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Implanted Stainless Steel

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G. J. Thomas, K. L. Wilson

Sandia Laboratories

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Microstructure of Low Energy Deuterium
Implanted Stainless Steel*

G. J. Thomas
Theoretical Division 8341
K. L. Wilson
Physical Research Division 8347
Sandia Laboratories, Livermore

ABSTRACT

Transmission electron microscopy has been used to determine the defect structure produced in stainless steel by deuterium ions of energies from 0.3 to 5 keV. These energies are typical of neutral atoms incident on the first wall of current tokamak devices. At room temperature, high defect densities were found at our lowest dose of $1 \times 10^{15} \text{ D}^+/\text{cm}^2$. With increasing dose, the defect structures coarsened, and lattice strain effects in the implanted region became noticeable. Following low temperature implantation, no gas bubbles were observed for deuterium concentrations as high as ~ 10 atom percent.

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I. Introduction

It is generally recognized that the first wall of tokamak fusion reactors will receive a flux of energetic neutral hydrogen isotopes produced by charge exchange at the plasma edge. Calculations of the energy distributions of these particles suggest they will have mean energies ≤ 1 keV.⁽¹⁾ Hydrogen, which has a finite solubility in stainless steel, may not precipitate into gas bubbles, such as observed following helium implantations⁽²⁾, for typical tokamak wall fluxes. Displacement effects, however, will generate point defects and defect clusters. This damage will alter the microstructure of the metal and may act as trapping sites for gas atoms. The fraction of injected gas atoms retained by the wall material can be a significant factor affecting hydrogen isotope recycling and tritium inventory. Several gas retention measurements have recently been made⁽³⁻⁶⁾ to determine the magnitude of this effect.

The purpose of the present study is to examine, with transmission electron microscopy, the defect structure produced in the near-surface region of stainless steel by low energy deuterium ions, and to correlate this structure with gas trapping and retention measurements. In addition, low temperature implantations were performed to look for the effects of high gas concentrations. Previous studies have shown that blistering occurs in stainless steel⁽⁷⁾ implanted with hydrogen at low temperatures (≤ 200 K), where the gas mobility is sufficiently reduced to achieve high concentrations in the implanted layer. Since all of our implantations were performed at room temperature or below, our results apply mainly to near-term devices, such as TFTR.

II. Experimental

Specimens of 316 stainless steel were thinned for transmission microscopy before implantation. Monoenergetic deuterium ions were implanted to

doses ranging from 1×10^{15} to 1×10^{18} D^+/cm^2 at room temperature and 77K. Samples implanted at 77K were warmed to room temperature for study. Ion energies of 0.3, 1, and 5 keV were obtained by deceleration of higher energy beams; D_3^+ beams at three times the desired energy were employed at 1 keV and below. Current density during implantation was typically $10 \mu A/cm^2$, corresponding to a flux of 6×10^{13} D^+/cm^2 sec.

Ion stopping distributions for deuterium in iron have been calculated in the low energy regime by Haggmark⁽⁸⁾ using Monte Carlo techniques⁽⁹⁾. Values of the range (depth of peak concentration) and the peak width at half maximum points (FWHM) for the energies used in this study are shown in Table I. The FWHM parameter is used to describe the peak width because the stopping profiles are skewed from the normal gaussian distributions. It can be seen from Table I that the two lower energy implants will affect only the near-surface portion of the sample foil (foil ~ 100 nm thick), whereas in the 5 keV case the FWHM is of the order of the sample thickness.

111. Results

In general, low energy D^+ implantations produced high densities of small defect clusters. Figures 1a and b show two examples of 1 keV D^+ implantations at room temperature for doses of 1×10^{16} and 1×10^{17} D^+/cm^2 , respectively. Taking account of the ion range and extinction distance, the dominant black-white contrast direction observed under strong two-beam imaging conditions indicate the clusters to be interstitial loops at 1 keV. Detailed analyses of the defects with stereomicroscopy were not performed. At a given energy (in this case 1 keV), increasing the dose tended to reduce the loop density, but increase the loop diameter. This coarsening was most noticeable between dose levels of 10^{16} and 10^{17} D^+/cm^2 . In addition, at 10^{17}

D^+/cm^2 and above, curling of the foil edges was obvious, as shown in Fig. 2 for a dose of $1 \times 10^{18} cm^{-2}$, indicating substantial strain within the ~ 20 nm implanted layer. This effect was not observed at 5 keV.

