

I-1. PROJECT RESEARCHES

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OBJECTIVES: High dose neutron irradiation is usually used to investigate radiation effects in solid materials and develop new materials. Unfortunately, KUR could not supply the neutron irradiation field and the laboratory for neutron-irradiated samples was closed in 2015. The research activities were severely restricted. Most of researchers had to change their research purpose to get the results. Therefore, instead of neutron irradiation, electron and γ ray irradiations were used to estimate the damage induced by the irradiation in materials.

RESULTS: The carried out research subject (ARS) and the name of co-researches in each ARS are listed below.

ARS-1

Modification of the yellow luminescence in gamma-ray irradiated GaN bulk single crystal

(K. Kuriyama, A. Shikawa, N. Nishikata, K. Kushida, and Q. Xu)

The results show that gamma-ray induced shallow donor relating to nitrogen vacancy is located at about 50 meV below the conduction band.

ARS-2

Photoluminescence of natural and synthesized calcites (T. Awata, S. Tanaka and Q. Xu)

Photoluminescence (PL) of natural calcites and impurities of these, and compared spectra of artificially synthesized calcite in chemical reaction have been measured.

ARS-3

Thermal diffusivity measurement of small specimens to achieve the measurement of heavily neutron irradiated tungsten

(M. Akiyoshi and Q. Xu)

The measurement results of thermal diffusivity between small specimen and 10 mm standard specimen are different. The result of small specimen is small even above 373 K compared with 10 mm standard specimen.

ARS-4

Electron irradiation induced damage structure in

intermetallic alloys

(F. Hori, Y. Ueno, T. Ishiyama, K. Kobayashi, A. Iwase, K. Ohsawa, Q. Xu and N. Abe)

Before irradiation, the single Al-vacancy remains in Fe-50Al alloy. After irradiation, however, the different types of vacancy were produced into Fe-Al compound alloy.

ARS-8

Effects of high energy particle irradiation on hydrogen retention in refractory metals

(K. Tokunaga, M. Matsuyama, S. Abe, H. Osaki, K. Araki, M. Hasegawa, K. Nakamura, Q. Xu and K. Sato)

The amount of tritium increases with increasing the irradiation dose. In addition, tritium is easy to diffuse parallel direction for rolling direction after irradiation.

ARS-9

Positron annihilation spectroscopy of Ferritic/Martensitic steels irradiated with protons and spallation neutrons

(K. Sato, Q. Xu, T. Yoshiie, Y. Dai and K. Kikuchi)

Vacancy clusters are formed by the irradiation in F82H. The vacancy clusters including small and large amount of He atoms up to 15 dpa of irradiation.

ARS-10

Positron annihilation behavior in Fe-Cr alloys

(R. Kasada, K. Sato and Q. Xu)

In unaged Fe_{0.91}Cr_{0.09} and Fe_{0.84}Cr_{0.16} alloys, there are not enhancement of positron density. In contrast, an enhancement of annihilation in Fe cell in other Fe-Cr alloys, such as, Fe_{0.7}Cr_{0.3}, Fe_{0.55}Cr_{0.45} and Fe_{0.5}Cr_{0.5}.

CONCLUSIONS: The defects induced by irradiation degrade the physical mechanical properties of solid materials. Some experimental results are important for development of nuclear industry. For example, the results of ARS-8 indicate that the retention of tritium, which is the isotope of hydrogen, in refractory metals used in fusion reactor may be an important issue because the plasma-facing materials suffer two types of damage: displacement damage caused by high energy neutrons and surface damage.

PR1-1 Modification of the Yellow Luminescence in Gamma-ray Irradiated GaN Bulk Single Crystal

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INTRODUCTION: Examining the defects caused by various radiations to GaN under the space environment is important. In our previous study [1, 2], we reported that the energy levels relating to nitrogen vacancy (V_N) and gallium vacancy (V_{Ga}) were induced by neutron and proton irradiated GaN. The neutron irradiation has been used as the neutron transmutation doping into semiconductors such as GaAs [3], GaP [4], and GaN [5]. Atoms in semiconductors mainly transmute by a (n, γ) reaction. Therefore, to survey the radiation effect of gamma ray alone is meaningful. In the present study, we report the variation of the yellow luminescence (YL) in GaN bulk single crystals by gamma-ray irradiation.

