

Investigation of Point Defect Distribution in Fe-Cr Alloy Using Positron Annihilation Technique

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1. Introduction

As reduced-activation ferritic-martensitic steels, Fe-Cr alloys have been proposed for Gen-IV reactor materials due to the higher swelling resistance, higher thermal conductivity, lower thermal expansion and better liquid-metal compatibility than austenitic steels. However, the mechanical properties of the materials are degraded by irradiation in the end. It is known that the amount of Cr in the alloy determined the properties of the material. For the nuclear reactor materials, the concentration of 8-9% Cr is generally proposed because of good ductility and high corrosion resistance [1].

In order to predict the response of materials to irradiation, it is essential to understand the basic properties of radiation-induced defects, which include the point defects and their clusters. Positron annihilation technique provides information on the characteristics of radiation-induced defects in the atomic level. In particular, positron annihilation lifetime spectroscopy (PALS) is the only technique that can sensitively detect vacancy-type defects in a material. In this study, we investigate the features of open-volume type defects as a function of the electron-irradiation time (2h, 5.5h, 15h) for various of Fe-Cr alloys using PALS.

2. Experimental

2.1 Positron Annihilation Techniques

PAT can be classified into two groups which are distinguished by the sensitivity of positrons to the electron density (① PALS - Positron Annihilation Lifetime Spectroscopy) and to the electron momentum distribution in the sample (② DOBS - Doppler-broadening spectroscopy).

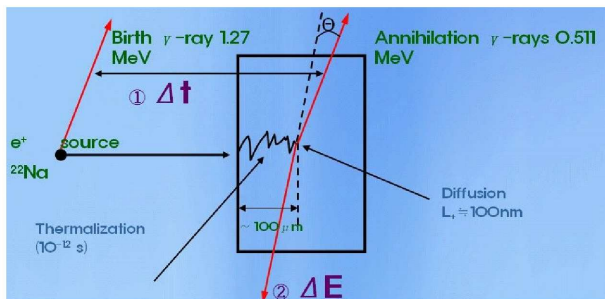


Figure 1. Scheme of positron annihilation. Positrons (start signal with the 1.27 MeV birth- γ) from Na-22 source penetrate the sample, and thermalize (~few ps), and diffuse and annihilate with 0.511 MeV annihilation- γ (stop signal).

We can measure time difference between the appearance of two γ -quanta (start and stop gamma) using BaF₂ scintillators from the PALS. The detected two γ -quanta are converted into analog pulses, which start and stop a time-to-amplitude converter. The amplitude of the output pulse is proportional to the time difference. The PALS system that we set up has a time resolution of 270 ps. On the other hand, in the DOBS, the energy distribution of the annihilation γ -quanta is measured with the germanium detectors. Under the applied high voltage of 2~3kV, the annihilation photons cause a charge separation that is converted into an electrical pulse. Its amplitude is a measure of the photon energy. The positron sources used for measurement consist of ²²Na sandwiched between two Ni foils. The equipments are kept in a room at the constant room temperature to reduce the electronic drift. A simplified diagram of PA measurement system is given in Fig. 2. We used the PATFIT package in PALS and the DualNTW program in DOBS to analyze the data [2].

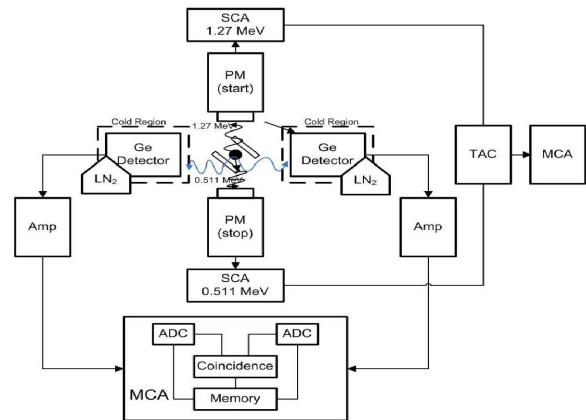


Figure 2. Scheme of positron experiments. (Amp: Amplifier, PM: Photomultiplier, TAC: Time to Amplitude Converter, SCA: Single Channel Analyzer, LN₂: Liquid Nitrogen)

2.2 Samples Examined

The model alloys considered in this study are as follows; pure Fe, Fe-5Cr, Fe-9Cr, Fe-12Cr, Fe-15Cr, pure Cr. Specimens of 10×10×1 mm, attached to a water-cooled target holder made of SS, were subjected to 2 MeV electrons. The samples were irradiated for 2h, 5.5h, and 15.5h with electron flux of 5.625×10^{16} e/m²·s.

