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Variation of Radiation Damage in Stainless Steel with Temperature and Dose*

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Abstract: Dependence of radiation damage in the modified 316L stainless steel has been investigated on irradiation temperature from room temperature to 802 ℃ at 21 and 33 dpa and on irradiation dose up to 100 displacemets/atom(dpa) at room temperature by the heavy ion irradiation simulation and positron annihilation lifetime techniques. A radiation swelling peak was observed at ~580 ℃ where the vacancy cluster contains 14 and 19 vacancies and has an average diameter of 0.68 and 0.82 nm, respectively for the 21 and 33 dpa irradiations. The size of the vacancy clusters increases with the increasing of irradiation dose, and the vacancy cluster produced at 100 dpa consists of 8 vacancies and reaches a size of 0.55 nm in diameter. The experimental results show that the radiation damage in this modified 316L stainless steel is more sensitive to irradiation temperature.

Key words: modified 316L stainless steel; radiation damage; positron annihilation lifetime techniques; heavy ion irradiation simulation

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1 Introduction

Stainless steel (SS) is an important target structural material for spallation neutron source. Spallation neutron source is one of the key parts of the accelerator driven system (ADS) [1-3], which provides source neutrons to drive a sub-critical assembly. As a target structure material of spallation neutron source, SS is irradiated by high-energy and intense protons and/or neutrons during operation. The accumulated displacement damage dose per year is estimated to be a couple of hundred displacemets/atom (dpa) in ADS. Severe radiation damage in SS would lead to a breakdown or accident of ADS. Therefore, investigation of radiation damage in SS at such high doses is of great importance for the design and safe operation of ADS. It is desirable to use SS with good radiation resistant properties as the target structural materials of spallation neutron source.

Radiation damage produced in SS depends on the irradiation temperature and dose. The present work was motivated to investigate variation of radiation damage in the home made modified 316L stainless steel with irradiation temperature and dose. No available neutron and proton sources can be employed directly in laboratory to study radiation damage at such high doses encountered in

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ADS. The radiation damage rate of heavy ions is much higher than that of neutrons and protons and it just takes minutes or hours to reach tens of dpa by energetic heavy ion irradiation[4], which makes it possible to investigate the radiation damage at high doses. Therefore, in the present study the heavy ion irradiation was adopted to simulate the high dose proton and neutron irradiation. The positron annihilation lifetime technique was utilized to look at the produced radiation damage microscopically.

2 Experimental

The SS samples used in the experiment were the home made modified 316 austenitic stainless steel (MSS), the size of which was ϕ23 mm $\times$0. 5 mm. The samples were mechanically polished to a mirror-like surface. MSS was made of Cr-15. 05%, Ni-14. 76%, Ti-0. 32%, P-0. 007%, S-0. 007%, Mn-1. 78%, Si-0. 52%, C-0. 048% and Fe balanced to 100% and treated by the 20% cold working. The cold-working and Ti-addition aimed at improving radiation resistant property, especially reducing the radiation swelling.

In the measurement of temperature dependence the MSS samples were irradiated by the 70 MeV carbon ions from the HI-13 tandem accelerator at China Institute of Atomic Energy. The irradiation temperature ranged from room temperature to 802 ℃, and the temperature accuracy was $\pm$5 ℃. The temperature dependence was performed at two irradiation doses of 21 and 33 dpa. In the measurement of dose dependence the MSS samples were irradiation by the 80 MeV fluorine ions up to 100 dpa at room temperature. The displacements per atom introduced by the heavy ion irradiation in the sample were calculated by a TRIM program[5,6].

The radiation damage generated in the samples was examined by a positron annihilation lifetime technique. The positron lifetime measurements were performed at room temperature for the un-irradiated samples and the samples irradiated at different irradiation temperatures and doses. A fast-fast coincidence positron lifetime spectrometer with a pair of BaF_2 scintillation detectors was used, the time resolution of which is 210 ps to ^{60}Co γ rays. Two identical samples irradiated at the same condition were arranged as a sandwich with a 1. 1 MBq ^{22}Na positron source in the center. Besides the source components, all measured positron lifetime spectra were fitted by a PATFIT[7] or LT[8] program with two lifetime components τ_1 and τ_2, and the fitting variance was less than 1. 3.

3 Results and Discussion

3.1 Dependence of irradiation temperature

The annihilation lifetime τ_f of free positrons is 110 ps in SS and the annihilation lifetimes of positrons trapped at the mono-vacancy, di-vacancy and dislocation are $\tau_{1v}=1.3\ \tau_f$, $\tau_{2v}=1.5\tau_f$, and $\tau_{dis}=$ 169 ps, respectively [9]. In the data analysis, τ_1 is assumed to be a weighted average of annihilation lifetimes of the free positrons and the positrons trapped at mono-and di-vacancies and dislocation, and τ_2 is attributed to small vacancy clusters or voids.

