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Vacancy migration process in F82H and Fe-Cr binary alloy using positron annihilation lifetime measurement

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Abstract. Microstructral evolution of electron-irradiated F82H and Fe-8%Cr at 77 K was studied using positron annihilation lifetime measurements. Irradiation-induced vacancies started to migrate at 300 K and 180 K in F82H and Fe-8%Cr, respectively. Solute Cr atoms did not suppress vacancy migration, but they made di-vacancies more stable. Microvoids were not formed by annealing. In F82H, solute atoms acted as trapping site of irradiation-induced defects and annihilation of vacancies and interstitials was facilitated. Pre-existing dislocations and precipitates were also their sinks. These lead to the suppression of microvoids formation. In Fe-8%Cr, small vacancy-type dislocation loops were formed by isochronal annealing test.

1. Introduction

Fusion reactor structural materials are irradiated with high dose of neutrons, and a large amount of defects is formed. The formation leads to void swelling and irradiation embrittlement, which are main problems in fusion reactor materials. Reduced activation ferritic/martensitic steel F82H is recognized as attractive candidate materials for neutron interactive structural components of fusion energy systems. Ferritic/martensitic steel has good dimensional stability under high irradiation doses and is suitable for commercial production without a large industrial investment. It is difficult to irradiate them with high dose of neutrons corresponding to actual fusion reactor at the present day. The effects of irradiation on the properties of materials at high doses must be derived from fission neutron irradiation experiments. Kiritani *et al*. reported the comparison of defect structure in many fcc metals irradiated with fission and fusion neutrons [1]. Sato *et al*. also reported the difference of microstructural evolution in vanadium alloy and ferritic/martensitic steel F82H irradiated with fission and fusion neutrons [2]. Lifetime prediction of fusion reactor structural materials is performed by computer simulation, and many physical properties, which are derived from fission neutron irradiation experiments, are necessary for the calculation. But even the vacancy migration energy has not been obtained exactly in F82H. The purpose of this study is to obtain the vacancy migration energy in F82H using positron annihilation lifetime measurement. Fe-Cr binary alloy is also used in order to investigate the effect of added elements except for Cr.

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2. Experimental

F82H sheets were prepared by Japan Atomic Energy Agency (JAEA). Thickness of them was 0.2mm. They were punched to 5 mm in diameter and chemically polished to remove the oxidized area. Composition of F82H is Fe-7.65Cr-2W-0.16Mn-0.16V-0.02Ta-0.11Si-0.09C. Fe-8%Cr alloy was produced by plasma jet melting. It was cold rolled to a thickness of 0.1 mm and 5-mm-diameter discs were punched out. The discs were annealed at 1313 K for 0.5 and then tempered at 1013 K for 2h in vacuum. The electron irradiation was performed by using the electron linac in Research Reactor Institute, Kyoto University under the conditions of 30 MeV, 4×10^{18}electrons/cm^2, 77 K. Irradiation dose was 1.2×10^{-3} dpa with the displacement threshold energy of 40 eV. Positron annihilation lifetimes were measured using a fast-fast time spectrometer with the BaF_2 scintillators and the Hamamatsu photomultiplier tubes H3378. The time resolution of the system was about 190 ps (full width at half maximum). Each spectrum was accumulated to a total of approximately 1.2×10^6 counts. Lifetime spectra were analyzed using the PALSfit program [3]. Isochronal annealing experiments were performed at intervals of 20 K and 50 K up to 600 K and above 600 K, respectively. Annealing time was 0.5 h. Measurement temperature of positron annihilation lifetime was liquid nitrogen temperature (77 K) and room temperature (300 K) up to 280 K and above 300 K, respectively.

3. Results and discussion

Figure 1 shows the positron annihilation lifetime of electron-irradiated F82H. Lifetime of matrix, single vacancies and di-vacancies in figure is that of pure Fe from reference 4. The long lifetime τ_2 at annealing temperature of less than 280 K was less than lifetime of single vacancies (180 ps [4]). The long lifetime of unirradiated F82H is about 140 ps caused by pre-existing dislocations and precipitates. The long lifetime component contains that of not only single vacancies formed by electron irradiation but also pre-existing defects. After 300 K annealing, the long lifetime increased. The long lifetime of 184ps is shorter than that of di-vacancies (202 ps). A part of vacancies form into di-vacancies and other vacancies which are tightly trapped at solute atoms cannot migrate by annealing from 300 K to 400 K. Vacancy clusters gradually grew up to 420 K. At the same time, the long lifetime intensity (defect concentration) decreased. The reason of suppression of large void formation is that the migrating vacancies find interstitial type defects trapped at solute atoms or sinks (dislocations and interfaces of precipitates) and disappear. The long lifetime decreased to 160 ps by 440 K annealing. By annealing at above 460 K, the long lifetime was about 140 ps and positrons were mainly trapped at dislocation loops and precipitates, which were formed from irradiation-induced defects and existed before irradiation. The decrease of mean lifetime stopped at 600 K.

