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Cooling condition dependence on ion etching rate in a softmagnetic metallic glass

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Taking into consideration the knock-on cascade mechanism and the free volume theory, the cooling condition dependent R_s (ion etching rate) is investigated for a liquid-quenched Fe–10at%Si–15at%B alloy glass. A high R_s is found in a slow cooled glass.

1. Introduction

Several attempts, such as powder metallurgy [1] and cold-working [2], have been made to form alloy glasses. Ion etching is able to work materials precisely [3–6]. It is also useful for working brittle glasses without fracture [7, 8]. Heat-treated Fe–Si–B alloy glass, which is brittle, often shows effective magnetic properties [9, 10]. It is used for magnetic components. Cooling conditions effect various properties in glasses [11–13]. Rate of ion etching is one of these properties, it is important in forming the glass precisely. The purpose of this study is to investigate the ion etching mechanism and describe R_s changes with the cooling glass using rate process.

2. Experimental procedure

2.1. Sample preparation

The Fe–10at%Si–15at%B alloy was prepared by melting commercially pure iron (99.9%), silicon (99.99%) and boron (99.5%) in an induction furnace under a protective argon atmosphere. Foil samples of the Fe–Si–B alloy were prepared by liquid-quenching with a twin-type piston-anvil apparatus under a protective Ar–5%H_2 atmosphere [9]. The samples were quenched from approximately 1700 K. The amount of sample melted in one run was about 0.2–0.8 g and the speed of the piston was about 0.12 m s^{-1}. Cooling rate (R) was varied by controlling the thickness (D) of the samples [13]. Thus D is used as a parameter to represent the cooling condition. The glassy structure of the sample was monitored by X-ray diffraction.

2.2. Ion etching system

The ion etching was carried out with an ion etching apparatus with a Kaufman-type ion source (ISM-S, Elionix, Tokyo). The ion etching system can be considered in three major sections: a plasma source which generates argon ions; three extraction electrodes (acceleration grids) which extract the argon ions from the plasma source and accelerate them towards the sample, and a water-cooled sample holder. The ion source has been operated with diameters of 70 mm for use in the ion etching apparatus.

2.3. Experimental condition

Ion beam energy and ion current density were 1.0 keV and 0.6 mAcm^{-2}, respectively. The ion etching was performed under a argon atmosphere with pressure of 2.1×10^{-2} Pa just above the sample. Since the sample holder was cooled by flowing water, the temperature at the sample surface was about 350 K during ion etching. The temperature was measured by a 0.1 mm diameter of CA thermocouple attached to the sample surface. The glass was not apparently relaxed (changed) at the temperature. Ion etching rate was measured with a surface roughness tester. The resolution was estimated to be less than 10 nm.

3. Experimental results

Fig. 1 shows the relationship between R_s and D in the Fe–Si–B alloy glass [7]. It is obvious that the increase

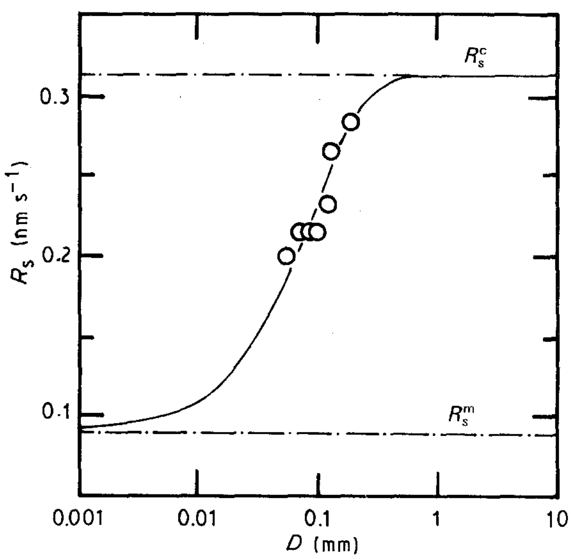

Figure 1 Change in the ion etching rate, R_s (nm s^{-1}) with sample thickness D (mm) in Fe–10at%Si–15at%B alloy glasses [5]. Solid line is explained by Equation 2. $R_s^c = 0.311$ and $R_s^m = 0.089$ nm s^{-1}.

in D, which corresponds to a decrease in the cooling rate, enhances the R_s. To describe R_s change with respect to cooling conditions, we suggest a following model.

4. Discussion

4.1. Ion etching model

Fig. 2a shows a schematic representation of an ion etching model. The behaviour of ions in a schematic crystal model has been deduced by P. Sigmund [14]. If the energy gained by the atoms is larger than bonding energy (E_b) of the atoms, the atoms knock-on. Thus knock-on cascades are formed. When the knock-on cascade arrives to the atoms on the sample surface, the surface atoms are ejected from the surface. We suggest a new model including defects as shown as Fig. 2b. Lattice defects in crystal are similar to free volume which are defects in glass [15, 16]. The faster the cooling rate, the higher the defects density becomes. If the defects decrease the kinetic energy (E_d) of knock-on cascade, a fast ion etching rate may be found at a large D value (a low cooling rate). With this model, it can explain that a fast cooling rate decreases R_s.

