A STUDY OF THE HELIUM IMPLANTED INTO HR–1 STAINLESS STEEL BY PROTON ELASTIC SCATTERING

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ABSTRACT

Small discs of type HR- 1 austenitic steel ($0Cr_{17}Ni_{14}MnMo$) have been irradiated with 30- 170 keV He^+ for doses 10^{16}- $1\times10^{18}/cm^2$ at 300K. 2.5 MeV enhanced proton backscattering, TEM, SEM and CEMS are used to investigate the He trapping, bubble structures and the phase stability. It is found that a maximum He concentration of ~28 at. % was obtained after implantation with 70 keV He^+ at a dose just below critical. The micro- Vickers hardnesses of irradiated layers decrease with increasing dose, particularly when dense bubbles formed. The isomer shift of CEMS increases in the negative direction after irradiation. The austenite is believed to be stable against radiation induced martensitic transformation.

Keywords: Helium Ion implantation Proton elastic scattering Stainless steel

I . INTRODUCTION

Austenitic Fe- Cr- Ni alloys are candidate materials for tritium storage containers and reactor structures. Therefore understanding the behavior of helium produced by tritium decay, (n,α) reactions or α – particle bombardment and its influence on the properties of these alloys is of critical importance. He irradiation of metals and stainless steels have been studied extensively over the last 20 years [1-4]. However the overall behavior is not yet fully understood. It has been shown in our previous paper [5] that, for He implantation of type 316L stainless steel the target temperature has pronounced effects on the He depth profiles and the microscopic bubble structures.

In the present work, a further study has been made over an wider range of doses in order to elucidate the He trapping,bubble structures and their influence on the microhardnesses, and phase stability.

II . EXPERIMENTAL

The austenitic stainless steel HR- 1 studied has major constituents $0Cr_{17}Ni_{14}MnMo$. Small discs of this steel, 20mm in diameter and 2mm thick, were prepared as described in Ref [5] before irradiation. He ions were implanted with energy 30- 170 keV (mostly

70 keV) for doses of 10^{15}– $1\times10^{18}/cm^2$ at 300K. The target temperatures were controlled.

The He depth profiles were examined by means of 2.5 MeV proton elastic backscattering [6]. This permits detection of He concentration as low as 1 at. % within the first 10μ m of the surface layers. The depth resolution was better than 40nm by using 60° tilt incidence. The error of He concentration determination was $\pm10\%$.

The TEM foils were prepared by masking the irradiated surface of discs and backthinning. A JEM– 100cx electron microscope was used. The bubble diameters and the number density were measured on the TEM micrographs. For typical specimens conversion electron Mössbauer spectra (CEMS) were taken at room temperature with ^{57}Co source. The probing depth of backscattered electrons is 200– 300nm which is about the implantation range of 70 keV He^+. In addition, microhardnesses of the irradiated layers were measured using an applied load of 5 g.

Ⅲ. RESULTS AND DISCUSSION

1) Helium trapping Some typical depth profiles after implantation with 70 keV He^+ for various doses at 300K are shown in Fig.1. It is noted that for the doses $\Phi<\Phi_c$ ($\Phi_c \cong 6\times10^{17}/cm^2$, is the critical dose for blistering) the profiles are essentially Gaussian- like and the projected range (~220nm) is in good agreement with the range data calculated with TRIM program (230nm). In Fig.2 the total amount of retained helium is plotted as a function of ion dose. When $\Phi<\Phi_c$, He is efficiently trapped so that the retained He, Q, and the peak concentration, C_{He}, increase linearly with increasing Φ. For $5.8\times10^{17}/cm^2$ which is just below Φ_c, a maximum C_{He}, of about 28 at. % is obtained (i.e. $N_{He}/N_{\mathrm{M}}\cong38\%$ in Fig.1). When $\Phi>\Phi_c$, the surface blistering and flaking occur, Q deviates from the linear relation and changes irregularly near a level of $\sim5\times10^{17}/cm^2$. Meanwhile the profiles lowered and broadened, and the maximum concentration shifted towards the surface. But the profiling is less accurate in this case due to the surface roughness. Similar phenomena have been observed in Cu[7] where the He concentration peaks before the onset of blistering, and after blistering it decreases to a lower stationary value.

