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# Positron annihilation lifetime measurements of vanadium alloy and F82H irradiated with fission and fusion neutrons

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# ABSTRACT

V-4Cr-4Ti, F82H, Ni and Cu were irradiated with fission and fusion neutrons at room temperature and 473 K. Defect structures were analyzed and compared using positron annihilation lifetime measurement, and microstructural evolution was discussed. The mean lifetime of positrons (the total amount of residual defects) increased with the irradiation dose. The effect of cascade impact was detected in Ni at room temperature. The size and the number of vacancy clusters were not affected by the displacement rate in the fission neutron irradiation at 473 K for the metals studied. The vacancy clusters were not formed in V-4Cr-4Ti irradiated at 473 K in the range of  $10^{-6}$ – $10^{-3}$  dpa. In F82H irradiated at 473 K, the defect evolution was prevented by pre-existing defects. The mean lifetime of positrons in fission neutron irradiation was longer than that in fusion neutron irradiation in V–4Cr–4Ti at 473 K. It was interpreted that more closely situated subcascades were formed in the fusion neutron irradiation and subcascades interacted with each other, and consequently the vacancy clusters did not grow larger.

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# 1. Introduction

Vanadium allovs and ferritic/martensitic steels are recognized as attractive candidate materials for neutron interactive structural components of fusion energy systems. Vanadium alloys have high temperature strength, high thermal stress factors and low activation properties. Reduced activation ferritic/martensitic steels have good dimensional stability under high irradiation doses and are suitable for commercial production without a large industrial investment. As materials irradiation with fusion neutrons at high doses is not available at present, the effects of irradiation on the properties of materials at high doses must be derived from fission neutron irradiation experiments. Therefore, the understanding of fission-fusion correlations is important. Sato et al. reported the microstructural evolution of V-4Cr-4Ti and F82H irradiated with fission and fusion neutrons at room temperature [1]. The effects of the fission and fusion neutron irradiation on the point defect production were almost the same if they were compared at the same dpa (displacement per atom). This is because the number of subcascades, which is proportional to dpa, was almost the same in V and Fe between fission and fusion neutron irradiations by the simulation. In the present work, the irradiation effects of fission and fusion neutrons on fusion reactor candidate alloys V-4Cr-4Ti and F82H, and for comparison Ni and Cu were studied using the Fusion Neutronics Source (FNS, the deuterium-tritium (D-T) neutron source facility) [2] at Japan Atomic Energy Agency (JAEA) and the Kyoto University Reactor (KUR) [3]. As the irradiation dose was low in the present study, the vacancy-type defects were analyzed and compared using positron annihilation lifetime spectroscopy, and the microstructural evolution was discussed.

# 2. Experimental

High purity V–4Cr–4Ti alloy of NIFS-HEATs [4] was used as a vanadium alloy. The specimens for positron annihilation lifetime spectroscopy were prepared from cold-rolled sheets by punching (3 mm in diameter) followed by annealing at 1373 K for 2 h in a vacuum. F82H alloy prepared by JAEA [5] was used after cutting and chemical polishing to remove the deformed area. Pure Ni (99.99%, Johnson-Matthey) and pure Cu (99.999%, Johnson-Matthey) were also prepared. Three millimeter diameter discs were punched out after rolling to 0.1 mm thickness. These specimens were annealed at 1173 K for 1 h and 1273 K for 20 min in a vacuum, respectively.

Fusion neutron irradiation was performed using a rotating tritium target of the FNS facility [2]. During irradiation, specimens are held with less than  $10^{-4}$  Pa using a turbo-molecular pump, and temperature fluctuation was within ±1 K. Fission neutron irradiation was performed using the KUR, 5 MW light water reactor. The irradiation experiment at room temperature was carried out in the Hydraulic Conveyer Facility (Hyd.) and that at 473 K was





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#### Table 1

Displacement rate of fusion neutron irradiation by the FNS and fission neutron irradiation by the SSS under 5 MW and 300 kW reactor operation in V-4Cr-4Ti.

	Displacement rate (dpa/s)
Fusion neutron (FNS)	$6.9 imes10^{-10}$
Fission neutron (SSS, 5 MW)	$1.8 imes10^{-8}$
Fission neutron (SSS, 300 kW)	$1.1  imes 10^{-9}$

carried out in the Materials Controlled Irradiation Facility (SSS) [3]. The reactor power was reduced to 300 kW to avoid an increase of specimen temperature due to nuclear heating during irradiation at room temperature. In the 473 K irradiation, the reactor power was 5 MW and 300 kW in order to investigate the effect of displacement rate. Temperature fluctuation was within  $\pm 0.1$  K. The displacement rate is shown in Table 1. The neutron dose was in the range of  $10^{-6}$ – $10^{-3}$  dpa, which was calculated with threshold energy for knock-on, 24 eV for V–4Cr–4Ti, Ni and Cu, and 40 eV for F82H. The irradiation temperature was room temperature and 473 K.

Positron annihilation lifetime measurements were performed at room temperature using the fast–fast coincidence system, whose lifetime resolution FWHM was 190 ps. The counting rate was about 80 cps. The positron lifetime spectra were collected with a total count of about  $1-3 \times 10^6$ . The spectra were analyzed using the Resolution and Positronfit programs [6].

