Production and Recovery of Radiation Damage in α-Particle Irradiated Fe-Si Alloys

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Ohura M., Nagata S., Hanada R. and Yamaguchi. S.*

Department of Nuclear Engineering, Institute for Materials Research,Tohoku University*

Introduction

The production and recovery of radiation damage in metals have been extensively studied for past three decades. Although there still exists some controversy on the nature of defect in stage III, basic properties of point defects in irradiated metals are now believed to be well understood. Most of the knowledge of point defects in metals, however, have been obtained using indirect methods as resistivity measurement and so application of direct methods as Mossbauer spectroscopy, PAC or NMR/ON or channeling are urgently needed.

Here in the present the resistivity measurement, however, is applied to study high concentrated Fe-Si alloys. The purposes of experiment are threefold. Namely, (1) To obatin general recovery behavior in a concentrated Fe-alloy.(2) To study the effect of Si concentration on Fe recovery stages, mainly, on stage I and (3) To develope a new method of irradiation to avoid beam heating and to examine whether it works or not.

Experimentals

The Fe-Si alloys specimens used in the present were prepared by melting high pure electrolytic iron (Showa Denko) with a known amount of Fe-Si mother alloy in an Ar atomosphere. The alloys ingots were cold rolled to 0.2mm thick sheet and cut into specimens of 1mm width and 20mm length. Specimens were thinned down to 30-40 μm thickness chemically after an UHV vacuum annealing at 1000 K. Also Fe specimens (R.R.=1500.Materials Research Co.) were simultaneously studied for comparison purpose.

The specimens were irradiated by 50MeV α particles with the current density of 0.3 $\mu A/cm^2$ for about 10^4s. The irradiation dose was thereforeabout 10^{16} α/cm^2. To prevent the beam heating by the irradiation, specimens were dipped into liquid nitrogen during the irradiation.The irradiation chamber was separated from the cyclotron vacuum by a 20 μm stainless steel foil. The beam passed through the foil and also liquid nitrogen layer of about 1mm thickness. The overall energy loss of the beam was estimated as 13.6MeV by TRIM code.

The present irradiation method with dipping specimens in liquid nitrogen is quite convenient to perform irradiation in reasonably short time. However, if liquid nitrogen is exposed to the ambient atomosphere, oxygen is absorbed into it and ozone is formed by irradiation. The ozone is quite active to cause detonation when liquid nitrogen evaporates out. So cares must be taken if the method is applied.

After the irradiation specimens were transferred to a cryostat to measure the reisitivity increase by the irradiation and also to perform isochronal annealing. The annealing schedule was 2K/3 $\times 10^2$s between 77K and 130K and with wider temperature interval above 130K. All the resist-vity measurements were performed in liquid nitrogen.

Experimental results

(1) Production of defects by irradiation.

Fig.1 shows the resistivity increase for several specimens during the irradiation. The increase is due to the radiation damage created in the lattice by the irradiation of the energetic α-particles. The damage consists of Frenkel pair,namely, pair of a self-interstitial atom (SIA) and a lattice vacancy (V). Different from other metals, the migration energy of SIA has been known exceptionally high in Fe and so most of the damage remains in the lattice without annealing out even at 77 K.

The concentration of Frenkel pair is estimated as $5 - 10 \times 10^{-3}$ after a typical 0.3 μA/cm^2 $\times 10^4$s irradiation run. Because of the non-uniformity of the irradiating beam, the production rate is different by a factor of two among three specimens for each irradiation run. In the following annealing experimental results, therfore, the resistivity recovery will be shown by a percentage scale of $(\rho\ (T) - \rho_0) / (\rho_i - \rho_0)$,where ρ_0 and ρ_i are the resistivty at 77K before and after the irradiation, respectively. $\rho(T)$ is the resistivity at 77K after the annealing at T.

(2) Recovry result.

