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# Positron Annihilation Lifetime Measurement of Low Temperature Electron Irradiated Fe-Cr-Ni and Fe-Cr-Ni-P alloys

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Positron annihilation lifetime mesurements of the low temperature electron irradiated austenitic model alloys, Fe-16Cr-17Ni and Fe-16Cr-17Ni-0.1P, have been made. It has been shown that phosphorus atoms influence on the migration temperature of the self interstitial atoms. Moreover, it has also been shown that phosphorus atoms suppress the clustering of vacancies probably due to the precipitates $Fe_2P$ formation. The vacancy migration temperature was determined as about 350K and was not affected by the presence of phosphorus atoms, which suggests that phosphorus atoms do not capture vacancies.

## Introduction

For the future energy supply, the development of the fusion reactor is very important. Now, the materials used in high temperature heavy irradiation environments are studied, and the stainless steels, the high melting point metals, or the graphites, *etc.* are candidated as the first wall material. Especially, the austenitic stainless steel is widely noticed because of its high temperature strength, rich data base, and economic advantage. On the other hand, this material has serious problem of void swelling by irradiation, so it is necessary to study the way for suppressing this phenomenon. The fact that some solute atoms well control the void formation has become known by means of the electron microscopy. For examples of such solute elements, P, Ti, and Si are well known [1], and among them the phosphorus has the highest efficiency per atom [2, 3]. By the way, the positron annihilation lifetime measurement can present the information of smaller region than the electron microscopy. So, this method is used for studies of the recovery process of radiation induced defects before they become large enough for the EM observation.

In the present study, in order to study the effect of phosphorus doping on vacancy cluster formation, we made the positron annihilation experiment on low temperature electron-irradiated austenitic model alloys.

## Experimental

High purity Fe-16Cr-17Ni and Fe-16Cr-17Ni-0.1P specimens were prepared by zone leveling method in Pd-purified dry hydrogen gas. In this process the amount of interstitial impurities such as C, O, N is considered to be reduced to the lowest level as possible. The shape of specimens was $8 \times 8 \times 0.25mm^3$. These spcimens were solution-treated in a

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vacuum of $\sim 10^{-6}$ Torr at 1323K for 30min before chemically polished.

The electron irradiation experiments were performed with two different energy electrons. The low energy irradiation was made with 2.5MeV electrons, and its fluence was $1.2 \times 10^{22} e^{-}/m^2$ by using the JAERI-Dynamitron electron accelerator. High energy irradiation was made with 28MeV electrons of a fluence of $4.5 \times 10^{22} e^{-}/m^2$ by using the KURRI-LINAC. The irradiation temperature was 77K in both experiments.

Then, the specimens were isochronally annealed for 20min in steps of 20K from 100K (in cold $N_2$ gas for 100 to 260K, in ethanol for 290 to 350K, and in a vacuum of $\sim 10^{-6}$Torr for 370K and higher), and after each isochronal annealing the positron lifetime was measured at 100K. The used fast-slow coincidence circuit has a time resolution 220ps in FWHM. The obtained lifetime specta were analysed by using of the computer program "RESOLUTION" [4]. If necessary, the lifetime spectrum was separeted into two components.

## Results and Discussion

### 3.1 2.5MeV electron irradiation

In **Figures 1 and 2** the results for 2.5MeV electron irradiated Fe-16Cr-17Ni and Fe-16Cr-17Ni-0.1P are shown. The initial $\tau_2$ value corresponding to a single vacancy is $\sim$ 160ps, shorter than that in pure Fe. This can be attributed to that the volume of a single vacancy in fcc structured austenite is smaller than that in bcc Fe [5, 6], because fcc has a closed packed structure, but bcc has not. But the theoretical calculation is necessary to