2) He bubble structures and microhardnesses Two typical TEM micrographs of He bubbles are shown in Fig.3. In Fig.4 the average diameters D_b and projected bubble densities N_b are given as a function of dose. The microhardness of He irradiated surface layers relative to that of an unimplanted region of the same sample is plotted versus dose in Fig.5. We didn't find bubbles when $\Phi<1\times10^{16}/cm^2$. Then D_b and N_b increase as the doses increase up to $3\times10^{17}/cm^2$. Above this point, more and more bubbles become linked. An interconnected network of microchannels eventually formed at $5\times10^{17}/cm^2$ (Fig.3*b*). The variation of relative microhardnesses can be related to the changes of bubble structures. The hardnesses decrease significantly in the dose range 1×10^{17}– $3\times10^{17}/cm^2$. After that a steady value is obtained corresponding to the formation of linked bubbles.

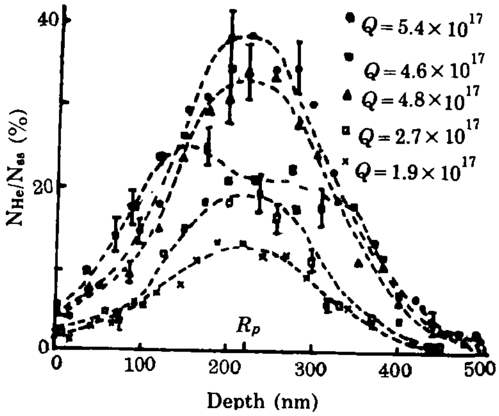

Fig. 1 He depth profiles of HR- 1 SS after 70 keV He$^+$ implantation at 300K

× 2×10^{17}/cm² □ 3×10^{17}/cm² ▲ 5×10^{17}/cm² ● 5.8×10^{17}/cm² ■ 7×10^{17}/cm² Q- - retained He in at./cm²

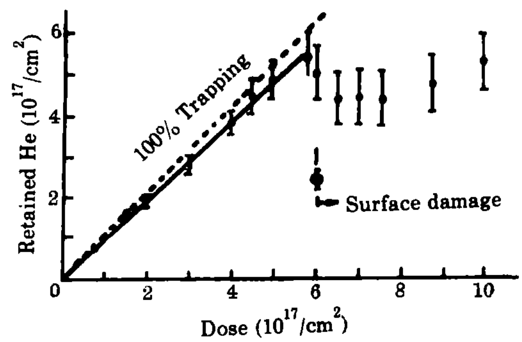

Fig.2 Total amount of retained He$^+$ in HR-1 SS vs implantation dose. 70 keV, 300K

