ELSEVIER

Journal of Nuclear Materials 271&272 (1999) 306–310

journal of nuclear materials

Surface morphology and void formation in 316L stainless steel irradiated with high energy C-ions

Z.G. Wang *, K.Q. Chen, L.W. Li, C.H. Zhang, J.M. Quan, M.D. Hou, R.H. Xu, F. Ma, Y.F. Jin, C.L. Li, Y.M. Sun

Institute of Modern Physics, Chinese Academy of Sciences, 730000 Lanzhou, People's Republic of China

Abstract

This work reports the study of changes of surface topography and bulk structure of 316L stainless steel (SS) irradiated at 773 K with 51.4 MeV C-ions to a fluence of 1.14×10^{22} ions/m^2. The calculated damage levels at the surface and at the damage peak position were 0.9 and 124 displacements per atom (dpa), respectively. The changes of surface topography and bulk structure were checked at room temperature by the use of scanning probe microscopy (SPM), scanning electron microscopy (SEM), 1 MV high voltage electron microscopy (HVEM) and transmission electron microscopy (TEM) with cross-section technique. The experimental results suggested that high dose carbon ion irradiation led to (1) serious pitting, flaking, and crazing along grain boundaries of the irradiated surface; (2) voids formed in the area around the damage peak and mean void swelling is about 4%. The void swelling data deduced from the SEM and TEM observations were the same within the experimental error. Furthermore, some phase change has been detected in the carbon ion stop region. All these observed phenomena were interrelated and have been discussed.

1. Introduction

The interaction of fast neutrons with solids results in a wide variety of phenomena including defect production, irradiation creep, void swelling, changes in surface topography, and so on. The confident design of nuclear power plants requires knowledge of these phenomena that develop in structural components during their service in radiation environments. Because radiation damage phenomena in reactors are a consequence of two elementary processes, that is, lattice atom displacement and helium production by (n,α) reaction, both processes can be reproduced separately or simultaneously by ion beam bombardment. Previous investigations of radiation phenomena at surfaces and in bulk induced by single ion impacts or by a certain dose of irradiation have been made using field ion microscopy (FIM) [1], transmission electron microscopy (TEM) [2] and scanning probe microscopy (SPM) [3]. Some more studies have been reviewed by Garner and Gells [4]. Unfortunately, the lack of studies on surface morphological changes associated with bulk structure variation made it difficult to completely evaluate and explain the fundamental aspects of irradiation occurring in a given material under a given set of irradiation conditions. The aim of the present work is to study surface morphology, void formation and the correlation between them in 316L SS under high energy heavy ion irradiation.

In the present work, changes of surface topography associated with bulk void swelling of 316L SS irradiated with 51.4 MeV C-ions have been studied experimentally. In the following, the experimental procedure was demonstrated at first, then experimental results were given and finally, the correlation between surface changes and bulk damage was discussed.

2. Experimental procedure

Solution-annealed and 20% cold-worked 316L SS sample (200 μm in thickness, preparation procedure was shown in Ref. [5]) was mounted on a sample holder

* Corresponding author. Tel.: +86-931 885 4867; fax: +86-931 888 1100; e-mail: hiam@ns.lbz.ac.cn.

0022-3115/99/$ – see front matter
PII: S0022-3115(98)00787-9

which could be heated with built-in tungsten coils by direct current and could be used to measure in situ the irradiated ion fluence. The sample was covered by a copper mask (1 mm in thickness) with a central hole of 8 mm in diameter. This sample was irradiated at 773 K with 51.4 MeV C-ions delivered from the injector of Heavy Ion Research Facility in Lanzhou (HIRFL) to an ion fluence of 1.14×10^{22} ions/m^2. During the irradiation, the C^{6+}-ion flux was limited to less than 5 mA/m^2 and the measured sample temperature fluctuations were within 15 K.

After irradiation, the sample was kept at room temperature. At first, surface morphological changes were checked by the use of SPM (EPM-810). Then the sample was prepared as cross-section specimens [6] which were used to study bulk void swelling and microstructural changes by the use of SEM (JEM-120X), TEM (JEM-2000FX) and HVEM (JEM-1000), respectively. According to the obtained micrographs, mean diameters of voids at different depths along the ion-incident direction were determined manually and the corresponding bulk void swelling was deduced.

Two methods were used to determine bulk void swelling. One based on SEM observations which was denoted as void swelling S_A and another based on TEM/HVEM observations which was denoted as void swelling S_B. They are

$$S_A = \frac{\Delta A}{A - \Delta A} \times 100\%, \quad (1)$$

$$S_B = \frac{\Delta V}{V - \Delta V} \times 100\%, \quad (2)$$

where A, V and ΔA, ΔV represent the measured area, volume and the parts of them occupied by voids, respectively. Specimen thickness under TEM/HVEM observations, t, was determined by diffraction fringes of an electron beam on the wedge foil. Because $V = tA$ and $\Delta V = t\Delta A$, S_A and S_B have the same meaning.