EXPERIMENTS: GaN bulk single crystals with a thickness of $450 \pm 50 \mu\text{m}$ were purchased from Furukawa Co. Ltd. The crystals were irradiated at room temperature with gamma-rays of 1.17 and 1.33 MeV from a cobalt-60 source of Kyoto University Research Reactor Institute. Samples were irradiated with an absorption dose rate of 1.771 KGy/h. Total gamma-ray dose was 160 kGy. The resistivity varied from $30 \Omega\text{cm}$ for an un-irradiated sample to $10^4 \Omega\text{cm}$ for gamma-ray irradiated one. Photoluminescence (PL) spectra were measured at 77 K using a He-Cd laser.

RESULTS: The band edge emission was observed at 3.48 eV in both un-irradiated and gamma-ray irradiated samples. The YL from the un-irradiated GaN with a peak at 557 nm (2.22 eV) was observed at around 440 nm to 800 nm, whereas that of the gamma-ray irradiated GaN showed a peak at 532 nm (2.33 eV) although the YL spectrum is almost overlapped with un-irradiated ones. Therefore, the modification of YL observed here would be attributed to a transition from the shallow donor induced by the gamma-ray irradiation to a native V_{Ga} . Since the usual YL has been proposed as a transition from a shallow donor to V_{Ga} lo-

cated at about 1.1 eV above the valence band [6], the gamma-ray induced shallow donor is located at about 50 meV below the conduction band. This energy level is close to a donor level ($64 \pm 10 \text{ meV}$ [7]) relating to V_N . Figure 1 shows the YL model estimated from the photoluminescence at 77 K.

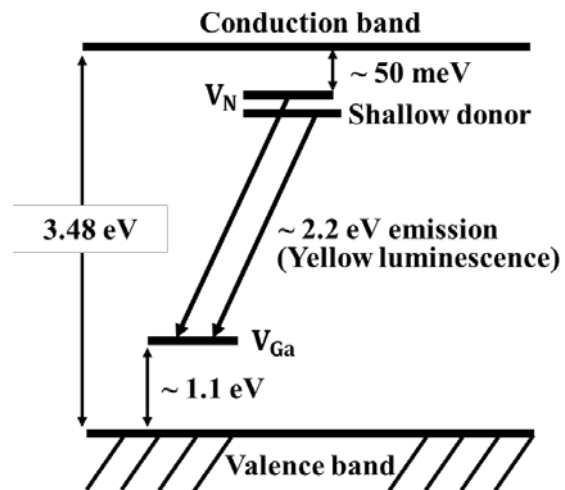


Fig.1 Yellow luminescence estimated from the photoluminescence at 77 K. The gamma-ray induced shallow donor relating to nitrogen vacancy is located at about 50 meV below the conduction band.

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INTRODUCTION: Thermoluminescence (TL) of natural calcite irradiated by gamma rays have only one orange emission peak and that may be originated from an impurity of Mn^{2+} in calcite [1-2]. At photoluminescence (PL) of natural calcite irradiated UV rays, the spectra of PL have 620nm emission peak and 270nm emission peak which is related Mn^{2+} and Pb^{2+} , respectively [3]. In the case of calcites, it has been shown the impurity plays a major role for luminescence. In this study, we first have measured PL of natural calcites and impurities of these, and compared spectra of artificially synthesized calcite in chemical reaction with resulted major impurities.

EXPERIMENTS: Natural calcite with colored pink was collected at Colorado State in USA. Artificial calcite was synthesized by a chemical reaction method in water solution [4]. 1.5% of $(\text{NH}_4)_2\text{CO}_3 \cdot 2\text{H}_2\text{O}$ (1.5g in 100mL pure water) and 1.8% of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ (2g in 100mL pure water) with 0.01g element impurity were mixed at room temperature. After mixed, these solutions were precipitated by holding for two weeks and filtering. After filtering, samples were shaped by a tablet machine. PL spectra have been taken using PL-84 (Seishin, SOEX1702/04, PM:Hamamatsu R928) with He-Cd laser (Kinmon 325nm). ICP-AES (iCAP6300Duo, Thermo Fisher) was also performed to measure impurity elements concentration of calcite.