4.2. Rate process

A density of a vacancy depends on relaxation time [17]. The concept of the free volume is similar to that of the vacancy in the crystal. Thus, R_s is related to the free volume. Relaxation ratio (X) is mainly depended on the sample thickness (D) which is approximately inversely proportional to the cooling rate (R; K s^{-1}) [13]. X is expressed as follows [18–20]

$$X = \exp(-\mathrm{k}D^{\mathrm{n}}) \qquad (1)$$

Here, k and n are constant. X is assumed to express

$$X = (R_s^c - R_s)/(R_s^c - R_s^m) \qquad (2)$$

where R_s^c is the ion etching rate of a extremely slow cooled glass and R_s^m is the ion etching rate of a extremely fast cooled glass, respectively. R_s^c of Equation 2 is 0.311 nm s^{-1}, when the correlation coefficient (F) of Equation 1 is maximum ($F = 0.9394$) as shown in Fig. 3. If the cooling rate (R) is extremely high, that is, D (mm) is extremely small, R_s approaches the limited value (R_s^m) of the ion etching rate. Thus, R_s^m is 0.089 nm s^{-1}. R^s (nm s^{-1}) is given by the following equation for the Fe–10at%Si–15at%B alloy glass (see Fig. 4).

$$\log(0.311 - R_s) = -0.65 - 4.61\,D \qquad (3)$$

The linear plots confirm the assumption of Equation 1. On the basis of the relaxation process, the solid line of Fig. 1 is calculated. Thus, the desired R_s is obtained at the suitable cooling rate.

5. Conclusion

In summary, the cooling conditions dependence of the rate of ion etching (R_s) has been investigated for the

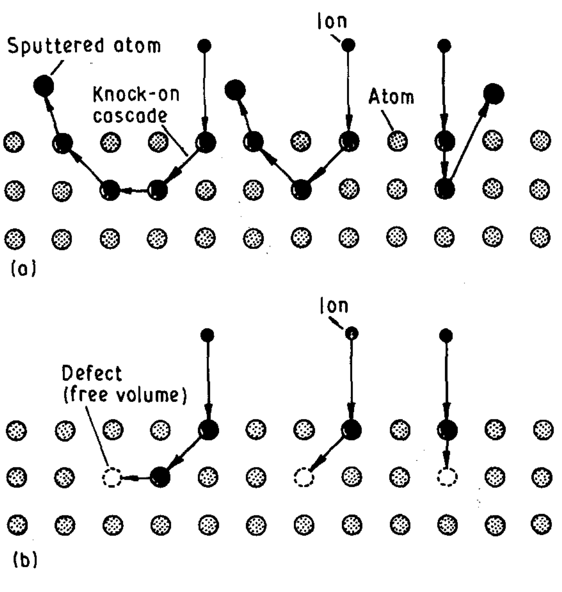

Figure 2 Schematic representation of the etching model: (a) shows schematic knock-on cascades model applied to defect-free sample; (b) is the present model applied to the sample including defects.

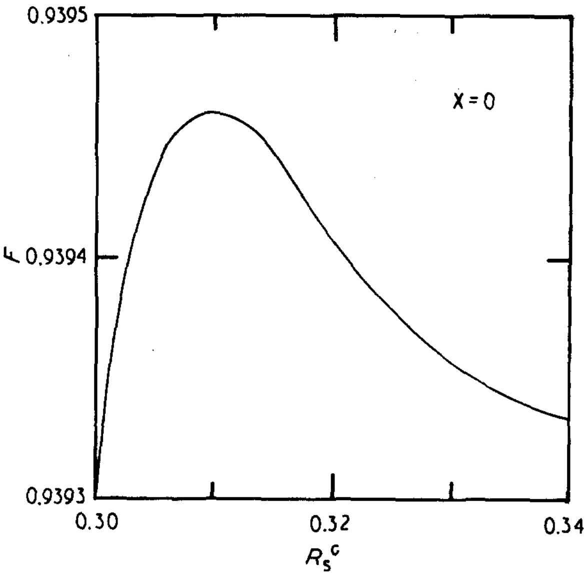

Figure 3 Change in correlation coefficient (F) with R_s^c: $X = 0$.

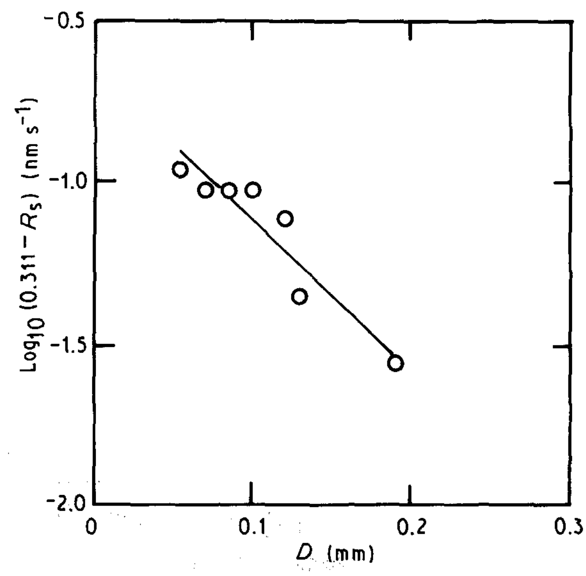

Figure 4 Linear plots between log (0.311 − R_s) and sample thickness (D) of Fe–10at%Si–15at%B alloy glass. D is inversely proportional to the cooling rate. Solid line is calculated by relaxation process.

Fe–10at%Si–15at%B alloy glass. The ion etching is able to work (shape form) the relaxed glass without fracture. The change of R_s is described by ion etching model. It shows defects decrease R_s. Since the high cooling rate increases defects density, it decreases R_s. Based on the relaxation theory, R_s is expressed by the relaxation process. Thus, the desired R_s is obtained at the suitable cooling rate.

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