# 3. Results and discussion

## 3.1. Room temperature irradiation

Fig. 1 shows the positron annihilation lifetime of pure Ni irradiated with fission and fusion neutrons at room temperature. Mean lifetime, long lifetime, and long lifetime intensity in Fig. 1 correspond to the total amount of residual defects, the size of vacancy clusters, and the amount of vacancy clusters, respectively. The mean lifetime of positrons increased with the increase of the neutron dose. In a dose higher than  $4 \times 10^{-4}$  dpa, single vacancies (180 ps) and vacancy clusters were detected in fusion and fission neutron irradiations, respectively. In fission neutron irradiation, the vacancy clusters grew larger. In a dose lower than  $10^{-4}$  dpa, the amount of vacancy clusters produced by the fission and fusion neutron irradiation was almost the same, if they were compared at the same dpa. At room temperature, the cascade structures are conserved and the number of subcascades per 1 dpa was almost



Fig. 1. The positron annihilation lifetime of pure Ni irradiated with fission and fusion neutrons at room temperature.

the same between fission and fusion neutron irradiation [1,7]. Therefore, it is expected that positrons are annihilated at the vacancy clusters made by subcascades.

It has been reported that the change in the increasing rate of defects was detected around  $10^{-4}$  dpa in fission neutron irradiated Ni and Cu, and the change was caused by the effect of impact on a cascade from other cascades [8,9]. This transition was not detected in fission neutron irradiation by TEM observation [9]. In a dose higher than  $10^{-4}$  dpa, the increasing rate of vacancy-type defects changed in this study, as shown in Fig. 1. The change must be the effect of impact and invisible defects by TEM can be detected by positron annihilation spectroscopy.

# 3.2. 473 K irradiation

Figs. 2–5 show the positron annihilation lifetime of V–4Cr–4Ti, F82H, pure Ni and Cu irradiated with fission and fusion neutrons at 473 K. Even if the displacement rate is different in the fission neutron irradiation, the microstructural evolution was almost the same in the same dpa for the metals studied. In the 473 K irradiation, cascades interact with each other because point defects can



Fig. 2. The positron annihilation lifetime of V-4Cr-4Ti irradiated with fission and fusion neutrons at 473 K.



Fig. 3. The positron annihilation lifetime of F82H irradiated with fission and fusion neutrons at 473 K.



Fig. 4. The positron annihilation lifetime of pure Ni irradiated with fission and fusion neutrons at 473 K.



Fig. 5. The positron annihilation lifetime of pure Cu irradiated with fission and fusion neutrons at 473 K.

perform a long-range migration. Therefore, it is expected that the difference of displacement rate was too small to cause the change in microstructural evolution. The mean lifetime of positrons increased with the increase of the neutron dose except for F82H. No change of positron lifetime in F82H means no growth of defects in low dpa region. The existence of sinks (dislocations, grain boundaries and so on) may prevent the formation of point defect clusters.

As shown in Fig. 2, unlike the results in the room temperature irradiation, the mean lifetime of positrons in the fission neutron irradiation was longer than that in the fusion neutron irradiation in V alloy. In pure Ni and Cu, the positron lifetime was also a little longer in fission neutron irradiation.

The vacancy clusters were not formed in V-4Cr-4Ti. Solute atoms prevent the migration of vacancies, interstitials and their clusters, and this leads to the pair annihilation of them. Subcascade analysis shows that the same number of subcascades was formed in the fission and fusion neutron irradiation in those metals at the same dpa [1,7]. As fusion neutron energy is higher than fission neutron energy, larger cascades are formed in the fusion neutron irradiation. Thus, more closely situated subcascades are formed in the fusion neutron irradiation compared with the fission neutron irradiation. Since vacancies can migrate at 473 K and the distance between subcascades is short, the subcascades interacted easily with each other and the vacancy clusters did not grow in the fusion neutron irradiation. On the other hand, subcascades did not interact with each other in the fission neutron irradiation. In pure metals, the same thing as V-4Cr-47Ti occurred though not so remarkable. One of reasons is the contribution of alloving elements in V allov to the formation of compact cascades by reducing the length of replacement sequence of collision.

# 4. Conclusion

V-4Cr-4Ti, F82H, pure Ni and Cu were irradiated with fission and fusion neutrons at room temperature and 473 K. Microstructural evolution was studied using positron annihilation lifetime measurement. The main results are as follows.

- (1) The effect of cascade impact for defect structural evolution was detected in Ni at room temperature.
- (2) The difference of the displacement rate did not result to the size and the number of vacancy clusters in the fission neutron irradiation at 473 K.
- (3) The vacancy clusters were not formed in V-4Cr-4Ti irradiated at 473 K in the range of  $10^{-6}$ - $10^{-3}$  dpa. Only single vacancies were formed.
- (4) In F82H irradiated at 473 K, the pre-existing sinks prevented the defect evolution.
- (5) In V-4Cr-4Ti, Ni and Cu irradiated at 473 K, the effective annihilation of point defects in large cascades was observed.

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