Theoretical distributions of damage level and doped C-atom concentration along the projectile range of the incident C-ion in 316L SS were calculated by the use of TRIM code.

3. Experimental results

3.1. Irradiation induced changes of surface topography

Fig. 1 shows the SPM photographs of surface morphological changes of 316L SS after carbon ion bombardments where (a) and (b) correspond to the shielded and the exposed regions, respectively. The comparison of them suggests that serious flaking and sputtering occurred in the exposed region. Fig. 2 shows the Y-axis modulation image (vertical scan modulated with video signal) of the fringe area of the irradiation spot. It shows that a horizontal step appeared at the boundary corresponding to the conversion from the shielded region to the exposed area, the exposed area as a plateau raised

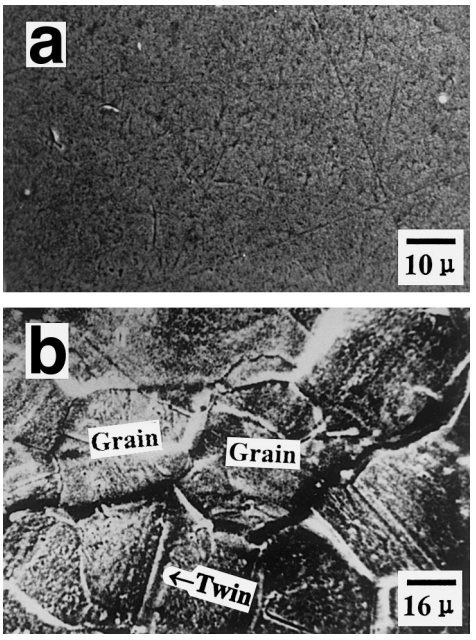

Fig. 1. SPM photographs of surface changes from unirradiated (a) to exposed (b) region of 316L SS sample surface bombarded at 773 K with 51.4 MeV C-ions to a fluence of 1.14×10^{22} ions/m^2. Serious flaking and sputtering occurred in the exposed region.

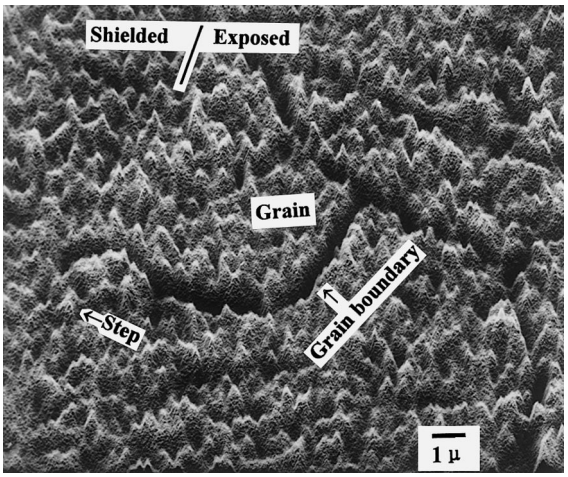

Fig. 2. Y-axis modulation SPM image obtained at the fringe area shielded region and exposed area. The exposed area is as a plateau raised from the initial surface and crazing along grain boundaries appeared on it.

from the initial surface, and cracks along grain boundaries occurred only in the exposed area.

3.2. Irradiation effects in the bulk

Fig. 3 shows the microphotographs of void formation in the irradiated sample by use of a cross-section technique [6]. Fig. 3(a) is a SEM image which represents an overview of void distribution along the direction of the incident C-ion beam. Voids scattered within 2.3 μm (damage level from 5.3 to 63.0 dpa) and the strip-like void area parallel to the irradiated surface. Fig. 3(b) is a HVEM image corresponding to the area of voids with dense number density. The measurements and analyses yield average void swelling $S_A = 3.9\%$ and $S_B = (4.23 \pm 0.52)\%$ which are of the same value with the experimental error. The measured void swelling peak position is located at about 25.1 μm ($S_B = 6.4\%$) whereas theoretical dpa peak position should be at about 26.4 μm. There is a significant position difference between these two peaks. Fig. 4 shows the measured void number distribution versus void diameter. Most voids are of one to two hundred nanometers but the size of little voids can reach up to a magnitude of 1 μm.