RESULTS and DISCUSSION: Figure 1 shows the PL spectra of Colorado natural calcite irradiated 325nm UV rays. It is shown that a spectrum has three peaks at 396nm, 564nm, 760nm and 564nm peaks. 564nm peak is

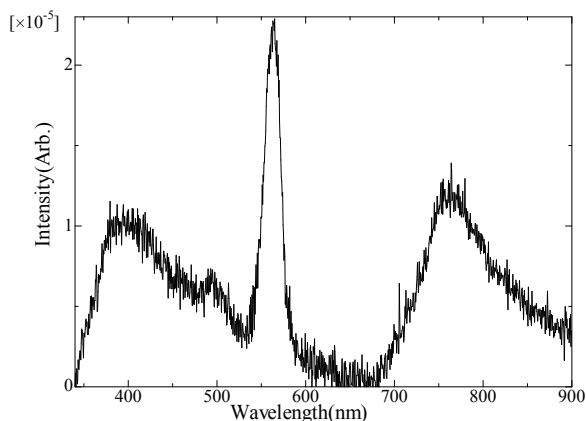


Fig. 1 Photoluminescence of natural Colorado calcite.

a strongest and most sharp (FWHM=4%). From ICP-AES results, Colorado calcite has impurities (ppm) of Sr (5.1), Mn (N.D.) and Pb (0.05). Nevertheless almost calcite has large amount of Mn impurity, Colorado calcite has very little amount. There is no peak at 620nm in figure 1, that is related to little Mn amount. Photoluminescence data reference [5] reported that emission peak at 560nm for defect center of carbonate ion. From experimental results, we have synthesized calcite artificially with controlled impurities. Fig 2 shows that PL spectra of synthesized calcite with an impurity of Sr and Sr + Mn. It is shown that both

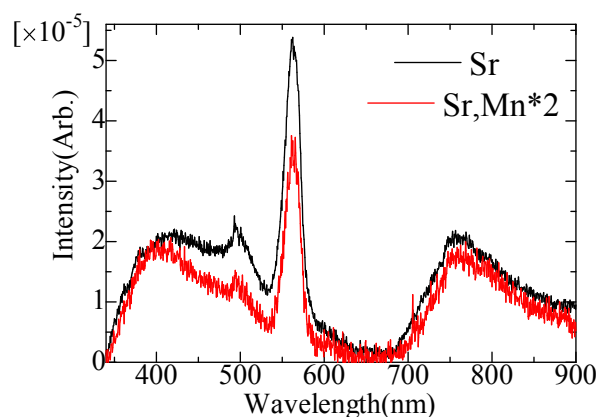


Fig. 2 Photoluminescence spectra of synthesized calcite with an impurity of Sr and Sr + Mn.

spectra have four peaks at 427nm, 493nm, 560nm and 756nm. Comparing PL spectra of natural and synthesized calcite, both spectra are almost same. PL spectra of synthesized calcite with Sr and Sr + Mn are almost same and there is no effect of presented Mn ion. Sidike et al., reported [3] that Mn ion cannot be an activator alone for PL, but can be activator with co-activator as Ce ion or Pb ion which can absorb UV rays. Relatively large amount impurity in Colorado calcite, Sr ion, is not activator but also co-activator for Mn ion.

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PR1-3 Thermal Diffusivity Measurement of Small Specimens to Achieve the Measurement of Heavily Neutron Irradiated Tungsten

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INTRODUCTION:

Usually, heat is mainly carried by free electrons in metals. On the other hand, it is well known that irradiation induced damages in ceramics showed significant degradation in thermal diffusivity unlike metals, because in ceramics, heat is mainly carried by phonon that is scattered by irradiation induced defects. In these days, development of materials for divertor in future fission reactor is very important and concerned study. Most of studies are focused on SiC ceramics and tungsten based materials. Tungsten is a metal, of cause, however heat is carried by phonon in tungsten partly. Then, thermal diffusivity of tungsten after severe neutron irradiation must be estimated in the same way as ceramics.