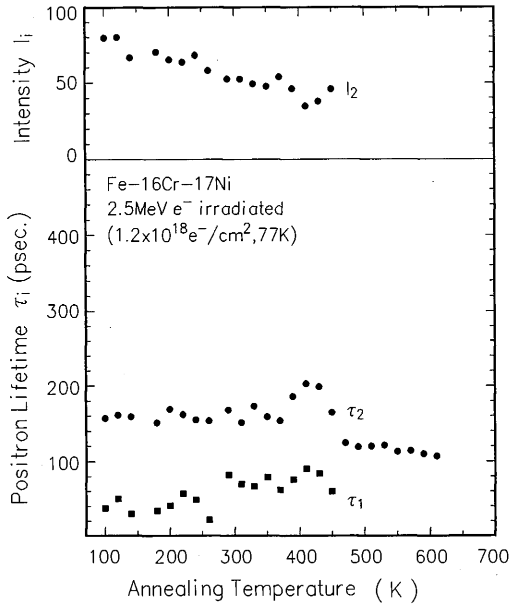

**Figure 1** Isochronal annealing result for Fe-16Cr-17Ni irradiated by 2.5MeV electrons obtained by positron annihilation lifetime measurement.

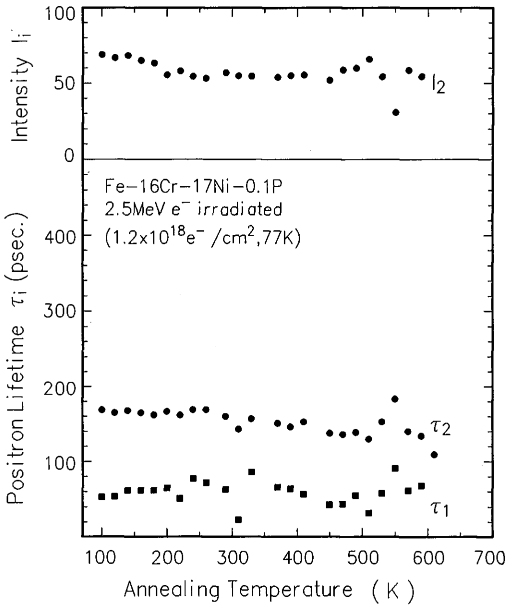

**Figure 2** Isochronal annealing result for Fe-16Cr-17Ni-0.1P irradiated by 2.5MeV electrons obtained by positron annihilation lifetime measurement.

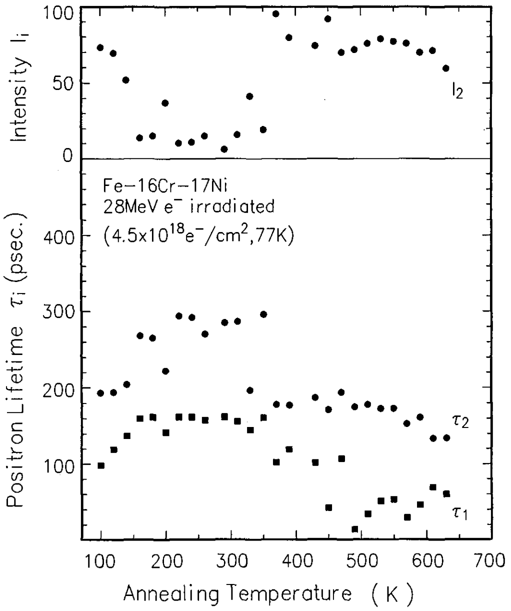

**Figure 3** Isochronal annealing result for Fe-16Cr-17Ni irradiated by 28MeV electrons obtained by positron annihilation lifetime measurement.

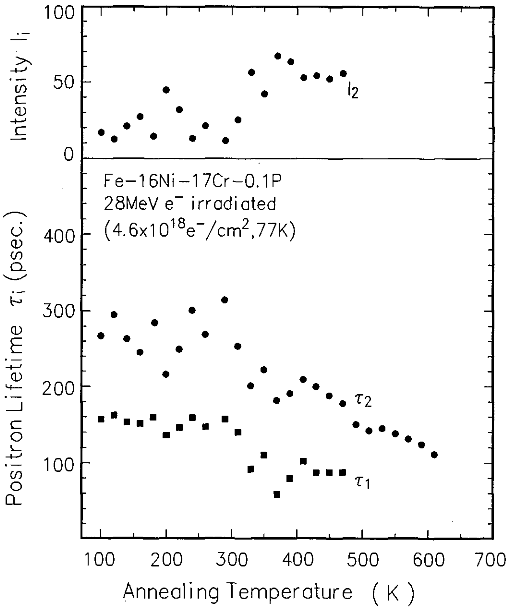

**Figure 4** Isochronal annealing result for Fe-16Cr-17Ni-0.1P irradiated by 28MeV electrons obtained by positron annihilation lifetime measurement.

confirm this.