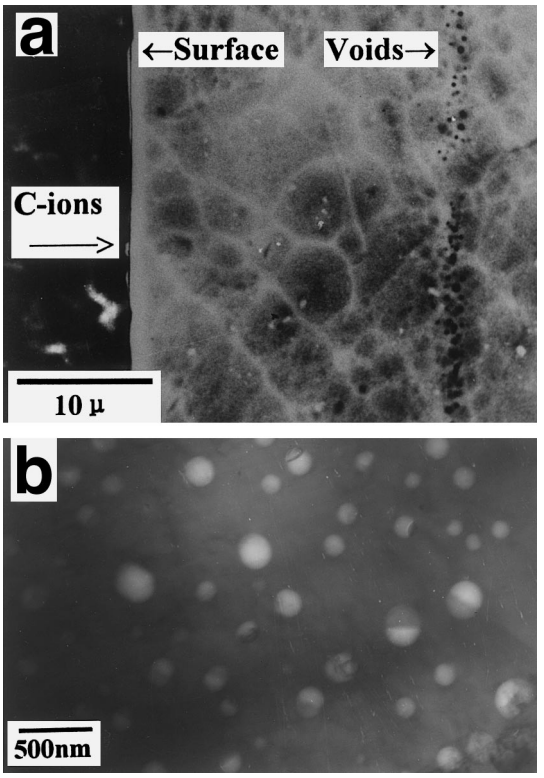

Fig. 3. SEM (a) and HVEM (b) microphotographs of void formation in the irradiated 316L SS: (a) represents an overview of void distribution along the direction of the incident C-ion beam and (b) shows the area of voids with dense number density.

4. Discussion

4.1. Surface morphology and void swelling

The significant surface morphological changes and void swelling have been shown in Section 3. For void swelling measurements, the agreement between S_A and S_B values suggests that both SEM and TEM/HVEM can be used to detect void swelling, especially for the condition that the formed voids are of relatively large diameters. The accuracy of S_A is mainly limited by the resolution of SEM and the accuracy of S_B is mainly limited by the determination of the thickness of the analyzed specimen. For the surface morphological changes, the flaking and cracking that occurred at the surface were driven by an anisotropic compressive stress limited in the damaged volume and released at the exposed area. Here, the exposed surface was free. The anisotropic compressive stress state was only determined by the ratio of the local swelling rate and the creep compliance. Because the total volume change was accommodated by movement of mass perpendicular to the sample surface [7,8], the height h of the plateau raised from the initial surface could be given by a rough estimation

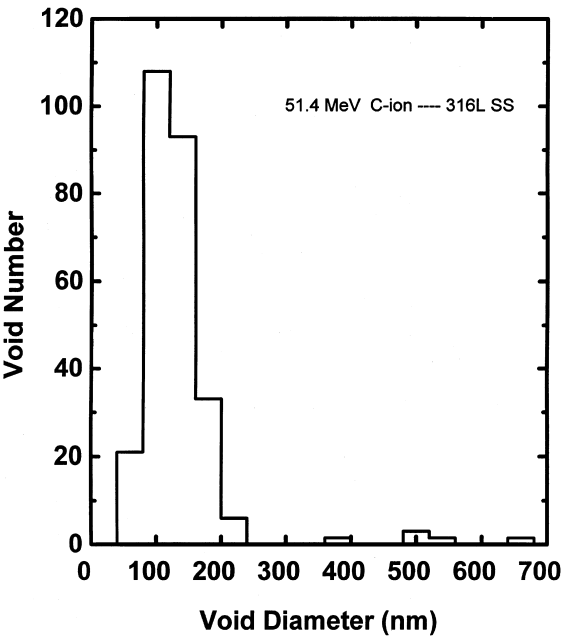

Fig. 4. The measured void number distribution versus void diameter. Most voids are of one to two hundred nanometers.

$$h = \frac{S_A}{1 + S_A} \Delta R, \quad (3)$$

where ΔR is the visible void's spreading depth width along the incident ion path and S_A the average void swelling in ΔR. In the present study, $S_A = 3.9\%$, $\Delta R = 2.3$ μm and we have $h = 90$ nm.

4.2. Shift of swelling peak

In principle, large void swelling corresponds to the region that was damaged due to a large dpa value. However, the above experimental observations show that there is a significant difference between the measured void swelling peak position (25.1 μm) and the theoretical dpa peak position (26.4 μm). This was mainly due to from two reasons:

(1) Surface flaking led to the measured void swelling peak position shifting towards the irradiated surface. As was shown in Fig. 1(b), a step of about 1 μm height existed between different grains which implies that a certain thickness of the initial surface was corroded by the incident ion beam. The removed thickness corresponds to the forward shift in the measured void swelling peak position.

(2) Doping in the carbon atom stop region. For 51.4 MeV C-ion irradiation on 316L SS, theoretical peak position of carbon atom deposition is at about 26.7 μm (FWHM ~1 μm, carbon concentration 1.6×10^{28} atoms/m^3). The diffusion of the implanted carbon atoms results in the combination of carbon atoms with the vacancies created by cascade collisions between incident ions and the target atoms. It can decrease the growth rate of void formation [9,10]. Furthermore, high concentration of doped carbon atoms will result in a new phase change formation. Fig. 5 shows the carbon enriched phase formed in the carbon atom stop region. The existence of carbonates leads to a decrease of void swelling [10]. Thus, the doping of the implanted carbon atoms can also result in the shift of the void swelling peak position to the irradiated surface.

