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Investigation by slow positron beam of defects in CLAM steel induced by helium and hydrogen implantation

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ABSTRACT

Hydrogen and helium ion beams delivering different doses are used in the ion implantation, at room temperature, of China Low Activation Martensitic (CLAM) steel and the induced defects studied by Doppler broadening of gamma-rays generated in positron annihilation. Defect profiles are analysed in terms of conventional S and W parameters, measures of relative contributions of low and high-momentum electrons in the annihilation peak, as functions of incident positron energies E up to 30 keV. The behaviours of the S–E, W–E and S–W plots under different implantation doses indicate clearly that the induced defect size has obvious variation with depth, taking values that interpolate between surface and bulk values, and depend mainly on helium ion fluences. The S–W plot indicates that two types of defects have formed after ion implantation.

1. Introduction

In the prospective International Thermonuclear Experimental Reactor (ITER), reduced activation ferritic/martensitic steels (RAFM steels) have been considered as primary first wall and blanket structural materials because of their better swelling resistance, thermo-physical and thermo-mechanical properties when compared with austenitic stainless steels [1,2]. Research on RAFM steels has been carried out in Europe, Japan and USA in the past 20 years and some inspiring progress has been made, including the development of F82H [3], JLF-1 [4], ORNL-9Cr2WVTa [5] and EUROFER97 [6]. In China, China Low Activation Martensitic (CLAM) steel has also been developed and a series of R&D activities are currently being carried out [7].

With regard to ITER, the high 14 MeV neutron flux will generate a continuous production of both hydrogen and helium in structural materials within the reactor via nuclear reactions (n, p) and (n, α) [8,9]. As a consequence, degradation of the materials can occur by the accumulation of hydrogen and helium, and hence the development and classification of materials capable of withstanding such extreme conditions is a critical step towards the actual realisation of ITER and any future fusion nuclear reactors.

Both hydrogen and helium are known to play an important role in the evolution of microstructural damage, and affect the mechanical properties and cracking behaviour of structural materials in proximity of the confined high temperature plasma [10,11]. In particular, it was reported that helium atoms can affect the Ductile to Brittle Transition Temperature (DBTT), swelling behaviour and irradiation hardening in martensitic steel [12]. A shift in DBTT is an indicator of degradation within the material. Thus, the effect of transmutation hydrogen and helium production on irradiation-related physical and mechanical properties is a key technical issue for RAFM steels. In order to study the synergistic effect of hydrogen and helium on microstructure, ion implantation experiments were carried out using hydrogen and helium ions. Hydrogen–helium interactions are interesting problems not only from an engineering perspective, but also a fundamental one.

Positron annihilation spectroscopy (PAS) is one of the more powerful and well-established techniques to characterise vacancy-type defects (micro-voids and open volume regions) of a material. This technique provides a highly sensitive penetrative probe combining the sensitivity of positrons to defects with the penetrability of gamma-rays. Lifetime spectroscopy of the mono-energetic positron beam provides a means to measure defect

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Table 1
Chemical composition of material studied (wt.%).

Material	C	Cr	W	V	Ta	Si	Mn	P	S	Fe
CLAM	0.15	9.23	1.37	0.20	0.15	0.083	0.43	0.0032	0.0083	Bal.

profiles in materials [13,14]. In particular, defect structures formed after ion implantation can also be investigated with PAS.

In this article, we specifically present a PAS analysis of defect profiles in CLAM steel as induced by ion implantation.

2. Experimental

The material analysed in this study is specifically CLAM (FDS-HEAT 0603A). Its chemical composition is listed in Table 1.

Samples of CLAM steel were melted in a vacuum induction furnace into 300 kg ingots, and subsequently hot-forged and rolled into 12 mm-thick plates. An austenitizing heat treatment regulated at 1253 K/30 min was applied followed by air cooling plus tempering at a 1033 K/1.5 h cooling rate. Specimens were fabricated into coupons of 16 mm × 16 mm × 1.5 mm sizes. Surfaces of the samples were mechanically polished initially with sandpapers of varying grits and finally to a fine mirror finish with a 1 μm diamond paste.