In addition, tungsten shows far stronger radioactivity after a neutron irradiation than SiC ceramics. In the PHENIX project for development of divertor materials in fusion reactor, many SiC and W specimens were loaded to HFIR reactor in ORNL, USA. To reduce the level of radioactivity, specimens for thermal diffusivity measurement is limited to 6mm in diameter or further small 3mm in diameter disk specimens with 0.5mm thickness.

Measurement of thermal diffusivity after the neutron irradiation in HFIR is must to be operated using Netzsch LFA-457 Micro Flash thermal analyzer in ORNL, which can measure 6mm disk specimens using commercial jigs, while measurement of 3mm small disk is not certified.

EXPERIMENTS:

To achieve a measurement of small specimens 3mm in diameter using Netzsch LFA-457, special jigs were designed and manufactured. The jigs shade the incident laser flash to 2.4mm in diameter and also cut the infrared respond signal radiated from the back side of the specimen to 2.6mm in diameter.

Measurements of thermal diffusivity using small disks were performed at ORNL, USA using Netzsch LFA-457, and also 10mm in diameter standard sample was measured using Ulvac TC-7000 at the Radiation Laboratory, Uji, Kyoto University. Specimens were ITER grade pure tungsten (supplied by A.L.M.T. Corp.) with the size of 6mm and 10mm in diameter and commercial grade pure tungsten (supplied by Nilaco) with the size of 3mm and 10mm in diameter. The measurements were operated at room temperature to 773K using Netzsch LFA-457, and to 973K using Ulvac TC-7000, respectively.

RESULTS:

Fig.1 shows thermal diffusivity of ITER Grade tungsten with the size of 6mm and 10mm in diameter. The measurement result of a 6mm sample showed large dispersion at around room temperature. The laser flash method uses uprising curve of temperature on the back side surface

after a laser flashing, and the Netzsch LFA-457 system uses IR detector to measure the temperature. Intensity of the IR is changed with a temperature according to the Planck radiation formula, and also with measuring area of a specimen. The back side area of a 6mm specimen is only 36% of a 10mm specimen. Then the wave-shape from the IR detector was noisy at around room temperature, while above 373K, the intensity of IR was increased enough to evaluate the thermal diffusivity correctly.

This result is almost same for a 3mm specimen shown in Fig.2, however measurement result of this small specimen is obviously small even above 373K compared with 10mm standard specimen. The difference in the shape of these specimens is not only the surface area but also the thickness, 0.973mm for 10mm diameter and 0.598mm for 3mm diameter specimen. The later thickness is too thin to evaluate correctly for tungsten before neutron irradiation. The difference between the thermal diffusivity of 10mm diameter and 3mm diameter getting smaller at higher temperature, because the uprising time of IR after the laser flashing getting longer and analyzed better. Neutron irradiated tungsten shows smaller thermal diffusivity than the unirradiated specimens here, so the difference is expected to be enough small with the specimen of 0.5mm thickness.

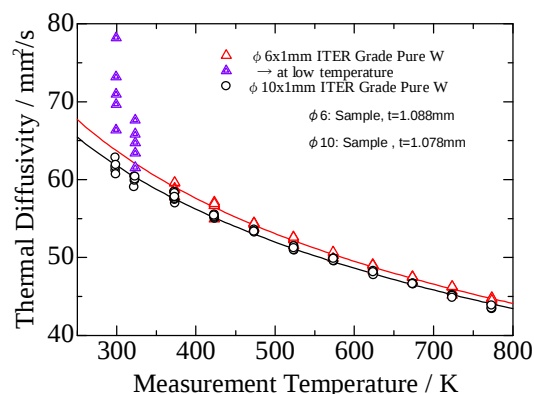


Fig.1 Validation of thermal diffusivity measurement using a specimen of 6mm in diameter.

