AN APPLICATION OF CYCLOTRON BEAM TO THE STUDY OF RADIATION DAMAGE IN METALS

Chen Keqin, Jin Yunfan, Hou Mingdong, Li Changlin, Cheng Jie, Luo Baoping, Yao Yuying, Wu Meizhen, Tong Zhishen*, Hou Yaoyong*, Li Li*, Cao Chuan**

Institute of Modern Physics, Academia Sinica
57 Nanchang Road, Lanzhou, China

Summary

Radiation damage produced by carbon ion beam of energy 70 Mev in nickel and stainless steel was studied with positron annihilation technique (PAT) and transmission electron microscopy (TEM). Experimental results obtained indicate that stainless steel has greater resistance to irradiation of heavy ions than pure nikel, and also show that temperature of the samples during irradiation seriously affects evolution of radiation damage.

Introduction

The efficiency of production of radiation damage in metals by heavy ion irradiation is much greater than that by neutron, therefore heavy ion beams become a useful tool for simulant investigation of radiation damage produced in materials irradiated by neutron, and that is of general interest in material reseach of nuclear fission reactors and fusion reactors. And it also extends the field of research of the physical process of radiation damage. The research has been carrying on in a wide range of ion energy. Ions with higher energy can produce radiation damage deeper under the surface and the effects will be suitable for research with different analytic methods. In this paper, an experiment is presented on radiation damage in nickel and stainless steel produced by irradiation with carbon ions from cyclotron with energy of 70Mev, and PAT and TEM have been used to study the differences of damage in different materials and the characteristics of damage produced at different temperatures during irradiation.

Experiment

Experiment of Comparison of radiation damage in different materials

The samples used were industrial nickel and martensitic ageing stainless steel. Before irradiation, conventional heat treatment of the samples was proceeded in vacuum. Carbon ions from cyclotron passed through a gold foil, then impinged on the samples. The temperature of the samples during irradiation was kept the room temperature on the average by cold water. The accumulated dose on the samples was given by means of the counts of Rutherford scattering from the gold foil. The level of irradiation by ions has been represented by the number of dpa (displacement per atom) N_d.

$$N_d = \overline{\sigma_d(E)}\,\nu(\overline{E}) \int_0^t \phi(t)\,dt$$

where $\overline{\sigma_d(E)}$ is the average value of displacement cross section $\sigma(E)$ over the energy range from 0 to E [1]. $\nu(\overline{E})$ is the number of displacement by primary knock on atom (PKA), calculated according to Kinchin Pease model. $\overline{E}$ is the mean energy of the PKA. E_d is the threshold energy of displacement, supposing E_d=25ev. $\phi(t)$ is the ion beam intensity. Transmission electron microscope of type JSEM-200 was used to observe the samples electrolytically polished beforehand. The positron liftime in the samples were measured by a conventional fast-slow coincidence positron lifetime spectrometer. The lifetimes τ_j and corresponding intensities I_j were obtained from solution of the equation

$$I(t)=I_1e^{-\lambda_1 t}+I_2e^{-\lambda_2 t}, \quad \tau_j=\frac{1}{\lambda_j}.$$

The Doppler broadening lineshapes were measured by a Ge(Li) spectrometer.

Experiment of irradiation at different temperatures

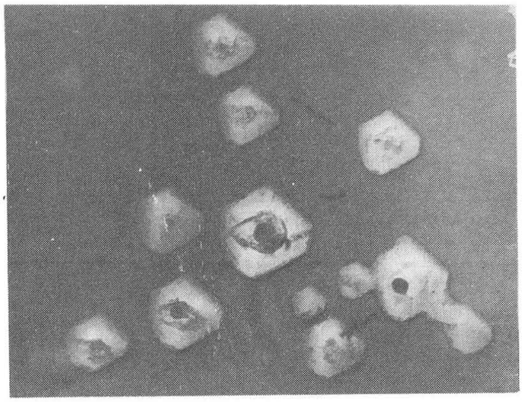
The samples of nickel with purity of 99.999% were annealed under 850°c for 30 min. and irradiated by carbon ions from cyclotron at temperature of 385°c, 485°c, 535°c respectively. Accumulated dose was 0.3--0.4 dpa on the samples. Other conditions were similar to the above experiment.

Results and discussion

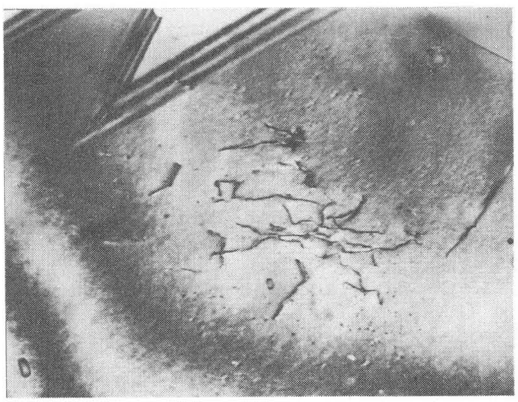
Comparison of different materials

Observation under TEM Figure 1 shows the micrographs of nickel samples irradiated and unirradiated by carbon ions. After irradiation a number of hexagonal voids appeared in the samples of nickel. They are similar to those in the paper reported by M.B.Lewi et al.[2] Because the density of ion beam might have greater change during irradiation, the temperature of the samples might rise locally for a moment. The voids are