H^+ and He^+ implantations were performed at room temperature with a 200 kiloelectron volt (keV) ion implanter located in the Accelerator Lab of Wuhan University. Energies of both hydrogen and helium ion beams were fixed at 80 and 140 keV, respectively. Depth distributions of implantation were calculated by the SRIM code [15]. H^+ and He^+ ions were implanted to depths of about 600 nm; surface-to-peak depths were about 400 nm. Initially, He^+ ions were implanted into the CLAM sample with fluence of 4×10^{15} ions/cm^2, followed by H^+ with fluences of 8×10^{15} and 2.4×10^{16} ions/cm^2. During all experiments, the mean temperature of samples was maintained below 473 K. Implantation conditions are listed in Table 2.

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, The Chinese Academy of Sciences. The equipment used a HpGe detector mounted perpendicular to the beam direction. The positron annihilation spectra obtained were characterised by the conventional *S* and *W* parameters, the *S* parameter is defined as the ratio of counts in the central energy region around 511 keV to the total counts of the entire spectrum, the *W* parameter is the ratio of the wing area to the total area under the entire spectrum [15,16]. The *S* and *W* parameters yield information on low-momentum and high-momentum electrons, respectively, within materials.

By varying the incident positron energies, the depth dependence of both *S* and *W* can be obtained. Variable mono-energetic positron beams from 0.1 to 30 keV were used to determine the depth profile of defects in CLAM steel after ion implantation.

Table 2
Conditions of ion implantation. All implantations were performed at room temperature.

Sample	Ions	Current density (A/m^2)	Fluence (ions/cm^2)
1	He^+	0.021	4×10^{15}
	H^+	0.016	8×10^{15}
2	He^+	0.021	4×10^{15}
	H^+	0.013	2.4×10^{16}
3	He^+	0	0
	H^+	0	0

3. Results and discussion

The evolution of the *S* parameter as a function of incident energy of implanted positron for ion-implanted samples is shown in Fig. 1, and that of the *W* parameter in Fig. 2.

The mean implantation depth of the positron is defined by the incident energy and is calculated using the following established relation [17]:

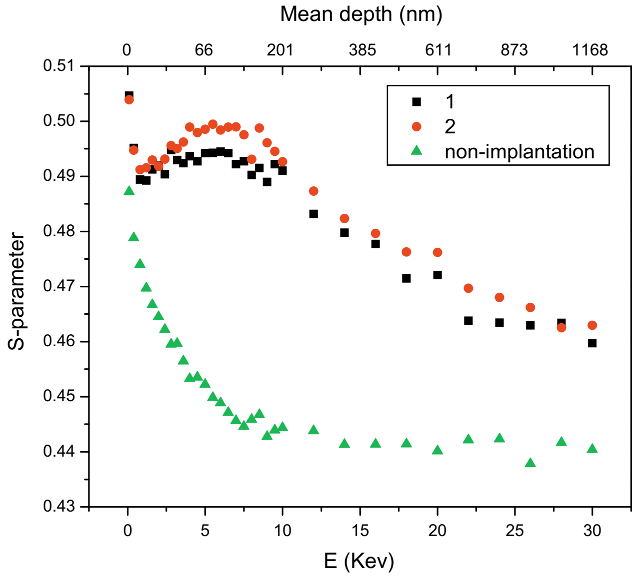

Fig. 1. Position annihilation with low-momentum conduction electrons in lattice defects increase in ion-implanted samples compared with the raw sample.

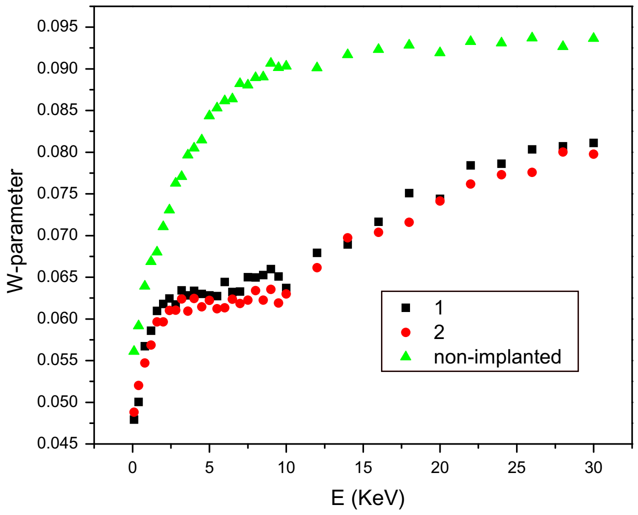

Fig. 2. Position annihilation with high-momentum core electron decrease in ion-implanted samples compared with the raw sample.