Fig.2 shows a typical recovery spectrum for a pure Fe specimen . As is well known, the recovery takes place in three stages, namely stage I(77-150K), stage II(150-180K) and stage III(150-220K). The stage I seems to consist of several substages, although the populations of them are not reproducible for each run and so the presence of them is not conclusive at present.

Fig.3 shows the recovery spectrum for the concentrated Fe-Si alloys together with a pure Fe specimen. From the figure, the trend is observed that the amount of stage I recovery decreases with the Si concentration. This trend is observed more clearly in Fig.4 (the right side) where the amount of each recovery stage is plotted against the Si concentration together with the same plot for the dilute Fe-Si alloys by Maury et al.$^{1)}$ (the left side).

The main conclusion of the present experiments for the concentrated alloys is that the reduction of the stage I is in the stage I_D region. This is in contrast to the dilute alloy1) result where only I_E and not I_D is found to be reduced by Si alloying.

Discussion

The present observation of the I_D reduction in the concentrated Fe-S_i alloys is quite reasonable in the framework of stage I recovery model of pure metal proposed by Corbett et al.2,3) Namely, when irradiated by an energetic particle, an atom is displaced from the lattice site leaving a vacancy behind and eventually it sits on an interstitial site to form SIA. The distance between them is rather small and so most of them recombine with each other when the temperature becomes high enough for SIA to migrate. ID corresponds to this "correlated" recombination of SIA and V. During the process, however, part of SIA fails to recombine with it's own vacancy and has to migrate a quite long distance before it meets with other vacancy. The I_E corresponds to this SIA's long distance migration and recombination. The experimental evidence by Corbett et al for this model are (1) the activation energy for I_D and I_E is the same, (2) the order of reaction is the 1st for I_D and 2nd for I_E and (3) the experimental isochronal and isothermal curves can be fitted on an universal curve calculated by Waite's diffusion limited reaction theory.

If this model of I_D and I_E is applied, the effect of Si may be interpreted as follows. First we must assume Si atoms act as a trapper for SIA. Namely when a SIA meets wit a Si atom, they form a pair which is immobile at the temperature where otherwise a SIA is mobile. Because of the trapping, the recombination of SIA and V is hindered thus giving rise to the reduction of a recovery stage. This trapping takes place when the diffusion distance of SIA(r_D) is comparable with the distance (r_{si}) between Si atoms. For the case of Maury et al dilute alloy, rsi is a few ten atomic distance which is comparable with the r_D at 130 K(the center temperature of I_E). So SIA trapping by Si atom only takes place in I_E where a SIA performs long range migration. On the other hand in the present concentrated alloys, r_{si} is a few atomic distance which is comparable with r_D at 115 K(the center temperature of I_D). So SIA trapping can take place even in I_D where SIA performs short range migration. It should be noted the above estimation of r_D has been done with assuming the same activation energy of 0.27 eV (4) for both I_D and I_E.

The present results together with Maury's result can be well explained Corbett's stage I model with Si atom trapping effect for SIA. This suggests properties of the SIA in concentrated Fe alloys are similar with that of pure Fe at least up to the Si concentration of 6 at.%..