At 140K, in **Figure 1** decrease of the relative intensity $I_2$ is considered to correspond to the short range migration of the self-interstitial atoms and the annihilation with the vacancies [7]. But no changes are observed in the case of P-doped specimens (**Fig. 2**). This suggests that the interaction between a phosphorus atom and a self-interstitial atoms is strong.

In **Figure 1**, $\tau_2$ increases at 390K, indicating the growing of small microvoids which consist of three or four vacancies. On the other hand, in the case of P doped specimen (**Fig. 2**), this change of $\tau_2$ is not at this temperature but at higher temperature ~530K. So, the phosphorus atoms possibly suppress the formation of microvoids.

### 3.2 28MeV electron irradiation

In **Figures 3 and 4** the results for 28MeV electron irradiated Fe-16Cr-17Ni and Fe-16Cr-17Ni-0.1P are shown.

In both figures, $\tau_2$ indicates long lifetime corresponding to microvoids consist of about 7~8 vacancies in the low temperture of 100 to 260K. This was not observed in the bcc Fe [8]. These microvoids are possibly formed in the cascades produced by high energy electrons. The thermal conduction of the austenitic alloy is much lower than that of pure Fe, so, the temperature rise in the cascade region is considered to be higher in the austenitic alloy than that in bcc Fe, which results in the higher jump rate of vacancies, namely, higher probabiliy of vacancy clustering during a cooling phase of a cascade in the former. Thus microvoids are formed below 100K.

At about 350K, there is a stage where the $\tau_2$ decreases and $I_2$ increases. This stage is possibly attributed to the free migration of single vacancies and the cluster fomation of them. Below this clustering temperature, there are two types of positron annihilation site, the one is the single vacancies represented by $\tau_1$, and the other is the microvoids formed in cascades represented by $\tau_2$, where $\tau_1 \sim 170$ps and $\tau_2 \sim 300$ps. Above this clustering temperature, vacancies cluster as SFTs (stacking fault tetrahedra) [9, 10], because the stacking fault energy is low enough in this alloy. It is known that a SFT gives lower positron lifetime than a single vacany (probably about 140psec) [11]. At the end of clustering process, so many SFTs are formed, and then the $\tau_2$ comes to represent the average lifetime of two sites—microvoids and SFTs, which is about 180ps. On the other hand, during this process the total defect concentration decreases probably due to the recombination of vacancies and interstitial clusters, and then $\tau_1$ becomes to correspond to the matrix lifetime only. Simultaneously, relative intensity $I_2$ becomes to indicate higher value$\sim$80%, because it contains both SFTs and microvoids. As a result, the stage at about 350K is considered to be a vacancy migration stage.

The temperatures of the vacancy free migration is not so different between the two specimens with and without P. This means that the phosphorus atom does not capture the vacancy. But the intensity $I_2$ after clustering is lower in the case of P-doped specimen, so the phoshours atoms suppress the cluster formation.

Beyond 500K, the mean lifetime in P-doped specimen is higher than that without P. One possibility of this is precipitation of $Fe_2P$ [2, 3] enhanced by radiation defects, especially mixed dumbells (P is undersized in this matrix). This process also can explain the suppression of vacancy cluster formation, because it requires vacancies so that P atoms may sit at the lattice sites. Probably positrons are trapped at the boundary sites of the precipitates. However, this model remains no more than speculation, because no calculation of positron lifetime at this boundary has been made. This is the future problem.

## Conclusion

The positron annihilation lifetime mesurements of Fe-16Cr-17Ni and Fe-16Cr-17Ni-0.1P have been made. Some results are obtained to reach the following conclusions.

1. The phosphorus atoms influenced the migration temperature of self-interstitial atoms.
2. A phosphorus atom did not capture a vacancy, but suppressed the vacancy cluster formation, probably due to precipitates $Fe_2P$ formation.
3. The vacancy migration temperature was determined as about 350K and not affected by the presence of P atoms.

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