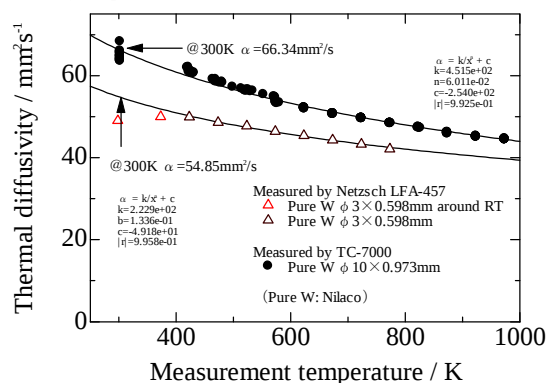


Fig.2 Validation of thermal diffusivity measurement using a specimen of 3mm in diameter.

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INTRODUCTION: It is well known that intermetallic compound alloys possibly include more than two types of vacancies basically, that is A-vacancy and B-vacancy in A-B compound alloy. Thermally equilibrium defects and chemically deviated structural defects in these compound alloys are very complicated. However, the amount of defect and the defect structure affects the various characteristic features, such as strength and electronic conductivity and so on. Moreover, first principle calculation shows that different number of hydrogen atoms can be trapped by Al- or Fe-vacancy in B2 type Fe-Al alloy. On the other hand, although the electron irradiation produces vacancies and interstitials in metallic alloys, the threshold energy of displacement for each elemental atom in compound alloys is not necessarily same. Then the defect type introduced by electron irradiation depends on its incident energy. Kosugi et al. reported that the type of defects produced by electron irradiation in B2 type Fe-Rh intermetallic alloy changes with increasing of incident electron energy [1]. Then we supposed that radiation induced vacancy type defects can be controlled by changing the energy of electron irradiation. In this study, we have performed electron irradiation with different energy for Fe-Al alloys.

EXPERIMENTS: Fe-50%Al alloy specimens with B2 structure were prepared by arc melting method. Sliced samples with the thickness of 0.5 mm were annealed at 973 K for 120 h followed by air-quenched in vacuum. These specimens were irradiated with 2 MeV electron to the fluence of 1×10^{17} and 1×10^{18} /cm² at JAEA-Takasaka and with 9 MeV electron to the fluence of 5×10^{17} and 3×10^{18} /cm² at KURRI, Kyoto University. In both cases, irradiations were carried out at about 330 K controlled by water flow system. After and before irradiation, samples were examined by positron annihilation lifetime and coincidence Doppler

broadening measurements. The positron lifetime spectra were analyzed by using POSITRONFIT program. Also, the positron lifetimes in B2 type Fe-Al alloys were calculated using the superimposed atom method [2].

RESULTS: Before irradiation, positron lifetime of this alloy was 186 psec showing that single vacancy still remains in this alloy. This positron lifetime value is close to that in Al-vacancy by the calculation. The positron annihilation lifetime value of this alloy increases to 200 psec after 2 MeV electron irradiation and decreases to 172 psec after 9 MeV electron irradiation, respectively. Also, the Doppler broadening S-parameter showed different changing trend for each electron irradiation with different energy. That is S-parameter increases for 2 MeV irradiation and decreases for 9 MeV irradiation case. Figure 1 shows the changing relation between positron lifetime and S-parameter for each energetic electron irradiations. This result suggests that different types of vacancy were produced into Fe-Al compound alloy by electron irradiation with different energies.

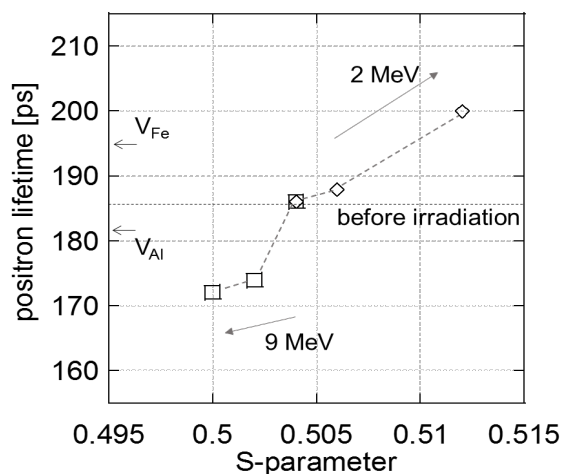


Fig. 1 Positron lifetime and S-parameter relation for before and after electron irradiated Fe-Al alloy.