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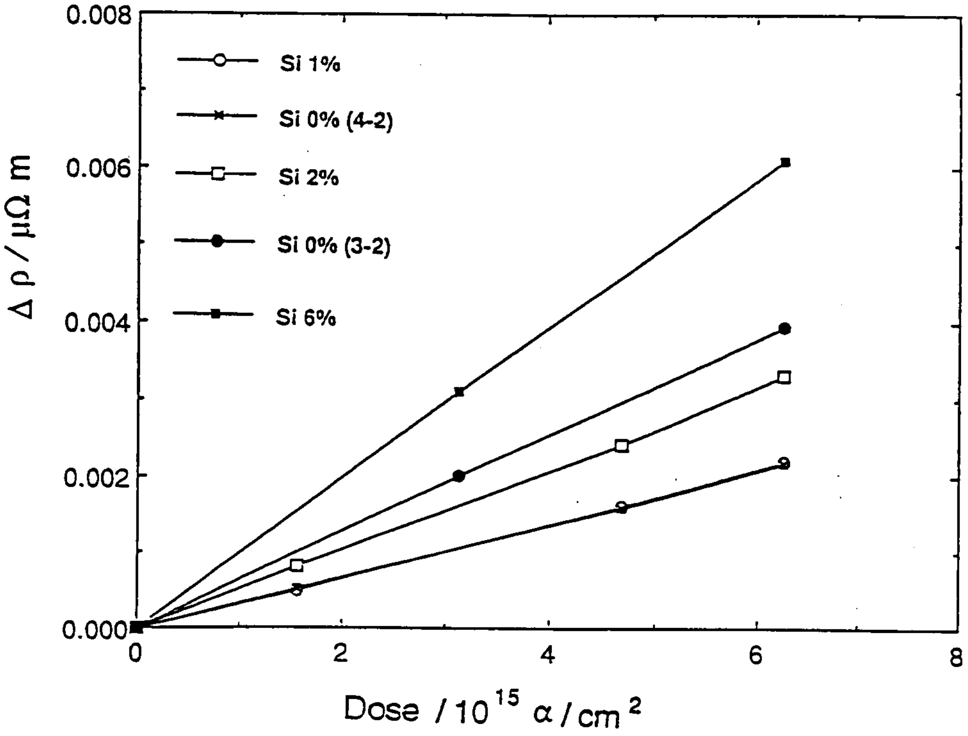

Fig.1 Resistivity production curves for Fe-Si alloys and pure Fe.

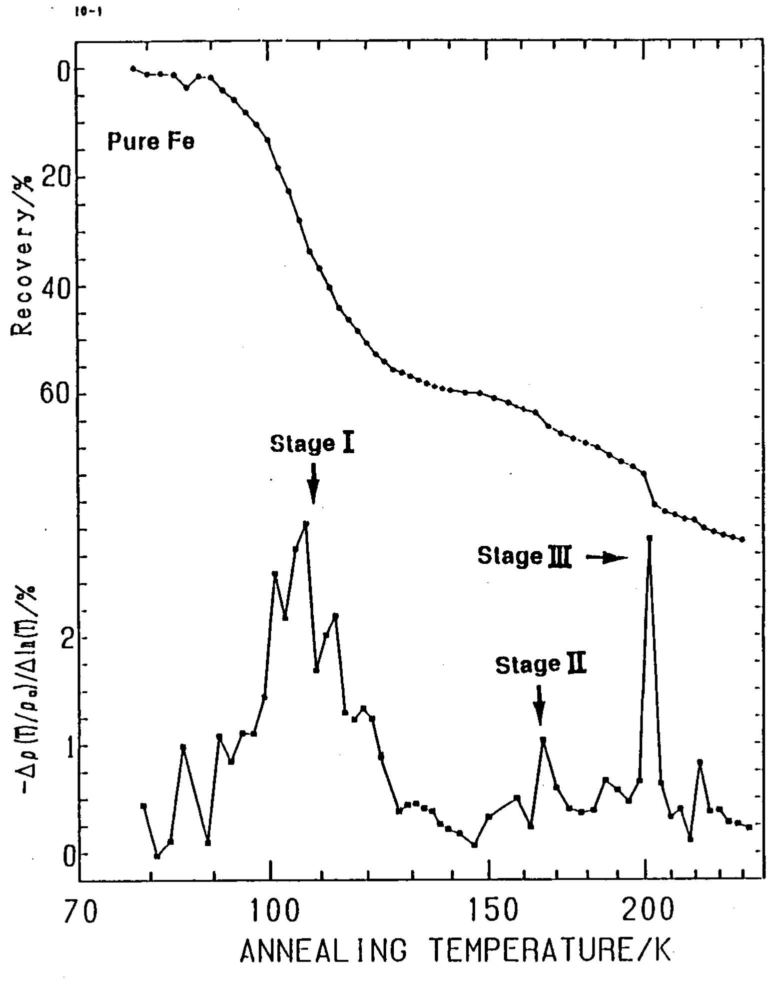

Fig.2 Resistivity recovery curve for pure Fe. The resistivity change after the annealing at each temperarure are plotted.(upper). Corresponding derivatives with respect temperature are shown (lower).