The temperature dependence of τ_1 and τ_2 was measured at first for the un-irradiated MSS samples in order to know the variation of the intrinsic defects with annealing temperature. $\tau_1=$ 147 ps and $\tau_2=$ 271 ps were obtained at room temperature. τ_1 decreases with increasing the annealing temperature and arrives at 110 ps at 800 ℃, and τ_2 approaches to 255 ps at 800 ℃.

The dependence of τ_1 and τ_2 on irradiation temperature is shown in Fig. 1 for 21 dpa irradiation. At room temperature τ_2 is almost the same as the one in the un-irradiated samples, while τ_1 is 155. 3 ps. Both τ_1 and τ_2 reach their peak values of 157. 4 ps and 373. 0 ps at the irradiation temperature of 580 ℃. At 802 ℃ $\tau_1=128.2$ ps and $\tau_2=$ 307. 1 ps, and both of them are larger than the values of τ_1 and τ_2 in the un-irradiated sample annealed at 800 ℃. It can be seen from Fig. 1 that the mono- and di-vacancies, dislocation and different-size vacancy clusters (or voids) were produced in the MSS irradiated by 70 MeV carbon ions to a dose of 21 dpa at different irradiation temperatures. The relative intensities of the mono- and di-vacancies and dislocation decrease with the increasing of the irradiation temperature. The dependence of τ_2 on irradiation temperature shows that the biggest vacancy cluster appears at 580 ℃.

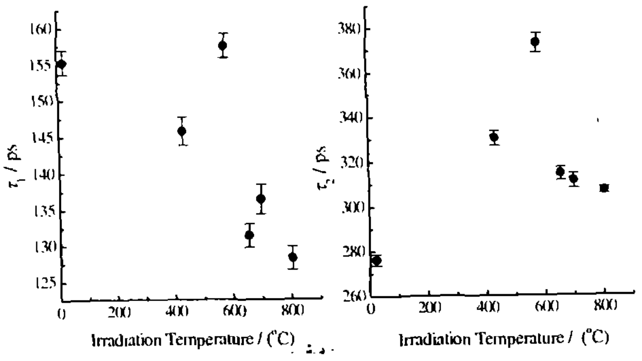

Fig. 1 Positron annihilation lifetimes τ_1 and τ_2 as a function of irradiation temperature in MSS irradiated by 70 MeV carbon ions to 21 dpa.

Fig. 2 shows the temperature dependence of positron lifetime τ_2 in MSS irradiated to 21 and 33 dpa. It can be seen that before the peak temperature the variation of positron lifetime τ_2 with irradiation dose increases with the increasing of irradiation temperature. The higher the irradiation temperature, the larger the increase of lifetime τ_2 with irradiation dose. This indicates that the radiation damage depends on irradiation temperature more sensitively than on irradiation dose.

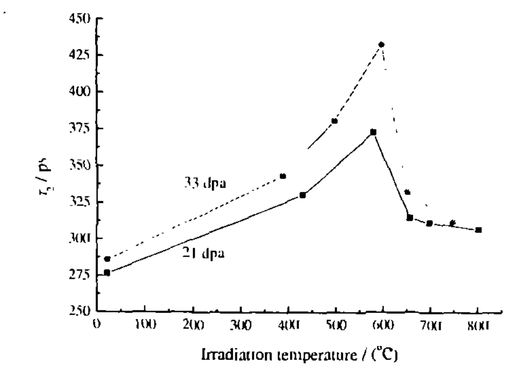

Fig. 2 Temperature dependence of positron lifetime τ_2 in MSS irradiated to 21 and 33 dpa.

The radius of voids can be estimated from the measured lifetime τ_2 by $R_v = (NZ)^{1/3}\ r_s$ or $R_v = (N)^{1/3} R_{ws}$[10,11], where R_{ws} is the Wigner-Seits radius, N is the number of vacancies in a void and can be assigned by the lifetime τ_2, Z is the valence number, $r_s = (0.75\ \pi n)^{1/3}$ is the density parameter in the unit of Bohr radius a_0 and n is the number density of conduction electrons. For iron $r_s = 2.12\ a_0$, $Z = 2$ and $R_{ws} = 2.67\ a_0$[12,13]. Fig. 3 shows the calculated average diameter of the clusters observed at different irradiation temperatures up to 802 ℃ for the 21 and 33 dpa irradiations. The size of the vacancy cluster or radiation swelling depends on irradiation temperature significantly, and the radiation swelling peak was observed at ～580 ℃ where the corresponding void contains 14 and 19 vacancies and has an average diameter of 0.68 and 0.82 nm, respectively for 21 and 33 dpa irradiations.

