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Vacancy-type defects and hardness of helium implanted CLAM steel studied by positron-annihilation spectroscopy and nano-indentation technique

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ABSTRACT

China Low Activation Martensitic (CLAM) steel was implanted with helium up to 1e + 16/cm^2 at 300–873 K using 140 keV helium ions. Vacancy-type defects induced by implantation were investigated with positron beam Doppler broadening technique, and then nano-hardness measurements were performed to investigate helium-induced hardening effect. He implantation produced a large number of vacancy-type defects in CLAM steel, and the concentration of vacancy-type defects decreased with increasing temperature. Vacancy–helium complexes were main defects at different temperatures. Irradiation induced hardening was observed at all irradiation temperatures, and the peak value of hardness was at 473 K. The result suggested that both vacancy–helium complexes and helium bubbles had contribution to irradiation induced hardening. The decomposition and annihilation of irradiation-induced defects became more and more significant with increasing temperature, which induced the increment of hardness became more and more small.

1. Introduction

Reduced-activation ferritic/martensitic (RAFM) steels are the prime candidate structural material for fusion reactor blanket/first walls, due mainly to their superior resistance to radiation-induced changes in their physical and mechanical properties [1]. F82H [2], JLF-1 [3], ORNL-9Cr2WVTa [4] and EUROFERE97 [5] are outstanding examples of the materials. One of the most important remaining key technical issues for the RAFM steels as fusion structural materials is the influence of transmutant helium production on the irradiation-related microstructure and mechanical property changes. Helium can have a pronounced effect on the radiation damage of materials and often may be an important reason in catastrophic degradation of their properties and shortening of the useful life of reactor constructional elements.

It is well known that He stabilizes vacancy clusters into helium–vacancy complexes which evolve into bubbles or voids. Helium bubbles [6,7] were formed in low activation martensitic steels implanted with helium at 673–873 K. Voids were also generated in F82H after in situ He injection at 773 K [8]. Both the bubble and void flow from the evolution of helium–vacancy complexes. During irradiation, bubbles nucleate and grow by forming vacancy–gas atom complexes as they accumulate gas atoms. When bubbles exceed a certain critical size or accumulate a certain number of gas atoms, they can grow inexorably by absorbing vacancies without the need for gas atoms. When bubbles are smaller than a certain size, thermal emission prevails over the absorption of vacancies. This demarcates bubble sizes below and above the critical size, which causes development of a bimodal cavity size distribution [9] which was found in experiment [10,11], too.

Several methods are used to simulate the He effect on mechanical properties. Single and dual beam irradiation at 533–693 K indicated that helium caused an increase in strength [12,13]. Helium effects on mechanical properties were identified to RAFM steel with different contents of constituent element boron [14–16]. Helium implantation techniques reach high concentration of helium and allow the investigation of specific effect of helium on materials properties. Damaged volume induced by helium ion implantation with different energies is limited to the thin layer close to specimen surface, so positron annihilation techniques have been applied to investigate the defects introduced during irradiation for different kinds of materials. It is very sensitive for

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the open-volume defects such as vacancy and vacancy clusters [17]. With a beam of controllable energies, the positron may be implanted at various depths beneath the surface and thus probe the defect depth profile. This technique has been widely used for detecting point defects in first wall and structural materials for future fusion reactors which were irradiated by high energy particles [18,19]. Application of the dynamic indentation technique using load- and displacement-sensing instruments, which provides continuous apparent hardness profiles of graded materials, enhanced the applicability of indentation technique to the ion-induced hardening evaluation [13,20,21]. In the present work, both the positron annihilation and dynamic indentation techniques are used to investigate microstructure and nano-hardness of helium irradiated CLAM steel. CLAM steel had been developed several years and a series of R&D activities for this material are currently being done [22–24]. The results of helium implanted CLAM steel at room temperature had been reported in our previous work [25], but the results on high temperatures irradiation are few. So the helium implantation at high temperatures was performed to investigate the helium effect.

2. Experiment

The composition of the CLAM steel (USTB) is 9.08Cr, 1.48W, 0.18V, 0.097Ta, 0.098C, 0.46Mn in wt% and Fe for the balance. The steel was normalized at 1233 K for 0.5 h and tempered at 1033 K for 1.5 h. Specimens were fabricated into coupons of 10 mm × 10 mm × 1.5 mm sizes. Surfaces of the samples were mechanically polished, initially with sandpaper of varying grits and finally, to a fine mirror finish with 1 μm diamond paste. Helium implantation is carried out by an implantation apparatus located in the Accelerator Lab of Wuhan University. The CLAM steel was implanted by using 140 keV helium beam with fluences up to 1e + 16/cm^2 at different temperatures with 300, 473, 673 and 873 K.