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INTRODUCTION: It is of importance to clarify phenomena of implantation, retention, diffusion and permeation of tritium on surface of the armor materials of the first wall/blanket and the divertor on fusion device from a viewpoint of precise control of fuel particles, reduction of tritium inventory and safe waste management of materials contaminated with tritium (T). Refractory metals such as tungsten (W) is potential candidate for an armor of the first wall and the divertor plate of the fusion reactor because of its low erosion yield and good thermal properties. The armor material will be subjected to heavy thermal loads in the steady state or transient mode combined with high energy neutron irradiation that will cause serious material degradation. In addition, high energy runaway electrons would bombard the armor materials along the equatorial plane in fusion device. It is considered that these cause radiation damage and enhance tritium retention. In the present works, T exposure experiments have been carried out on W samples which were irradiated by high energy electrons using LINAC in KURRI of Research Reactor Institute, Kyoto University to investigate effects of high energy electrons irradiation and micro-structure on tritium retention of W. In the report of the last fiscal year, results of T exposure experiments on ITER grade W which was fabricated by powder metallurgy. In the present report, results of T exposure experiments on W which was fabricated by Hot Isostatic Pressing (HIP-W) and comparison of results with ITER grade W and HIP-W will be described.

EXPERIMENTS: W samples used were ITER grade W (IG-W) and Hot Isostatic Pressing W (HIP-W). In the case of IG-W, one was W sample (ITER grade W(1)) which the surface were manufactured to be oriented parallel to the rolling surface and rolling direction. The other W sample (ITER grade W(3)) which the surface were manufactured to be oriented perpendicular to the rolling surface and rolling direction. On the other hand, structure of HIP-W is equiaxed deriving from the nature of the consolidation process. The sizes of W samples were 10mm x 10mm x 1mm. The surface of the both samples were polished to be mirrored. High energy electrons irradiation has been carried out using LINAC in KURRI of Research Reactor Institute, Kyoto University. An energy of electron irradiated was 10 MeV and DPA was

3.26×10^{-3} . Temperature during the irradiation was measured by thermocouples which was contacted with a backside of the W samples. After the electron beam irradiation, T exposure experiments have been carried out using a T exposure device in University of Toyama. Pressure of the T gas was 1.3 kPa and T exposure was kept for 4 h. T concentration in the gas was about 5 %. Temperatures of pre-heating and T exposures were 100 oC. After the exposure to T gas, T amount retained in surface layers of the sample was evaluated by β -ray-induced X-ray spectrometry (BIXS) and imaging plate (IP) measurements.

RESULTS: In the condition of 10 MeV electrons irradiation, electrons go through in 10 mm thickness W sample. As a result, uniform defects were formed in the W samples. Fig. 1 shows IP images of HIP-W, IG-W(3) and IG-W(1). It can be seen that amount of T near surface depends on kinds of W. Fig. 2 shows amount of T as a function of DPA. In general, the amount T increases with increasing DPA. In addition, it is considered that T is easy to diffuse parallel direction for rolling direction from comparing with IG-W(3) and IG-W(1) after the electron irradiation.

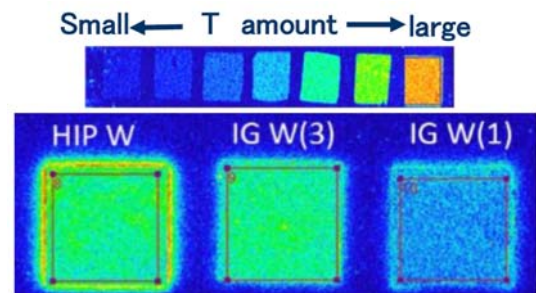


Fig. 1. Tritium image of HIP-W, ITER grade W(3) (IG W(3)) and ITER grade W(1) (IG W(1)). Upper parts are standard samples.

