Reversible Structural Relaxation in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ Amorphous Alloys

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The reversible processes of structural relaxation in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ amorphous alloys were investigated by means of electrical resistivity and Mössbauer effect measurements, and these are then compared to each other and discussed. A significant difference in the contribution of chemical short range ordering (CSRO) to the electrical resistivity is observed between the two amorphous alloys: the resistivity increases with annealing temperature in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy but decreases in $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy. The crossover effect for the resistivity is also observed in both alloys. A kinetics of CSRO in each alloy can be described as a log-normal distribution of relaxation times. The mean activation energy of the CSRO process is 222.0 kJ/mol for $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy and 135.1 kJ/mol for $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy. As a result it is suggested that the reversible resistivity changes of $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloys are responsible for CSRO between Fe and Si atoms in the former, and that between Fe and Ni atoms in the latter.

(Received July 16, 1988)

Keywords: amorphous alloy, chemical short range ordering (CSRO), structural relaxation, effects of annealing, crossover effect, electrical resistivity, Mössbauer effect

I. Introduction

Since structural relaxation has considerable influence on the various physical properties of amorphous alloys, it is quite important to clarify relaxation phenomenon not only for technical applications but also for understanding their structure. Structural relaxation involves the following two kind of atomic rearrangements, namely topological short range ordering (TSRO) and chemical short range ordering (CSRO)⁽¹⁾. The changes of TSRO are irreversible, in which the ordering is considered to influence the topology of amorphous network with the reduction and redistribution of free volume. On the other hand, the changes of CSRO are reversible, in which the ordering is considered to occur through short range atomic diffusion without a significant change of the amorphous network. Strictly speaking, these two processes may not be totally independent.

So far, extensive studies have been carried out on the changes of short range ordering during structural relaxation in amorphous alloys⁽²⁾⁻⁽⁵⁾. Very few studies have, however, been made on amorphous Fe-B-Si alloys in spite of the importance of its applications as magnetic material. We have studied structural relaxation in amorphous Fe-B-Si alloys by electrical resistivity measurements⁽⁶⁾⁽⁷⁾ and found that the reversible changes of resistivity due to CSRO during temperature cycles were observed in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy⁽⁸⁾.

The purpose of this paper is to report the results of comparison between the reversible processes of structural relaxation in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy.

II. Experimental Procedure

Two kinds of amorphous ribbons of compositions $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ were used: the former was prepared about 1 mm in width and 25 μm in thickness by a single-roller melt spinning method and then its amorphousness was checked by X-ray diffraction, and the latter, obtained from Allied Corporation, was about 2.5 mm in width and 38 μm in thickness.

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The electrical resistivity was measured at the liquid nitrogen temperature by a standard four-terminal method. The specimens were equipped with current and potential leads by spot-welding, and then placed in a quartz tube (inside diameter = 10 mm) with thermocouple. The as-quenched specimens were first given the preanneal treatment for 30 ks at 623 K ($\text{Fe}_{78}\text{B}_7\text{Si}_{15}$) or for 1.8 ks at 593 K ($\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$) in order to remove the further ordering of TSRO. For the annealing treatments, the quartz tube containing the specimen was evacuated to a vacuum of 10^{-3} Pa and then dipped into a salt bath held at annealing temperature. The typical heating-up time to annealing temperature from room temperature was about 120 s. At the end of annealing the quartz tube was quenched into a brine held at 253 K, and simultaneously a cold helium gas was introduced into the tube. An initial quench rate was about 30 K/s.

The Mössbauer effect measurement were carried out at room temperature using a constant acceleration type spectrometer in transmission geometry. The gamma-ray source was 0.37 GBq (10 mCi) ^{57}Co doped in Rh. The velocity scale was calibrated against a 25 μm foil of natural $\alpha\text{-Fe}$.

III. Results and Discussion

Figure 1 shows the electrical resistivity change R/R_0 caused by isochronal annealing in the preannealed amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy,

Fig. 1 Temperature dependence of electrical resistivity of amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ preannealed at 623 K for 30 ks during isochronal annealing ($t_a = 1.2$ ks), measured at 77 K.

where the annealing time t_a is 1.2 ks at each annealing temperature. It can be seen that the values of resistivity changes in annealing cycles (cooling and heating runs), within experimental error, lie on the same straight line in the annealing temperature range from 580 K to 643 K. This indicates clearly a reversible change, which can be considered to be caused by chemical short range ordering (CSRO). Hence, the evolution of CSRO associated with decrease of temperature contributes to decrease of the resistivity. At annealing temperatures below 580 K the resistivity is almost constant, which can be ascribed to the situation that the annealing temperature is too low for CSRO to take place in an isochronal annealing time (1.2 ks). It is to be noted that the amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy with a single metallic species shows the reversible change of resistivity.