Positron Doppler broadening measurements were performed at room temperature using a slow positron beam available at the Key Laboratory of Nuclear Analysis Techniques, Institute of High Energy Physics, Chinese Academy of Sciences. In this measurement system, the β+ decay of a 22Na radioactive source was used to obtain positrons, which were slowed down and transmitted through the vacuum pipe of a magnetically controlled system. This yields a slow positron beam with energy of 24 eV. The implantation depth of the slow positron beam in the samples can be varied by changing the negative high voltage on the sample holder. Data on the 511 keV annihilated photons was collected using a high-purity Ge detector (ORTECGEM-1075), which has an energy resolution of 1.2 keV (FWHM) for the 514 keV γ-rays of 85Sr. The positron annihilation spectra were characterized by the conventional S and W parameters, where the S parameter is defined as the ratio of counts in the central energy region (511 ± 0.8 keV in this paper) to the total counts of the photo peak of 511 keV. The W parameter is the ratio of the wing area (504.2–508.4 keV and 513.6–517.8 keV in this paper) to the total area under the entire spectrum. The S and W parameters yield information on low-momentum and high-momentum electrons respectively.

Nano-hardness is an effective method to study mechanical properties of materials in micro-scale. The profiles of hardness vs. depth for helium irradiated CALM steel were detected by Nano Indenter II system at the State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing. The calculation of hardness was conducted by the method used by Oliver and Pharr [26]. The method of continuous stiffness measurement (CSM) was employed to record continuously the hardness as a function of depth.

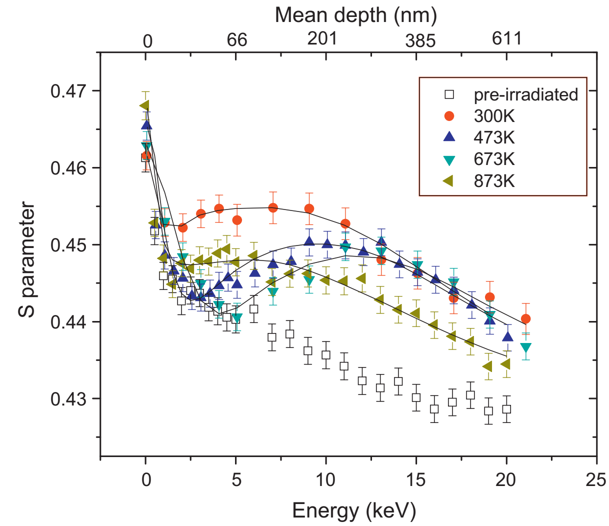

Fig. 1. S–E curves of helium ion implantation at different temperatures.

3. Results and discussion

3.1. Results of positron annihilation at different irradiation temperatures

Fig. 1 shows the dependence of the S parameter on implanted positron energy (S–E curves) for pre and post implanted at 300, 473, 673 and 873 K. The S parameter for the implanted sample became larger than that for the pre-implanted ones, which indicates that positrons detect the presence of vacancy-type defects generated during implantation, regardless of the temperature. The lines in Fig. 1 show the fitting curves calculated with VEPFIT [27]. The top *x*-axis of Fig. 1 is the mean depth of the annihilating positrons.

The theoretical helium atoms and defects profiles determined from SRIM [28] calculations are shown in Fig. 2. It indicates that both the helium atoms and defects peaks at about 400 nm for 140 keV He. In Fig. 2, the irradiated region can be divided into TR, BR and NIR [25] regions and their meanings are explained as follows: TR corresponds to the zone where ions slow down mainly by electronic energy loss processes. BR is the Bragg peak region, corresponding to the region where incident ions interact with atoms of the solid via nuclear collisions, and finally stop within the lattice. NIR is the non-implanted region. The VEPFIT analysis of the data identifies four layers in the irradiated samples, and the four layers are surface, TR, BR and NIR layer, respectively. In Fig. 3 the normalized S parameter (normalized to the bulk value) and

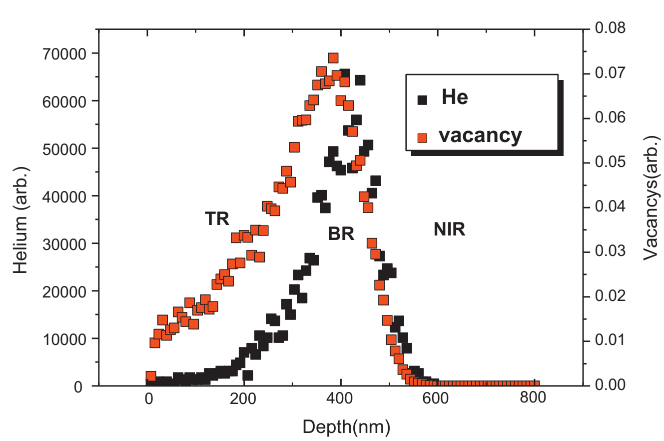

Fig. 2. The distribution of helium atoms and vacancies.