If all vacancies formed by electron irradiation remain, one vacancy in 833 atoms ($1 / 1.2\times10^{-3}$ dpa) exists. The radius of clusters consisting of 833 atoms is about 6 atomic distances. In order for vacancies to move up to 6 atomic distances in random motion, 36 jumps are needed. When the vacancy jumps is larger than 36 jumps, vacancies can aggregate. From the above results, single vacancies can migrate at 300 K in F82H, and vacancy clusters are formed. Vacancy jump frequency is expressed as $\nu_0\exp(-E_m/kT)$, where ν_0 is the frequency of lattice vibration (10^{13} /s), E_m is the migration energy, k is the Boltzmann constant, and T is absolute temperature. When the vacancy migration energy is 0.87 eV, vacancy jumps exceed 36 jumps at 300 K for 0.5 h. But, because a part of vacancies disappear by interstitial migration during irradiation, the longer-distance migration of vacancies is required for the formation of vacancy clusters. Therefore, the exact vacancy migration energy is expected to be a little lower than 0.87 eV. In order to obtain it, isothermal annealing experiment should be performed.

Figure 2 shows the positron annihilation lifetime of electron-irradiated Fe-8%Cr. Lifetime spectra were not resolved into two components below 120 K. Because the lifetime of 177 ps is almost the same as single vacancy lifetime, almost all positrons were trapped at vacancies. Lifetime spectra could be resolved into two components above 140 K, and the long lifetime increased to about 200 ps by 180 K annealing. This means single vacancy migration and di-vacancies formation. The long lifetime continued to decrease from 260 K to 420 K. By 460 K annealing, it increased once and then started to decrease again. Mean lifetime continued to decrease up to 600 K.

In Fe-8%Cr used in this experiment, α' phase is inevitably formed. There exist nuclei of the phase because of the slow cooling rate after heat treatment. Point defect migration helps the growth of α' phase. Using the method employed to F82H, the vacancy migration energy in Fe-8%Cr was obtained to be 0.52 eV. This value is almost the same as that in pure Fe (0.55 eV [5]). Therefore, solute Cr atoms do not influence the vacancy migration. The decrease of long lifetime intensity from 180 K to 280 K is due to interstitial migration. As binding energy of di-vacancies is 0.2 eV [5], dissociation energy of di-vacancies is 0.75 eV. Di-vacancies dissociate at about 260 K. But di-vacancies remained above 260 K. Although solute Cr atoms did not interact with single vacancies, they made di-vacancies more stable until 300 K. The change in lifetime above 300 K can be explained by the growth of vacancy-type dislocation loops. At annealing temperature of less than 360 K, the long lifetime is longer than that of single vacancies (180 ps) and it is too long compared with the lifetime of vacancy-type dislocation loops. Thus, it is expected that small three-dimensionally-agglomerating vacancy clusters also exist, but we cannot resolve the long lifetime component into two components (three-dimensionally-agglomerated vacancy clusters and dislocation loops). When the vacancy-type dislocation loops grow, the lifetime decreases because the gap between two planes decreases. The decrease of long lifetime from 320 K to 420 K is due to this effect. At 440−500 K, the existence of single vacancies was difficult and three-dimensionally-agglomerating vacancy clusters were not detected by the positron annihilation lifetime measurements. In this temperature range, the short lifetime component τ_1 agreed with that calculated by the two state trapping model, which assumed that positrons are annihilated at matrix and at one kind of defect sites. Therefore, the long lifetime of 160−175 ps at 440−500 K denotes that of small vacancy-type dislocation loops. If we assume that loops are not stable in α' phase at above 440 K, the decrease of long lifetime intensity, i.e., the