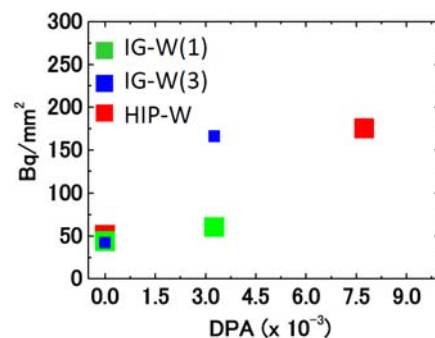


Fig. 2. Amount of tritium as a function of DPA

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INTRODUCTION: In fusion reactors and spallation neutron sources, irradiation damage is significantly more serious than fission reactors. There is a need for use of irradiation resistant materials in high neutron dose facilities. Due to its good thermal and mechanical properties, the reduced-activation ferritic/martensitic steel, F82H is a candidate material for the first wall and blanket of the future fusion reactor [1]. There have been a number of studies on the void swelling behavior of structural materials after neutron irradiation [2,3]. Recent theoretical and experimental analyses have revealed the importance of incubation period, a transient stage before the steady growth of voids. Positrons are very sensitive to vacancy type defects, and even single vacancies can be detected in positron annihilation lifetime measurements. In this study, defect structures in both F82H and SS316 steels during the incubation period were studied after proton irradiation using positron annihilation lifetime measurements.

EXPERIMENTS: The reduced activated ferritic/martensitic steel F82H was used in this experiment. F82H was irradiated in the second program (STIP-II). More details of the STIP-I and STIP-II experiment were previously reported [4]. Table 1 shows sample ID, irradiation dose, average irradiation temperature and calculated production of H and He. It should be noted that the actual H contents can be much lower than the calculated values due to fast diffusion of H in steels [5]. The chunks of tensile test samples of F82H were used for positron annihilation lifetime measurements at Paul Scherrer Institut.

RESULTS: Before irradiation, the long lifetime of about 150 ps denotes the lifetime of the dislocations and the lath boundaries. Vacancy clusters are formed by the irradiation. In F82H, He bubbles formed by the irradiation above 443 K and about 500 appm He are visible by TEM. He bubble size increases and the concentration decreases with increasing the irradiation dose and temperature by TEM. However, the long lifetime intensity does not increase with increasing the irradiation dose. Therefore, it is also possible to detect invisible He bubbles by positron annihilation lifetime measurements. The positron annihilation lifetime of vacancy clusters de-

creases to less than 200 ps when the vacancy clusters contain a large amount of He atoms. The short and long lifetime at 5.9 dpa is almost the same as the calculated lifetime of V_1 and V_{15} , respectively, due to a lot of gas atoms are produced during the irradiation. Therefore, the long lifetime and short lifetime up to 15 dpa denote vacancy clusters including small and large amount of He atoms, respectively. Decrease of long lifetime and mean lifetime up to 15 dpa is due to the absorption of He atoms. The concentration of vacancy clusters including small amount of He atoms decreases up to 10 dpa because they absorb He atoms and are classified into the short lifetime. After that, the concentration of vacancy clusters including small amount of He atoms increases again. In this stage, small vacancy clusters, which are classified into the short lifetime up to 10 dpa, grow more prominently and are classified into the long lifetime. From TEM observation, He bubbles grow with increasing irradiation dose above 10 dpa, but the positron lifetime does not increase. The absorption of He atoms is also large. If V_{341} (about 1.5 nm in diameter) contains 341 and 682 He atoms, the positron lifetime changes from 441 ps to 242 and 187 ps in Fe, respectively [6]. These results suggest that vacancy clusters contains more He atoms than number of vacancies which make up the vacancy clusters above 15 dpa.

Table 1. Irradiation condition of F82H.

