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Original Paper

Anisotropy of Amorphous Metal Induced by High Temperature Stress Application

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Effect of uniaxial stress application to Fe-Si-B amorphous metal has been investigated below and above glass transition temperature. Uniaxial stress effect was characterized, as a magnetic permeability measurement, by using Magnetic Permeability Resonance (MPR) method. The values of horizontal and vertical permeabilities from stress applied direction were measured. Amorphous metal, which is heat treated at near glass transition temperature , showed significant anisotropy of permeabilities with stress application. This anisotropy is the result from atomic structural change, which is caused by viscosity flow under amorphous state.

Key Words: amorphous metal, anisotropy, magnetic permeability, uniaxial stress, plastic transition temperature

I. Introduction

Amorphous metal has been regarded as a typical isotropic material. Recent studies show ,however, some permanent anisotropic properties in amorphous metals. For example, heat treated samples under strong magnetic field[1] show magnetic anisotropy along magnetic field direction, or some magnetic thin films show perpendicular magnetization[2]. As a reversible phenomenon, magnetic anisotropy caused by elastic deformation was also observed and is applied for stress-strain gage[3]. Residual effect of uniaxial stress application, with irreversible atomic structural rearrangement like plastic deformation, will be one of mechanisms to induce an anisotropy. However, many studies have not been done in this field. It is known that above plastic deformation temperature (Tp), some amorphous metals show uniform plastic deformation and below Tp they show non-uniform plastic deformation and break with necking. This uniform plastic deformation is caused by

high viscosity flow, and some amorphous metals such as Pd-Si result in more than 100% uniform elongation. This phenomenon, which strongly depends on speed of strain and temperature, is similar to high elongation properties in conventional oxide glasses and super plastic materials. Amorphous metal is, generally, an unstable phase and is easy to crystallize above glass transition temperature (Tg). Since Tp is lower but near Tg, amorphous metal is unstable during uniform plastic deformation. To be prevented from crystallizing and to perform high uniform elongation of a sample above Tp, amorphous metal is required high glass forming ability. Our specimen, Fe-Si-B, has been studied intensively, since it shows high magnetic permeability for utilizing electric transformer to reduce core loss. Since it does not perform high glass forming ability, large elongation is not expected. On the other hand, since this ferro-amorphous metal has high magnetic permeability, which is sensitive to its atomic structure, anisotropic magnetic permeability change is expected, as a result of small uniaxial plastic deformation by viscosity flow. In this investigation, residual effect of uniaxial stress application in Fe-Si-B amorphous metal was characterized by Magnetic Permeability Resonance (MPR) method, which can measure arbitrarily directional permeability.

II. Experimental

The specimen, $Fe_{78.0}$-$Si_{9.0}$-$B_{13.0}$ (at%) amorphous ribbon (Metglass 2605S2 Nippon Amorphous Metals Co., Ltd.), is 50 μm thick, 10 mm wide and 50 mm long. From X-ray Powder Diffraction measurement, it was an amorphous state. Fig. 1 shows Differential Thermal Analysis (DTA) measurement from room temperature to 823 K under 1 atom Ar gas atmosphere. It shows glass transition temperature: Tg and crystallization temperature: Tx. Each sample was cut by ceramic scissors, and was hung by chucks, which was specially made for thin ribbon-formed sample. Fig.2 shows schematic figure of experimental arrangement for heat treatment and stress application. The Sample was hung vertically straight on metal wire, which is fixed on the upper roof, and loaded by brass weight. Total load, including the chucks and the weight, was 300g, - that means applied stress was 5.9 MPa. The Ribbon sample is so weak for shear stress that it tears with ease under far below true stress. To prevent from adding shear stress, load application for the straight direction was required. From this reason, we designed light and flexible chucks. The chucks were made of aluminum and were fixed by wires at the center of gravity of them. The sample and the load apparatus were sealed in a fused quartz tube. Pure Ar gas continually flowed into the tube under 1 atmospheric pressure. Stress applied sample was heated by an electric furnace from outside of the sealed tube, and was kept at constant temperature either 573, 673, 723, 773K for 3 hours. The sample was cooled within the furnace after heat treatment. For comparison, some samples were also heat

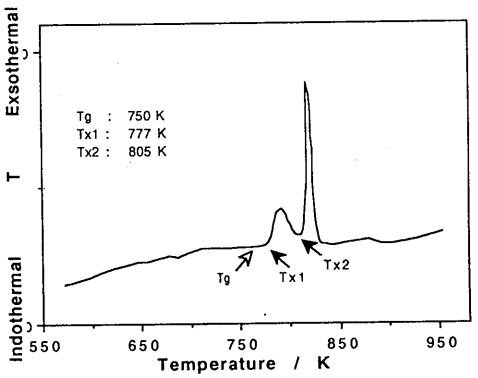

Fig.1 DTA data of Metglas 2605S-2

treated without any stress application at the same condition. All these samples were measured by X-ray Powder Diffraction analysis.

Magnetic permeabilities were measured by Magnetic Permeability Resonance (MPR) method, which was named by us. And same sample were measured by Vibrational Sample Magnetization (VSM) method. Fig. 3 shows schematic figure of MPR method. Two enamel wires were coiled with E-shaped ferrite core, which is 20mm wide, 10mm long, 5mm thick, as a detector. Drive coil is for the input of 1 kHz-100V Electric Source, and detect coil is for the output of excited voltage to be measured.

