanted and implanted iron-chromium alloy are shown in Fig. 2 (b). The characteristic peak is typical for He$^+$ implanted material. It can be clearly seen, after comparison with theoretical calculations obtained from SRIM program (Fig. 2 (a)), that the positions of peaks maximum are shifted to the ~ (100 – 200) nm surface area. This effect is caused by the volume of material affected by indent itself. For standard Berkovich tip indent is characteristic plastic zone five times larger as indent itself. In other words, if 200 nm deep indent is performed, about 1000 nm depth of the material is sampled and contributes to the hardness reading [20, 21]. Therefore the peak of hardness depth profile of implanted iron-chromium alloy is shifted to the surface area and investigation of hardness in the most affected area by implantation was performed in 200 nm beside 1000 nm depth.

2.3 Thermal treatment

The isochronal annealing was performed in ceramic furnace at temperatures 100 – 700 °C with 100 °C steps. After every annealing step (1 h) and air cooling to room temperature the experimental measurements were performed.

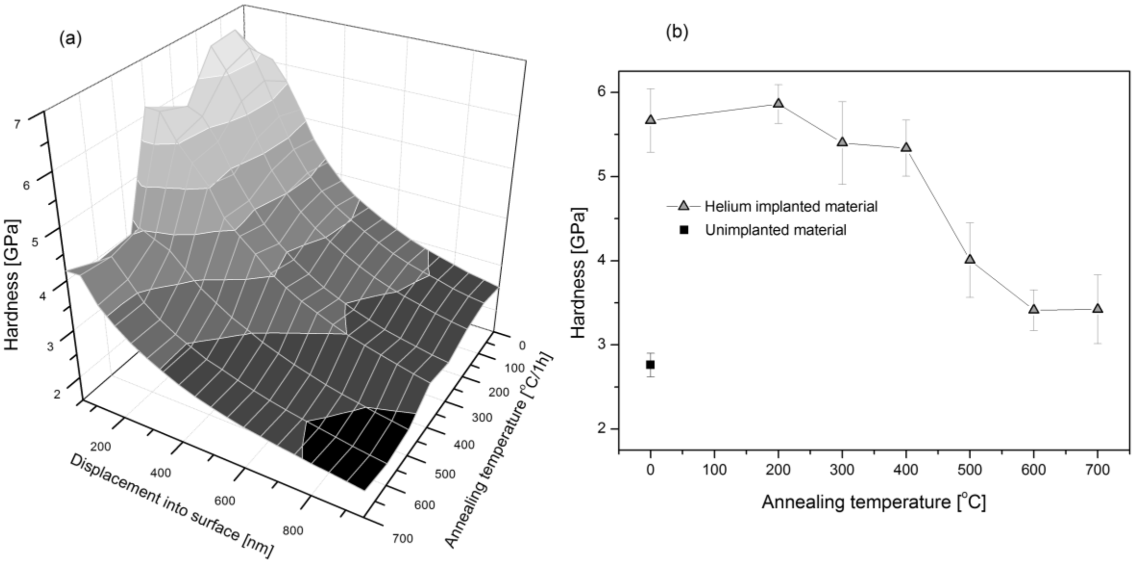

Fig.3: Hardness profiles 3D representation of He^+ implanted Fe-9%Cr alloy (a) and hardness of unimplanted and implanted Fe-9%Cr material in 200 nm depth during isochronal annealing up to 700 °C.

The annealing experiment of and He^+ implanted material is shown in Fig. 3 (a) in the 3D representations up to 700 °C with 100 °C temperature steps. The sample was slowly cooled on the air after each annealing and then investigated with nanoindentation. Every point of graphical view represents average value from ten measurements of indentation process. The annealing up to 700 °C causes softening of surface area, reducing stress and decreasing strain hardening after manufacturing and implantation itself.

The comparison of He^+ implanted Fe-9%Cr alloy hardness in 1000 nm investigated depth during thermal treatment up to 700 °C is shown in Fig. 3 (b). The softening occurs at higher annealing temperatures. It is well known that high helium concentrations and helium bubbles formation (~ 500 °C [15]) produce void swelling [22]. It is reasonable to expect that the concentration of helium in large bubbles is lower than in the small clusters and therefore they may act as a weaker barrier for dislocation movement. Such hypothesis could explain the decrease of hardness of implanted samples at higher temperatures.

3. Results and discussion

The study in nano-scale range was chosen for hardness investigation of damaged surface areas of implanted alloy. The sensitive nanoindentation was used with advantage, since, due to blistering, a manual selection of location of each indent had to be performed. Despite these complications the hardness profiles into the specimens up to 1 μm were realized. Hardness profiles of implanted Fe-9%Cr alloy implanted by helium ions during isochronal annealing up to 700 °C show behaviour of material, where the softening occurs. The main assumptions are that helium mostly still remains in material, but it form bubbles, which seem to be weaker barrier for dislocation movement.

Acknowledgement

We would like to acknowledge collaboration with Paul Scherrer Institute in Switzerland, the projects VEGA (Slovakia) Nos. 1/0366/12, 1/0204/13 and APVV project (Slovakia and Czech Republic) No. 2013-0197.

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