ID	Temperature (K)	Irradiation dose (dpa)	He (appm)	H (appm)
L11-L	371	5.9	413	1680
L12-L				
L11-H	397	8.5	650	2530
L12-H				
L13-L	406	9.2	715	2850
L14-L				
L13-H	448	12.3	1015	4200
L14-H				
L15-H	513	17.2	1505	6200
L16-H				
L17-H	560	20.4	1795	7720
L18-H				

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INTRODUCTION: Ferritic steels containing Cr are expected to be used for the first-wall component of the fusion reactors as well as for the fuel pin cladding of the Generation IV nuclear fission systems [1]. However, high-Cr steels may suffer from thermal aging embrittlement, which is well-known 475 °C embrittlement. This is mainly due to hardening phenomenon through the phase separation of Fe and Cr as shown in the phase diagram. In the previous study [2], we applied a positron annihilation spectrometry to detect the phase separation in the Fe-Cr alloys after thermal aging at 475 °C. Currently, this collaborative research is investigating neutron irradiation effect on the positron annihilation behavior of Fe-Cr alloys. Here, we reports a new model to explain the positron annihilation behavior in Fe-Cr binary alloys.

Experimental Procedure: Positron annihilation coincidence Doppler broadening (CDB) measurement were performed at the hot laboratory of KURRI.

Results and Discussions: Based on the theoretical model of positron annihilation in binary alloys suggested by Lock and West [3], we modeled the effect of Cr concentration and aging on positron annihilation in disordered (unaged) Fe-Cr alloys. The expression for the W-parameter of the Fe-Cr alloys may be represented by

$$W_{FeCr} = f_{Fe}^+ W_{Fe} + f_{Cr}^+ W_{Cr} \quad (1)$$

as the weighted sum of the W parameter of the constituent pure metals (W_{Fe} and W_{Cr}). The fractions f denote the fractions of positron density in the Fe cell and Cr cell. The normalized W-parameters of $Fe_{1-x}Cr_x$ alloys can be written as following equation:

$$\frac{W_{FeCr} - W_{Fe}}{W_{Cr} - W_{Fe}} = f_{Cr}^+ = 1 - f_{Fe}^+ = 1 - (1-x)\eta_{Fe} \quad (2),$$

where η_{Fe} is the enhancement of positron density in the Fe cell of Fe-Cr alloy compared with the constituent pure Fe.

Figure 1 presents the normalized W-parameter of the CDB measurement against the Cr content x in the $Fe_{1-x}Cr_x$ alloys. In Figure 1 the curve 1, which linearly connects the Fe and Cr, represents the simplest case that corresponds to no enhancement of positron density in the Fe-Cr alloys. The unaged $Fe_{0.91}Cr_{0.09}$ and $Fe_{0.84}Cr_{0.16}$ are well fitted to the curve 1. In contrast, the other unaged Fe-Cr alloys indicate an enhancement of annihilation in Fe cell. The nonlinear function of Cr content for the unaged $Fe_{1-x}Cr_x$ alloys suggests a presence of preferential positron annihilation (PPA) in Fe cell at their disordered state. Further experiments and discussion on the aged Fe-Cr alloys are now going.

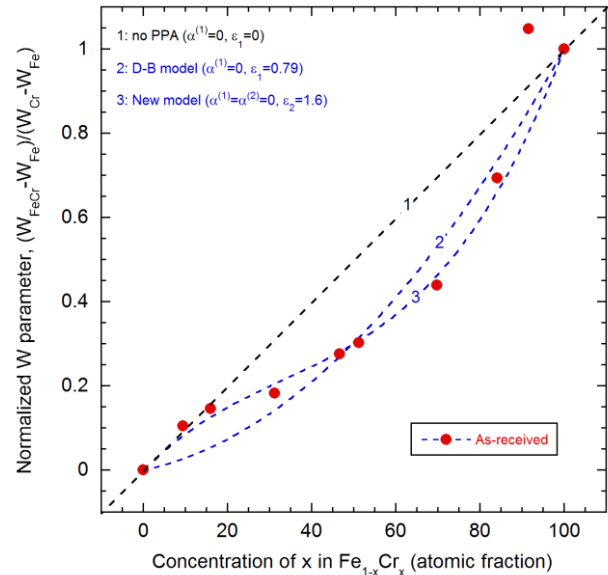


Fig. 1 Normalized W parameter of Fe-Cr alloys.

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