The calculated dose rates are 3.3×10^{-6} , 9.1×10^{-6} , and 2.6×10^{-5} dpa.

3. Results

3.1 Positron Annihilation Lifetime Spectroscopy

The variation of the “positron annihilation lifetime” reflects the changes in the defect type and concentration. Figs. 3 shows the results of positron annihilation lifetimes as a function of irradiation time and Cr concentration. In this analysis, the single positron lifetime was used. Attempts to fit the lifetime spectrum with two-lifetime components were not possible with reasonable statistics. The positron bulk lifetimes of Fe and Cr are known to 111 ps and 118 ps, respectively [3]. Before electron irradiation, the bulk values of Fe and Cr are almost same as the previous data. But after irradiation, the positron lifetimes are increased sharply after 5.5h irradiation. From this data, it can be found that as a result of electron irradiation, the defects of vacancy type were produced. It is noteworthy that the lifetime of high-Cr alloy is smaller than that of low-Cr alloy.

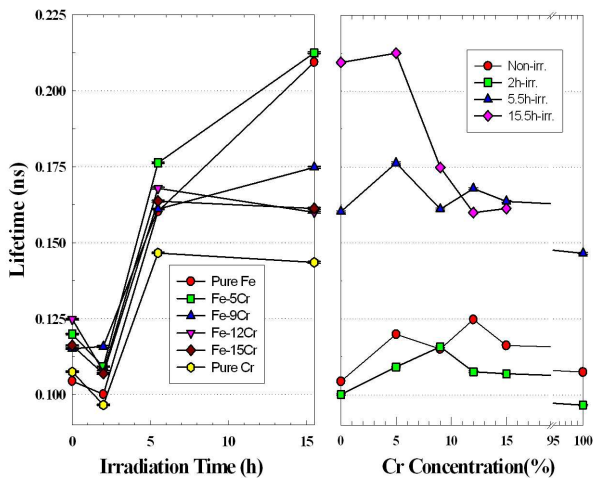


Figure 3. Dependence of positron lifetimes on the irradiation time and concentration percentage of Cr.

3.2 Positron Annihilation Coincidence Doppler Broadening Spectroscopy

From the DOBS, the chemical element can be identified by investigating the characteristic momentum distribution of the core electrons [4]. Fig. 4 shows the results of the ratio curves of the CDB spectra for different Cr-concentration after normalizing relative to pure Fe. In the high-momentum region, there is a clear difference in the CDB ratio spectra between Fe and Cr, which can be used to identify the chemical elements in principle.

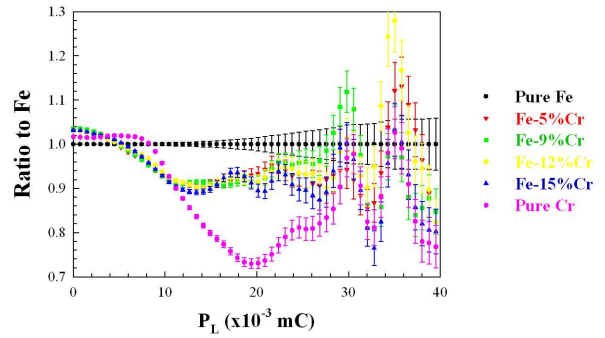


Figure 4. The CDB ratio spectra for different Cr concentration after normalizing relative to Fe.

4. Conclusion

The positron annihilation measurement techniques were applied to investigate the Fe-Cr alloys irradiated by high-energy electrons. From the PALS, it is found that the lifetimes of the irradiated samples increase due to the production of the open-type defects. However, the lifetimes of the high-content Cr alloys did not increase with the irradiation time. It is likely that the presence of Cr in the alloys play an important role in reducing the point defects. We also observed the ratio curves for the Fe-Cr alloys, which were measured by the DOBS. Such an analysis will be useful in identifying the chemical element in the future work. The two PA techniques proposed in this study suggest a high sensitivity method for understanding of materials property.

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