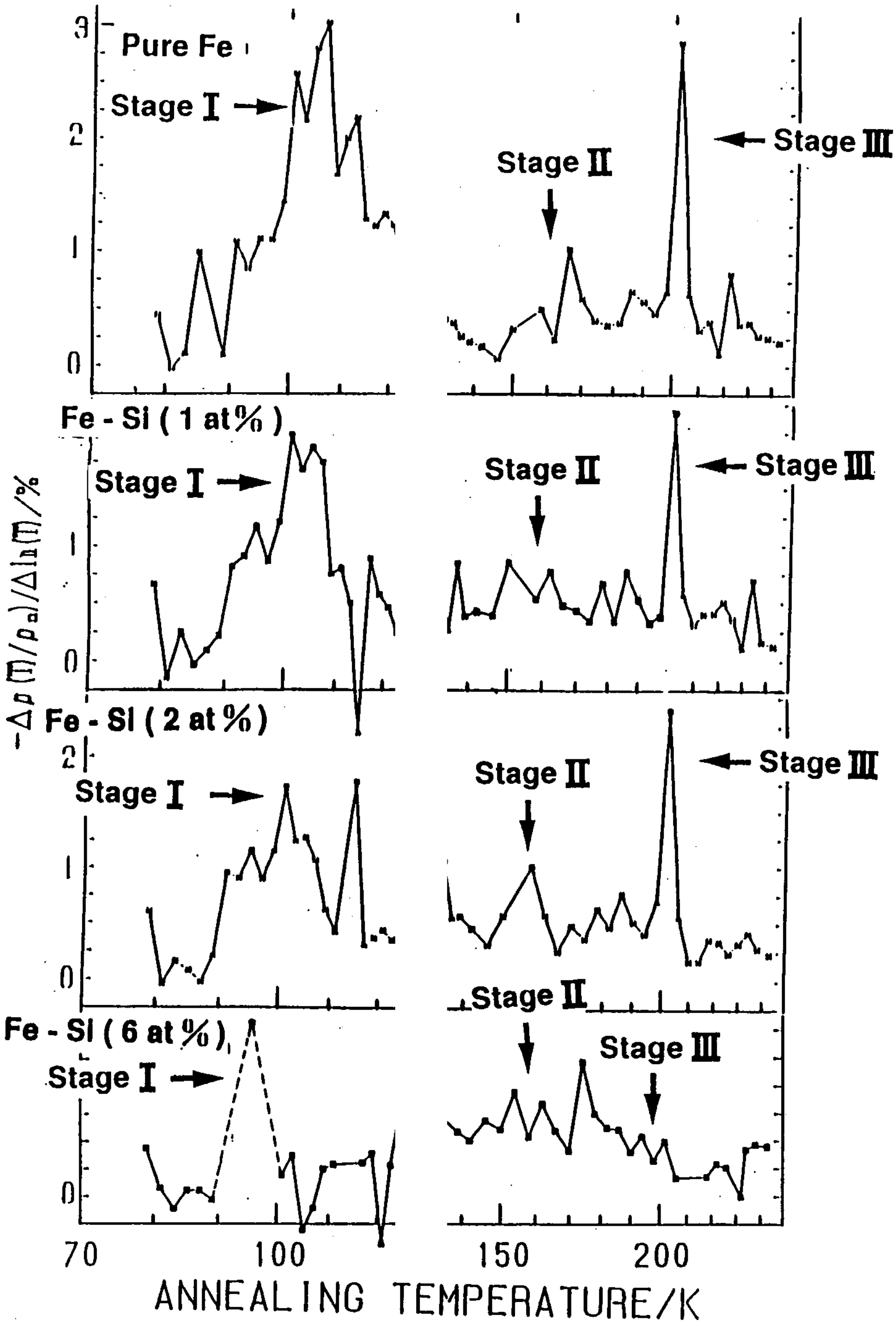

Fig.3 The recovery spectrum (the derivatives) for Fe-Si alloys (lowerthree) and for pure Fe(top).

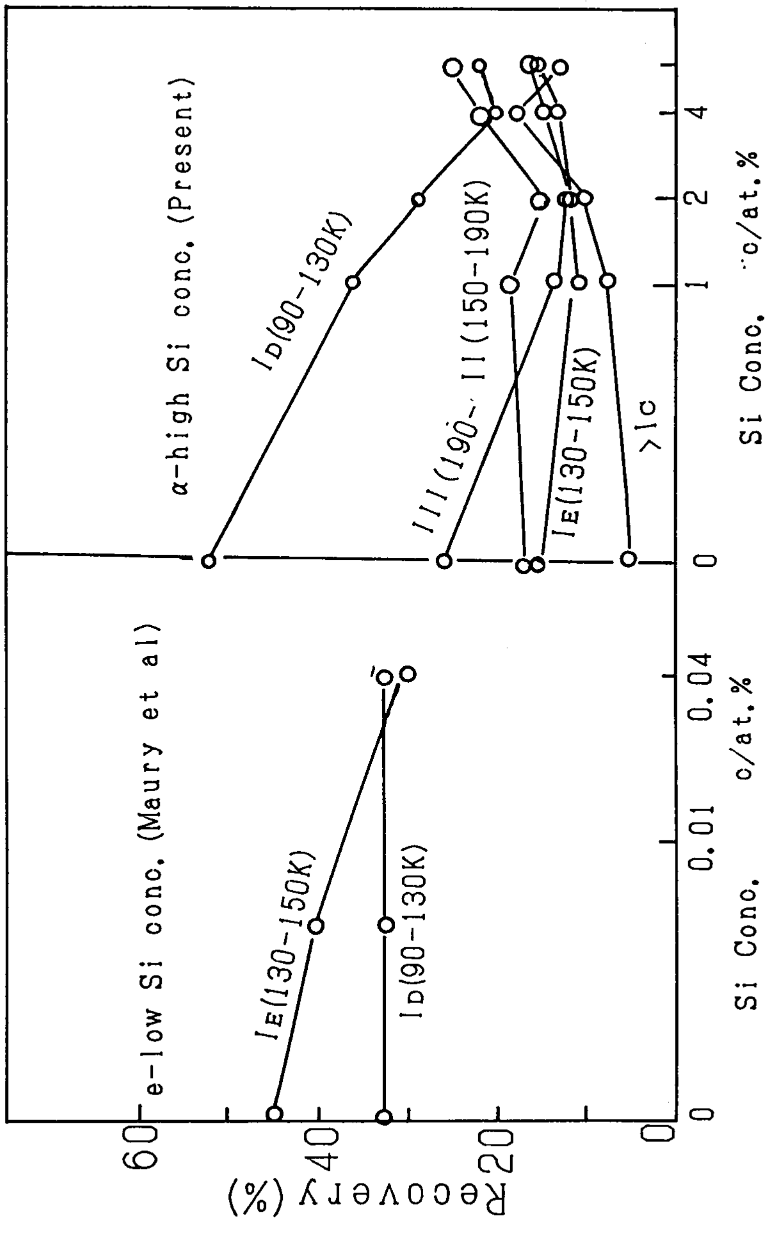

Fig.4 The amount of recovery in each stages.