5. Summary

Surface topography and bulk structure changes of 316L SS have been studied experimentally. The experimental results suggested that high dose carbon ion irradiation led to (1) serious pitting, flaking, and crazing along grain boundaries of the irradiated surface; (2) mean void swelling around the damage peak is about 4%. Both SEM and TEM observations were used to determine void swelling and the deduced data are the same with minor experimental error. The flaking and cracking that occurred at the surface were driven by an anisotropic compressive stress and correlated with the total volume change. The shift of the swelling peak mainly resulted from surface flaking and carbon atom doping in 316L SS.

Acknowledgements

The authors would like to acknowlege all the staff working in the operation group of the injector of

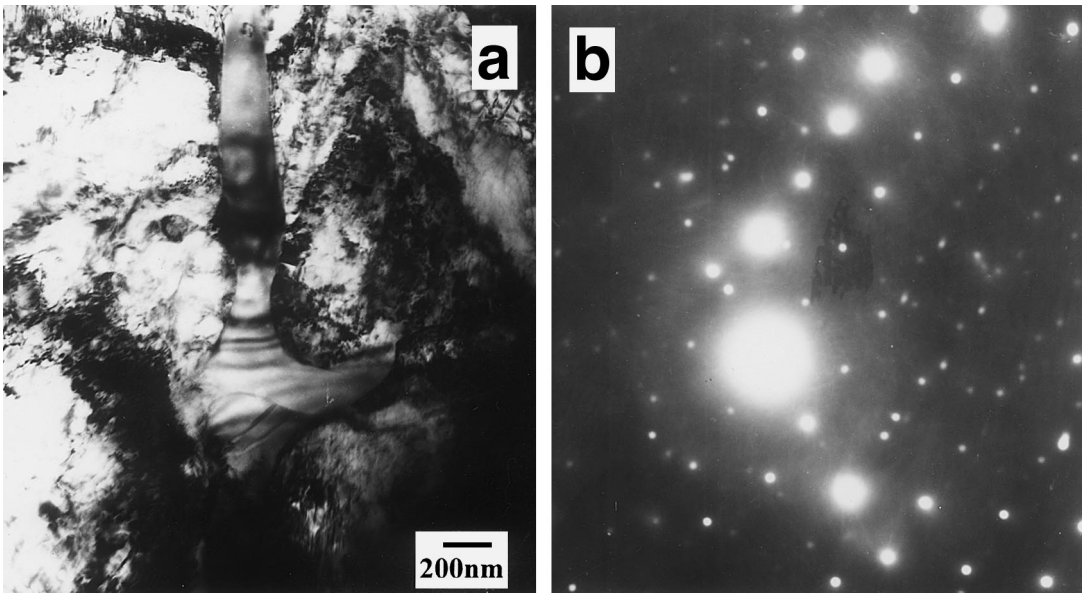

Fig. 5. Carbon enriched new phase ((a) pillar-shaped part; (b) diffraction pattern) formed in the carbon atom stop region.

HIRFL for their good cooperation during the irradiation experiment, Professor Sun Jiguang, Professor Fang Kemin and Professor Zhao Jiazhen for their help on electron microscopy observations, and also the Hybrid Reactor Committee of China for the financial support.

References

[1] J.M. Walls, A.D. Martin, H.N. Southworth, Surf. Sci. 50 (1975) 360.
[2] K.L. Merkle, W. Jager, Philos. Mag. A 44 (1981) 741.
[3] C. Teichert, M. Hohage, T. Michely, G. Comsa, Phys. Rev. Lett. 72 (1994) 1682.
[4] F.A. Garner, D.S. Gells, J. Nucl. Mater. 159 (1988) 286.
[5] K.Q. Chen, S.H. Yang, Z.G. Wang, J.G. Sun, J.M. Quan, L.W. Li, Y.M. Sun, J. Nucl. Mater. 191–194 (1992) 737.
[6] W. Zhiguang, C. Jiachao, C. Keqin, Ann. Rep. 1989, Institute of Modern Physics, Chinese Academy of Sciences, p. 149.
[7] F.A. Garner, G.L. Wire, E.R. Gilbert, in: Radiation Effects and Tritum Technology for Fusion Reactors, CONF-750989, vol. 1, Gatlinburg, TN, 1975, pp. 474–490.
[8] W. Primak, J. Appl. Phys. 35 (1964) 1342.
[9] F.A. Garner, J. Nucl. Mater. 117 (1983) 177.
[10] K. Nakata, Y. Katano, I. Masaoka, K. Shiraishi, J. Nucl. Mater. 122&123 (1984) 630.