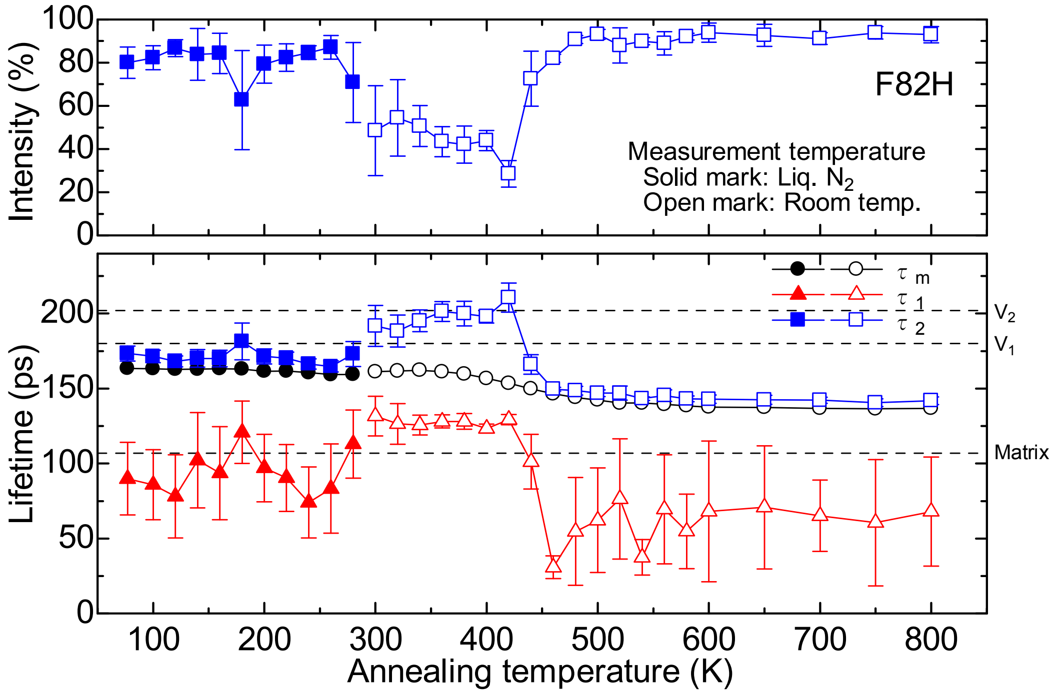

Figure 1. Isochronal annealing behavior of positron annihilation lifetime of electron-irradiated F82H. Annealing time was 0.5 h. Circles, triangles and squares denote the mean (τ_m), short (τ_1) and long (τ_2) positron lifetimes, respectively. Solid and open marks denote measurement temperature of liquid nitrogen and room temperature, respectively. Dashed line of Matrix, V_1 and V_2 denote the calculated positron lifetimes of matrix (106 ps), single vacancies (180 ps) and di-vacancies (202 ps) [4], respectively.

decrease of loop density, is possible to understand. Vacancies generated by evaporation of loops promote the nucleation of small loops and also the growth of α' phase. We have to investigate the phase growth in more detail. In F82H, the formation and growth of phase also should be studied.

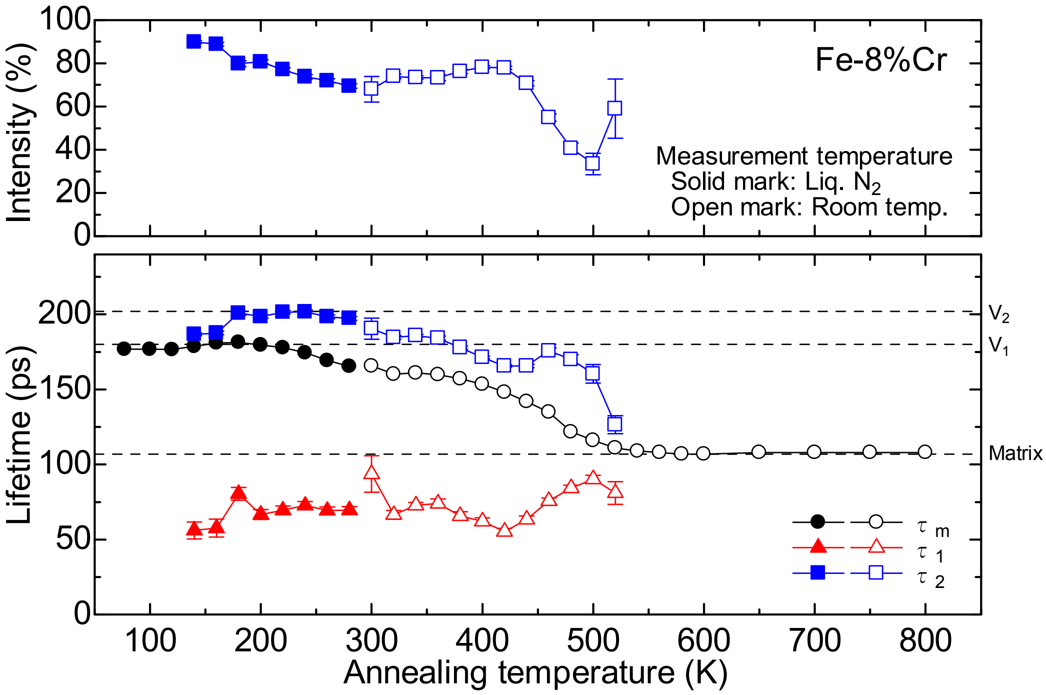

Figure 2. Isochronal annealing behavior of positron annihilation lifetime of electron-irradiated Fe-8%Cr. Annealing time was 0.5 h. Circles, triangles and squares denote the mean, short and long positron lifetimes, respectively. Solid and open marks denote measurement temperature of liquid nitrogen and room temperature, respectively. Dashed line of Matrix, V_1 and V_2 denote the calculated positron lifetimes of matrix (106 ps), single vacancies (180 ps) and di-vacancies (202 ps) [4], respectively.

4. Conclusion

Annealing behavior of positron annihilation lifetime of electron-irradiated F82H and Fe-8%Cr was examined. Vacancies started to migrate at 300 K and 180 K in F82H and Fe-8%Cr, respectively. Microvoids were not formed by annealing. In F82H, solute atoms act as trapping site of irradiation-induced defects, and pre-existing dislocations and precipitates also act as sinks. These lead to the suppression of microvoids formation. The effect of added elements except for Cr was quite powerful in F82H. In Fe-8%Cr, the change in long lifetime was related in the growth of vacancy-type dislocation loops.

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