Figure 2 shows the resistivity change caused by isochronal annealing ($t_a = 0.6$ ks) in the preannealed amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy. The reversible change of resistivity is observed in the annealing temperature range from 490 K to 593 K. In this case the development of CSRO with decreasing temperature increases the resistivity in contrast with case of $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy. A similar result has been reported by Balanzat⁽⁹⁾. The slopes of the lines which denote the reversible change in Figs. 1 and 2 are $1.2 \times 10^{-5} \text{ K}^{-1}$ and $-2.8 \times 10^{-5} \text{ K}^{-1}$, respectively. This means that there is a signifi-

Fig. 2 Temperature dependence of the electrical resistivity of amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ preannealed at 593 K for 1.8 ks during isochronal annealing ($t_a = 0.6$ ks), measured at 77 K.

Fig. 3 Change in electrical resistivity of amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ preannealed at 623 K for 30 ks and amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ preannealed at 593 K for 1.8 ks during the sequence of isothermal anneals at various temperatures, measured at 77 K.

cant difference between these two alloys with respect to the contribution of CSRO to the resistivity.

In order to see a kinetic behavior of the reversible resistivity due to CSRO in these alloys, the preannealed specimens mentioned in Section II were isothermally annealed again at various temperatures as shown in Fig. 3. It can be seen from the figure that a quasi-equilibrium value of resistivity for each annealing temperature exists and the same quasi-equilibrium value of resistivity can be reached in spite of starting from different initial states. This fact guarantees the existence of the quasi-equilibrium state corresponding to each annealing temperature adopted here. For a kinetic analysis of the above results, the relaxation function $\Psi(t)$ is calculated from the isothermal annealing curve of resistivity using the following equation:

$$\Psi(t) = \frac{R(t) - R_0}{R_\infty - R_0}, \quad (1)$$

where R_0 and $R(t)$ are the resistivities of the specimen at the beginning and at time t , respectively, and R_∞ their quasi-equilibrium value. The kinetics of CSRO in amorphous alloys is generally not simple and $\Psi(t)$ may be not reproduced by a kinetic equation with a single relaxation time. The evidence that at least two

relaxation times are required in the CSRO process has been provided by the crossover experiment, as first shown by Boesch *et al.*⁽¹⁰⁾ for the structural relaxation process of oxide glasses where they measured the refractive index. In the present work the crossover experiment for resistivity was carried out on both amorphous alloys. The preannealed specimen of $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy was annealed at 543 K until its resistivity reached the quasi-equilibrium value for the specimen annealed at 563 K, and then the annealing temperature was abruptly changed to 563 K and keep constant at this temperature. The resistivity change versus time at 563 K is shown in Fig. 4(a). There is a rapid initial increase in the resistivity after which it slowly decreases back towards the quasi-equilibrium value at 563 K. A similar experiment was also made on $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy. In this case the preannealed specimen was annealed at 430 K until its resistivity reached the quasi-equilibrium value for the specimen annealed at 473 K. The resistivity change at 473 K is shown as a function of time in Fig. 4(b). The crossover

Fig. 4 Crossover effect for electrical resistivity of amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$. Solid lines are determined by eq. (2) for the fast (M_1) and slow (M_2) relaxation shown by broken lines.

effect is clearly observed in both $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloys. To our knowledge there have been few reports of the crossover effect for resistivity so far, except that for the Curie temperature⁽¹¹⁾⁽¹²⁾ and Young's modulus⁽¹³⁾. The two-relaxation-time model proposed by Boesch *et al.*⁽¹⁰⁾ is then used to explain the results of the crossover experiment. The model assumes two relaxation processes of equal strength, each having a single relaxation time and both leading to the same quasi-equilibrium value of resistivity. The average resistivity $R(t)$ is thus represented by