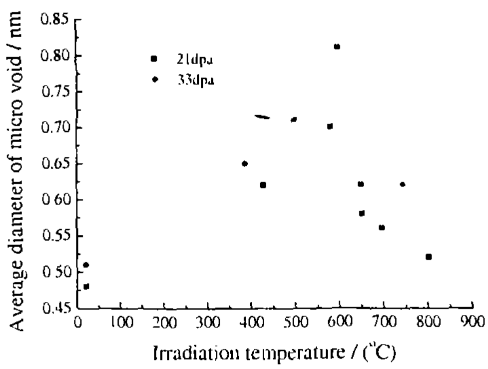

Fig. 3 Dependence of void diameter on irradiation temperature in MSS irradiated to 21 and 33 dpa.

The swelling peak takes place usually in the temperature region from 450 ℃ to 650 ℃, depending on the SS type, processing procedure, etc. This swelling peak can be understood easily. At lower irradiation temperatures the created defects are less mobile and the probability to form larger clusters is small, and at higher irradiation temperatures the defects annealing occurs. Thus, a swelling peak arises in an intermediate temperature. Though a swelling peak was detected, the swelling in MSS is much smaller than that in normal stainless steels. The void with an average diameter of

25.8 nm was determined for SS irradiated at 560 ℃ to a dose of 3.2×10^{22} n/cm^2 [14], which is equivalent to the present irradiation dose.

3.2 Dependence of irradiation dose

The dependence of positron annihilation lifetimes τ_1 and τ_2 and their relative intensities I_1 and I_2 on irradiation dose is shown in Fig. 4 for MSS irradiated at room temperature by 80 MeV fluorine ions up to 100 dpa. It can be seen that the positron lifetimes τ_1 and τ_2 and the relative intensity I_2 increase with the increasing of irradiation dose. This reveals that irradiation generates the mono- and di-vacancies, dislocation and vacancy clusters. Because the lifetime τ_1 is a weighted average, the increase of τ_1 means that the relative intensities of the produced mono- and di-vacancies and dislocation rise with the increasing of irradiation dose. The lifetime τ_2 is closely connected to the size of vacancy clusters or voids. The longer the lifetime τ_2, the larger the vacancy cluster size. The increase of τ_2 indicates the formation of larger size vacancy clusters. The vacancy cluster with 8 vacancies and a diameter of 0.55 nm was obtained at 100 dpa.

In summary, dependence of radiation damage in the home made modified 316L stainless steel has been investigated on irradiation temperature from room temperature to 802 ℃ and on irradiation dose up to 100 dpa. The variation of the positron annihilation lifetime τ_2 with irradiation temperature shows a peak at ~580 ℃ where the biggest vacancy cluster is observed. This biggest vacancy cluster contains 14 and 19 vacancies and has an average diameter of 0.68 and 0.82 nm, respectively for the 21 and 33 dpa irradiations. The size of the vacancy clusters increases with the increasing of irradiation dose, and the vacancy cluster possesses 8 vacancies and a diameter of 0.55 nm at 100 dpa. The variation of radiation effects with irradiation dose increases with the irradiation temperature, and the radiation damage in MSS is more sensitive to irradiation temperature than to irradiation dose. The experimental results demonstrate that this home made modified 316L stainless steel has a good radiation resistant property.

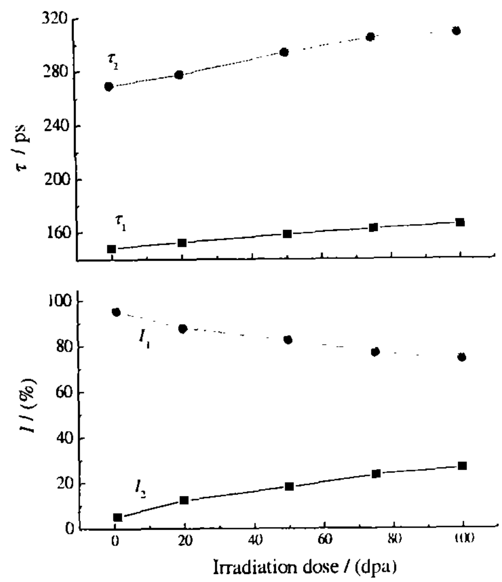

Fig. 4 Dose dependence of positron lifetimes and their intensities in MSS irradiated by 80 MeV fluorine ions at room temperature.

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不锈钢辐射损伤随温度和剂量的变化*

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摘　要：用重离子辐照模拟和正电子湮没寿命技术研究了改进型316L不锈钢在21和33 dpa辐照剂量下的辐照损伤在室温到802 ℃温度范围随辐照温度变化和室温下0—100 dpa剂量范围随辐照剂量变化. 在580 ℃左右实验观察到辐照肿胀峰，在21和33 dpa辐照剂量下相应的空位团分别由14和19个空位组成，尺度分别为0.68和0.82 nm. 空位团尺寸随辐照剂量增加，在100 dpa时空位团由8个空位组成，尺度为0.55 nm. 实验结果表明，在改进型316L不锈钢中辐照损伤随辐照温度变化更灵敏.

关键词：改进型316L不锈钢；辐照损伤；正电子湮没寿命技术；重离子辐照模拟

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