The concentrations of point defects visible in clusters were estimated from measurements of loop densities and diameters for all of the implant conditions. These results are summarized in Table II. Due to uncertainties in measuring high cluster densities and small cluster sizes, these values may be uncertain by as much as a factor of two and are intended mainly for comparison purposes. The FWHM values shown in Table I were assumed for the implanted layer thicknesses at 0.3 and 1 keV. It can be seen that a relatively high defect concentration is produced at low doses at all of the energies considered. In the 1 keV case, the defect concentration remained essentially unchanged at about 0.3 atom percent up to $1 \times 10^{16} D^+/cm^2$, but increased substantially at $1 \times 10^{17} D^+/cm^2$. Measurements were not made at $1 \times 10^{18} D^+/cm^2$ since the clusters had interconnected into a dense dislocation network. At similar doses, higher energies produced lower loop densities, but the visible defect concentrations were not significantly different since the loops were larger.

After implantation at 77K, samples were warmed to room temperature for observation. No gas bubbles were found, even in the highest dose case ($7.5 \times 10^{16} D^+/cm^2$) which was expected to reach ≥ 10 atom percent deuterium concentration in the implanted layer. In comparison to similar doses implanted at room temperature, the low temperature implanted samples showed somewhat higher loop densities with smaller loop sizes. The visible defect concentrations were similar at both temperatures, as seen in Table II.

IV. Discussion

At room temperature, vacancies should have negligible mobility in stainless steel, so that only interstitial defects and the implanted gas atoms should be mobile during implantation. Thus, interstitial atoms could form visible clusters, whereas an equal number of vacancies and vacancy complexes not annihilated by recombination may be present submicroscopically. At higher doses, where μ pa levels should exceed unity, athermal clustering of neighboring vacancies could lead to the formation of larger vacancy aggregates. The gas mobility is not nearly as large as that of the interstitial; a diffusion rate of ~ 10 nm per second at room temperature is estimated from published hydrogen diffusivity values⁽¹⁰⁾.

Our observations show that a high density of defect clusters were produced at relatively low dose levels ($\sim 10^{15}$ D⁺/cm²). For comparison, one discharge in TFTR is estimated to bombard the wall with $\sim 1 \times 10^{16}$ neutrals/cm² of mean energy ~ 200 eV. With increasing dose, it was found that the clusters grew and coarsened, most noticeably between 1×10^{16} and 1×10^{17} D⁺/cm². This increase may be due, in part, to oxide removal resulting in increased ion penetration into the metal. Lattice strain within the implanted layer also became noticeable within the same dose range. It is interesting to note that a significant increase in trapping of deuterium was observed by gas desorption measurements⁽⁶⁾ in stainless steels at dose levels of 5×10^{16} D⁺/cm² and above. Thus, it is possible that submicroscopic vacancies may be trapping sites for gas atoms, and the strain observed at high doses can then be attributed to the trapped gas atoms. The required concentrations of hydrogen traps are not inconsistent with the observed defect concentrations. Although this explanation is plausible, additional evidence would be needed for verification.

The increased cluster density at low temperatures is likely due to an increased nucleation probability. As seen in Table II, the defect concentrations were not significantly different from room temperature implantations. No gas bubbles were observed in our samples, however, at a concentration of ≥ 10 atom percent of immobile deuterium. As mentioned earlier, surface blistering has been observed with low temperature hydrogen implantation in stainless steel⁽⁷⁾ at a dose where the estimated peak gas concentration assuming a gaussian profile, was ~ 40 atom percent. In contrast, bubbles have been observed at room temperature in helium implanted stainless steel at concentrations ≤ 1 atom percent.⁽²⁾ The helium concentration required for blistering is ~ 30 atom percent⁽¹¹⁾, similar to the hydrogen case. These results suggest that hydrogen blistering may be associated with stress build-up within a subsurface implanted layer and not to gas pressure.