$$R(t) = 1/2[M_1(t) + M_2(t)] \quad (2)$$

and

$$M_i(t) = R_\infty + R_i \exp(-t/\tau_i), \quad (3)$$

where R_i is the initial value of resistivity associated with a process i having relaxation time τ_i . The results of the calculation using the model are shown by solid lines and also the contributions of the two processes (M_1 and M_2) by broken lines in Fig. 4. The agreement between the calculated and experimental results is satisfactory. The parameters used in the calculation are given in Table 1. The ratio of the relaxation times τ_2/τ_1 for $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy is much larger than that for $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy. Greer *et al.*⁽¹¹⁾ have reported a large ratio of the relaxation times in the crossover experiment for the Curie temperature of $\text{Fe}_{80}\text{B}_{20}$ alloy and suggested that two relaxation times might correspond to two distinct processes of atomic rearrangement: a fast process involving the variation of the equilibrium free volume with temperature (just like vacancies in crystals) and a slow process involving the variation of equilibrium chemical ordering. Any direct evidence supporting this suggestion is not yet available.

In practice the ordering process is unlikely to involve only two distinct processes with unique

relaxation times. It is more realistic to be described by a spectrum of relaxation times $Q(\tau)$ such that

$$\int_0^\infty Q(\tau) d\tau = 1. \quad (4)$$

It is then supposed that the relaxation times are distributed according to a Gaussian distribution in $\ln \tau$. In this case it is useful to adopt the following expression

$$1 - \Psi(t) = \exp[-(t/\tau_m)^{1/\beta}], \quad (5)$$

where τ_m is the mean relaxation time and β is the half-width in the distribution at $1/e$ of its maximum. The mean relaxation time can be obtained from the time to satisfy $1 - \Psi(t) = 1/e$ in eq. (5) for each annealing temperature. The estimated values of τ_m for both $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloys are plotted against $1/T_a$ in Fig. 5. Since $\ln \tau_m$ is linear with $1/T_a$, τ_m obeys an Arrhenius relation

$$\tau_m = \tau_0 \exp(E/kT), \quad (6)$$

where E is the mean activation energy of CSRO, τ_0 is a pre-exponential factor and k Boltzmann's constant. The values of E and τ_0

Fig. 5 Arrhenius plot of the mean relaxation time. $\circ$: amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$, $\bullet$: amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$.

Table 1 Relaxation parameters for the data of Fig. 4 fitted to two relaxation mechanisms.

Sample	R_1	R_2	τ_1/s	τ_2/s
$\text{Fe}_{78}\text{B}_7\text{Si}_{15}$	-6.6×10^{-4}	6.7×10^{-4}	177	1005
$\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$	49×10^{-4}	-49×10^{-4}	209	260

calculated by the method of least squares are as follows: $E=222.0$ kJ/mol, $\tau_0=6.1 \times 10^{-18}$ s for $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy and $E=135.1$ kJ/mol, $\tau_0=5.6 \times 10^{-13}$ s for $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy. Balanzat⁽⁹⁾ has obtained the values of $E=157$ kJ/mol and $\tau_0=10^{-14}$ s in $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy. Balanzat *et al.*⁽¹⁴⁾ have also reported that the reversible resistivity change during structural relaxation in $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy is due to CSRO between Fe and Ni atoms. It is interesting that the value of E for $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy containing one transition-metal element is larger than the value for $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy containing two transition-metal elements. The former closes to the activation energy for diffusion of Fe or Si in the Fe-base amorphous alloys⁽¹⁵⁾⁽¹⁶⁾. Hence, it is considered that the reversible resistivity change in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy is due to CSRO between Fe and Si atoms.

Figures 6 and 7 show the relation between $\Psi(t)$ and $\log(t/\tau_m)$ in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy, respectively. All experimental data points are plotted in the figures with the theoretical curves obtained by eq. (5). In either amorphous alloy, all the points almost lie on the theoretical curve for $\beta=2$. As a result the kinetics of CSRO in each alloy can be approximately described as a log-normal distribution of relaxation times with $\beta=2$. A similar value of $\beta(=2.5)$ has been obtained for the kinetics of CSRO in

Fig. 6 Change in normalized resistivity as a function of $\log(t/\tau_m)$ in amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ preannealed at 623 K for 30 ks. Solid lines are the theoretical curves expressed by eq. (5).

Fig. 7 Change in normalized resistivity as a function of $\log(t/\tau_m)$ in amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ preannealed at 593 K for 1.8 ks. Solid lines are the theoretical curves expressed by eq. (5).

$\text{Fe}_{40}\text{Ni}_{40}\text{B}_{20}$ ⁽¹⁷⁾ and CoNiFe-SiB amorphous alloys⁽¹⁸⁾.