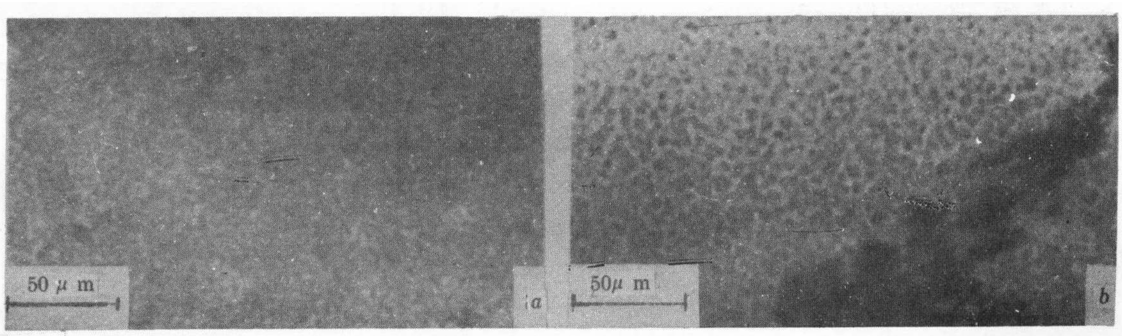

Fig. 3 TEM micrographs of HR-1 SS irradiated with 70 keV He$^+$ at 300K

a) 1.5×10^{17}/cm² *b*) 5.0×10^{17}/cm²

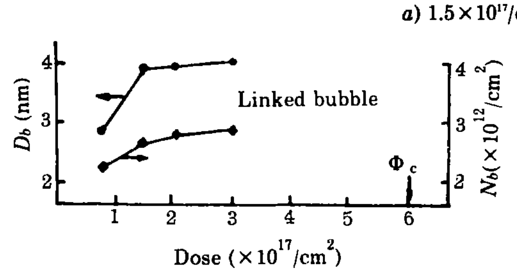

Fig.4 The average He bubble diameters and areal number densities in HR-1 SS as a function of dose

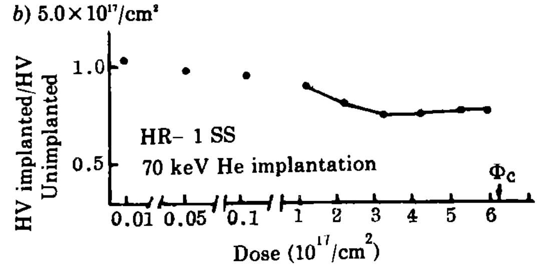

Fig.5 The relative micro-Vickers hardness vs implantation dose for HR-1 SS

It has been reported[8] that a radiation hardening of about 75 MPa was found by measuring micro- Vickers hardness for type 316 SS irradiated with 24 MeV He$^+$ to 3.2 $\times 10^{16}$/cm². The authors attributed that to the dislocation loops. Obviously it is not the case in the present work. We tend to assume that He bubbles dominate in the surface layers after irradiation with 70 keV He^+ for $\Phi \sim 10^{16}/cm^2$, which leads to the decrease of microhardness.

3) *CEMS* All of the CEMS taken from HR- 1 show a single pronounced peak of the paramagnetic austenite. However, the peaks for irradiated surfaces have negative isomer shifts compared to α - Fe and the absolute value of *I.S.* increases with increasing dose for $\Phi < \Phi_c$ (Table 1)

Table 1

Isomer shifts of HR- 1 SS irradiated with 70 keV He

Φ (10^{17} He^+/cm^2)	0	0.35	3	5	7	10
I.S.(mm/s)	- 0.07+0.02	- 0.08+0.02	- 0.12+0.02	- 0.15+0.02	- 0.11+0.02	- 0.12+0.02

According to the formulation[9], $I.S. = \text{const}\{|\psi(0)|^2 - \text{const}\}(\delta R/R)$, where $\delta R/R$ is relative change of the nuclear radius in going from the excited state to the ground state, and it is negative for Fe. So the observed change of *I.S.* in the negative direction indicates an increase of $|\psi(0)|^2$, which is the total relativistic *s*- electron density at the absorber Mössbauer nuclides. This increase coulde be explained by considering the existence of irradiation vacancies in the austenite lattices. Since the vacancies are electrically negative, they repulse valence electrons and hence lead to the increase of $|\psi(0)|^2$. A probable lattice contraction due to surrounding overpressurized He bubbles could also be the reason. However it needs experimental proof.

The $\gamma - \alpha$ transformation has been observed in 304 SS after implantation with $8\times 10^{17}/cm^2$ of 40 keV He^+ using CEMS[10]. It is noted that the temperature increased to ~ 200°C during implantation. However, in the present work no discernible contribution of any ferromagnetic phase could be identified for HR- 1 SS after irradiation for dose up to $1\times 10^{18}/cm^2$. Therefore we tend to conclude that the austenite remains stable under these conditions though TEM and SEM revealed significant damage in the surface layers.

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