In summary, a comparison of our data with the estimated first wall neutral atom flux in TFTR indicates that defect clusters should form in a ~ 10 nm layer of the first wall, with defect densities reaching ~ 1 atom percent after several discharges. After ~ 100 discharges, the surface layer should be highly strained, and the clusters coarsened into dense dislocation networks. With additional discharges, sputtering of the surface becomes important, and a steady state damage distribution should be attained in the sub-surface region. This description of damage and sputtering rates would be altered, however, if surface oxidation occurred between discharges or if redeposition of sputtered material were significant. Although trapping of hydrogen should occur within the damaged layer, gas concentrations should be too low to produce surface blistering. Gas bubbles would not be expected to form unless helium is generated from D-T fusion and, in this case, synergism between hydrogen isotope and helium effects may also play a role.

V. Acknowledgment

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Table I
Calculated Ion Ranges
(from Haggmark)⁸

Energy (keV)	Range (nm)	FWHM (nm)
0.3	4	7.5
1	12	21
5	48	72

Table II
Summary of Implantation Results

Energy (keV)	Temperature (K)	Dose (D ⁺ /cm ²)	Cluster Density (cm ⁻³)	Average Cluster Size (nm)	Visible Defect Concentration (atom %)
0.3	300	1×10^{16}	9×10^{18}	~ 2.0	0.5
1	300	1×10^{15}	3.5×10^{18}	2.5	0.3
1	300	1×10^{16}	1.5×10^{18}	4.0	0.3
1	300	1×10^{17}	4.5×10^{17}	10	1.0
5	300	1×10^{16}	2×10^{17}	7.5	0.3
5	300	1×10^{17}	1×10^{17}	10	0.3
1	77	1×10^{16}	2×10^{18}	3.5	0.3
1	77	7.5×10^{16}	6×10^{18}	4.0	1.5

References

1. S. A. Cohen, J. Vac. Sci. Technol. 13, 449, (1976).
2. G. J. Thomas and K. L. Wilson, Ans. Transactions, 27, 273, (1977).
3. R. E. Clausing, L. C. Emerson, L. Heatherly, and R. J. Colchin, Proc. Int. Symp. on Plasma Wall Interaction, Julich, Oct. 1976 (Pergamon Press, Oxford, 1977), p. 573.
4. C. Altstetter, R. Behrisch, J. Bottinger, F. Pohl and B. M. U. Scherzer, accepted for publication, Nucl. Instr. and Methods.
5. K. L. Wilson and M. I. Baskes, accepted for publication, J. Nucl. Mater.
6. K. L. Wilson and M. I. Baskes, this conference.
7. K. L. Wilson, G. J. Thomas, and W. Bauer, Nucl. Technol. 29 (1976), 332.
8. L. G. Haggmark, private communication.
9. J. P. Biersack and L. G. Haggmark, to be published.
10. T. S. Elleman and K. Verghese, J. Nucl. Mater. 53 (1974) 1299.
11. G. J. Thomas, to be published.

Figure Captions

Figure 1: Transmission electron micrographs of pre-thinned stainless steel samples implanted at room temperature with 1 keV D⁺ ions.

(a) 1×10^{16} D⁺/cm²

(b) 1×10^{17} D⁺/cm²

Figure 2: Low magnification transmission electron micrograph of pre-thinned stainless steel sample implanted at room temperature with 1 keV D⁺ ions to a dose of 1×10^{18} D⁺/cm². Note foldover of thin edge from implantation-induced strain.

$1 \times 10^{16} \text{ D/cm}^2$

1 keV RT

$1 \times 10^{17} \text{ D/cm}^2$

(a)

(b)

Figure 1

Figure 2