*Lanzhou University
**Institute of High Energy Physics. Academia Sinica



(a)



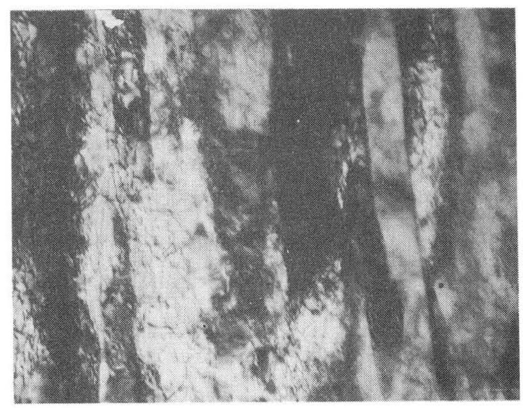
(b)

Figure 1: TEM micrographs of nikel samples showing a comparison between (a) sample irradiated with a dose of about 0.15 dpa and (b) unirradiated sample.

considered to airse from the diffusion and agglomeration of the radiation - produced vancanies at higher temperature. The dark spot obsereved in the voids perhaps is due to the gathering of carbon and other impurity. Only sparse dislocation loops and dislocation lines (in lower density) existed in the unirradiated sample. On the other hand, no change was obsereved in the irradiated samples of stainless steel (Fig 2). The reason probably is that the density of dislocation in the sapmles of stainless steel was much higher than that in the samples of nickel, and there also existed a large number of second phase precipitate and grain boundaries, which might restrain the formation and growth of voids.

Positron annihilation The results obtained are given in table 1. Both the annihilation lifetimes τ_1, τ_2 and the lineshape parameter S for the nickel samples increase after irradiation. The increase in τ_1(=168 ps) shows that irradiation of carbon ion created a number of displacement atoms in the nickel samples and many vacancies and dislocations were formed. Dlubek pointed that when some metals contained vacancies and dislocations the positron lifetime would have value between 150 to 200 ps [3]. The increase in τ_2 (=515 ps) indicated that voids have been formed simultaneously

(a)



(b)

Figure 2: The micrographs of stainless steel samples showing a comparison between (a) samples irradiated with a dose of about 0.15 dpa and (b) unirradiated sample.

Samples	τ_1(ps)	τ_2(ps)	I_2(%)	S
Nickel unirradiated	129	389	12	2.97
Nickel irradiated	168	515	7	3.39
Staintess seeel unirradiated	155	444	8	3.59
Stainless steel irradiated	150	432	9	3.48

Table 1: Experimental results of PAT. τ: lifetime, I: intensity, S: lineshape parameter with irradiation dose of 0.36 dpa.

in the nickel sample after irradiation.The lineshape parameter S for nickel samples increases after irradiation. It indicates that at the place, where positron annihilated in the irradiated Ni samples, the density of core electrons was reduced, comparing to that in the unirradiated ones. This also suggests that in the irradiated Ni samples there were vacancies and voids produced. But for the irradiated samples of stainless steel the liftimes τ and lineshape parameters S have not changed evidently.

Comparison of different irradiation temperatures

Data on the average size and concentration of voild from TEM are listed in table 2. It is seen that at temperatures

irradiation temperature(°c)	average diameter(Å)	average density(cm^{-3})
385	135	$2.01x10^{15}$
485	579	$3.56x10^{13}$
535	790	$1.60x10^{13}$

Table 2: The dependence of size and density of voids on irradiation temerature for nickel samples with dose of 0.3-0.4 spa.

between 0.2 and 0.6Tm (Tm is the melting point in absolute temperature scale) voids were formed in the irradiated nickel samples. The size of voids is several hundreds Å and increases with increase of irradiation temperature, while the concentration decreases with the increase of temperature.

The samples were also studied with the positron annihilation technique before being observed under TEM. The annihilation lifetime are given in table 3. For the

irradiation condition(°c)	τ_1(ps)	τ_2(ps)	2(%)
Unirradiated	110	369	4.5
385	115	432	4.6
485	116	437	4.5
535	117	497	3.3

Table 3: Experimental results of PAT. λ : lifetime, I:intensity with irradiation dose of 0.3-0.4 dpa.

irradiated samples τ_2 has obviously larger values than the unirradiated ones, and it also increases with the increase of irradiation temperature, The first lifetime τ_1 has not changed significantly and is almost equal to the bulk lifetime. This is different from the results shown in table 1. The reason is that the purity of the samples was better and the annealing temperature was higher than that in the first experiment.

Reference

(1) A.N.Goland, Ann. Rev. Nucl.Sci., 12(1969) 243
(2) M.B.Lewi et al., Nucl. Instrum. Meth., 167 (1979) 233
(3) G.Dlubek et al., J.Phys.F,9,10, (1979) 1961