Furthermore, the Mössbauer experiments were performed on both amorphous alloys in order to investigate changes in local environment around Fe atom with CSRO. Figure 8 shows the Mössbauer spectra taken at room temperature for $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy annealed at various temperatures for an annealing time 0.6 ks. Each spectrum corresponds to the evolution of reversible resistivity change. In this case it is seen that the intensities of the second and fifth lines reduce with decrease of annealing temperature T_a (development of CSRO). The changes in the shape of the spectrum with CSRO were however hardly observed in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy. The distribution of magnetic hyperfine field $P(H)$ associated with each spectrum was obtained using a truncated Fourier series method⁽¹⁹⁾. The resultant $P(H)$ are shown in Figs. 9 and 10. The curve of $P(H)$ for $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy becomes narrower with increase of CSRO, while no change with CSRO appears in the $P(H)$ curve for $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy. Since the curve reflects a distribution of structurally inequivalent Fe sites, narrowing of the curve for $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy indicates a variation of the local environment around Fe atom. On the other hand, it should be considered that such a local change in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy does not affect the magnetic

Fig. 8 Mössbauer spectra of amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ measured at room temperature after isochronal annealing ($t_a=0.6$ ks).

Fig. 9 Change in internal magnetic field distribution $P(H)$ of amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ after isochronal annealing ($t_a=1.2$ ks).

Fig. 10 Change in internal magnetic field distribution $P(H)$ of amorphous $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ after isochronal annealing ($t_a=0.6$ ks).

hyperfine interaction. These results obtained above explain that the CSRO dependence of change in reversible resistivity is much larger in $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy than in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy as seen in Figs. 1 and 2.

Figure 11 shows the temperature dependence of Mössbauer parameters obtained by using the method of Lines and Eibschütz⁽²⁰⁾⁻⁽²²⁾. In the figure $\langle\delta\rangle$, $\mu_N\langle H\rangle$ and $\langle\alpha_+\rangle$ are the mean value of isomer shift, hyperfine field and second-order perturbation of energy levels due to electric quadrupole interaction, respectively. The pointed brackets imply average over all iron sites in the specimen. The Mössbauer parameters do not change remarkably with CSRO in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy. On the other hand, $\langle\delta\rangle$ and $\langle\alpha_+\rangle$ decrease with CSRO in $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy. Pollard *et al.*⁽²³⁾ have reported that $\langle\delta\rangle$ decreases and $\langle\alpha_+\rangle$ tends to decrease with Fe content in amorphous $\text{Fe}_x\text{Ni}_{80-x}\text{Si}_{10}\text{B}_{10}$ alloys. Raj and Sathymoorthy⁽²⁴⁾ have also shown a similar result on the change in $\langle\delta\rangle$ with Fe content in the same system. Consequently, it is considered that the average number of Fe nearest neighbors around a particular Fe atom increases with decreasing temperature (development of CSRO). The reversible changes in the electrical

Fig. 11 Change in Mössbauer parameters of amorphous $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ after isochronal annealing.

resistivity in $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy presumably results from formation of CSRO between Fe and Ni atoms explained above. However, the details of ordering between Fe and Si atoms in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy is not clear at this point.

IV. Conclusion

The reversible processes of structural relaxation in $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ amorphous alloys were studied by means of electrical resistivity and Mössbauer effect measurements. The conclusion are as follows:

(1) There is a significant difference between these two amorphous alloys with respect to the contribution of CSRO to the resistivity: the slope of the line which denotes the reversible resistivity change is $1.2 \times 10^{-5} \text{ K}^{-1}$ for the $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy and $-2.8 \times 10^{-5} \text{ K}^{-1}$ for the $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy.

(2) The crossover effect for the resistivity was observed in both $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ and $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloys. The kinetics of CSRO in each alloy can be approximately described as a log-normal distribution of relaxation times with $\beta=2$. The mean activation energy of the

CSRO process was 222.0 kJ/mol for the $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy and 135.1 kJ/mol for the $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy.

(3) The reversible resistivity changes in the $\text{Fe}_{78}\text{B}_7\text{Si}_{15}$ alloy are ascribed to CSRO between Fe and Si atoms. On the other hand, the reversible changes in the $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ alloy is considered to be due to CSRO between Fe and Ni atoms. In this case the increase of the average number of Fe nearest neighbors around a Fe atom brings about the evolution of CSRO.

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