We call the former electric source Source Voltage: Vs, and the latter excited value Excited Voltage: Ve. The heat-treated ribbon sample was contacted with the open side of this E-shaped ferrite core in two different directions, parallel and perpendicular to the load direction. Each Voltage was measured by Digital Voltmeter and Oscilloscope.

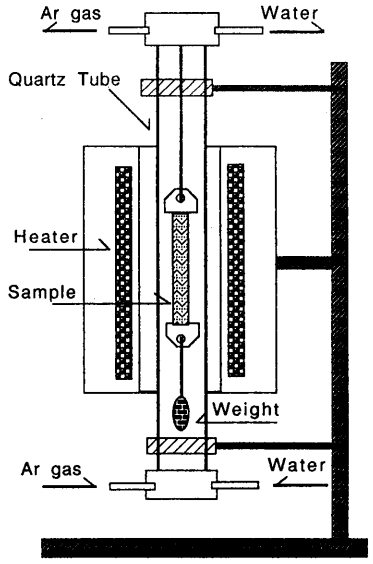

Fig.2 Ribbon Sample. Mechanical Heat Treatment Apparatus

To keep the gap between the ferrite core and the sample constant, same weight was put on this core. In VSM method, -250 to 250 Gauss magnetization was applied, and magnetic flux direction was set parallel to the thickness direction of the ribbon. Demagnetizing field was displaced from H-B curve, considering ribbon shape.

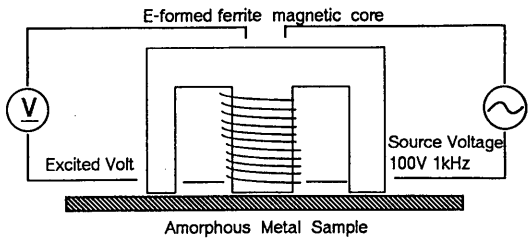

Fig. 3 Schematic Figure of Magnetic Permeability Resonance Method

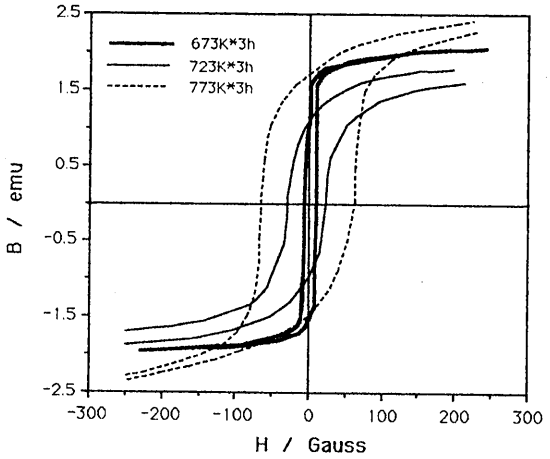

Fig.4 VSM Data of heat-treated Metglas 2605S-2

III. Results

DTA result show that the crystallization temperature was 777K, and the glass transition temperature is approximately 750K. Assume the glass transition temperature as a standard point, temperatures of the heat treatment were decided; (1) 573K and 673K and 723K as below the Tg, (2) 773K as above the Tg. Samples which were as-quenched and heat treated at 573K and 623K were amorphous phase by X-ray measurement. And samples which were heat treated at 673K and 723K were crystallized into α-Fe phase by the same measurement.

Fig.4 shows VSM results of each samples. Large increase of coercive magnetization and large decrease of magnetic permeability were observed respectively in the samples which were heat treated at 723K and 773K. Magnetic saturation was not observed. Fig.5 shows initial permeability from VSM measurement and Excited Voltage from MPR measurement. Both measurements show the same tendency - more than one fifth decreasing of initial permeability and Excited Voltage was observed in 723K and above heat treated samples. By VSM measurement, increase of magnetic permeability was observed in 523K heat treated sample , whereas in MPR measurement, values of Excited Voltage didn't show significant difference. Two different directional values of Excited Voltages were compared in Fig.6 to measure the influence of stress application. Only in the sample which was heat treated in 673K and was measured parallel to the applied stress direction, Excited Voltage of MPR method showed 5% increase. On the other hand, less than 2% decrease of Excited Voltage were observed in the other samples. All the permeabilities that are vertical tothe applied stress showed not much difference form that of the parallel direction.

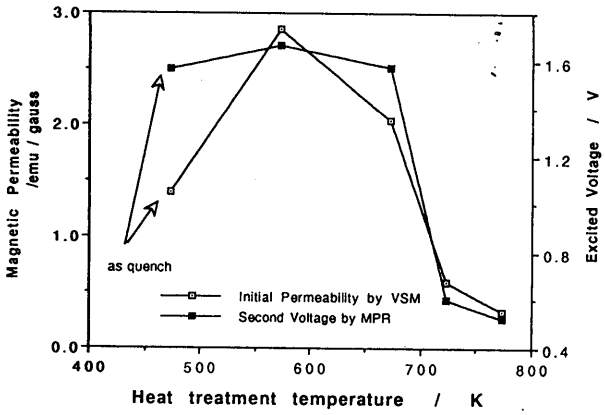

Fig.5 Initial Magnetic Permeability by VSM and Excited Voltage by MPR of Mechanical Heat Treated Samples

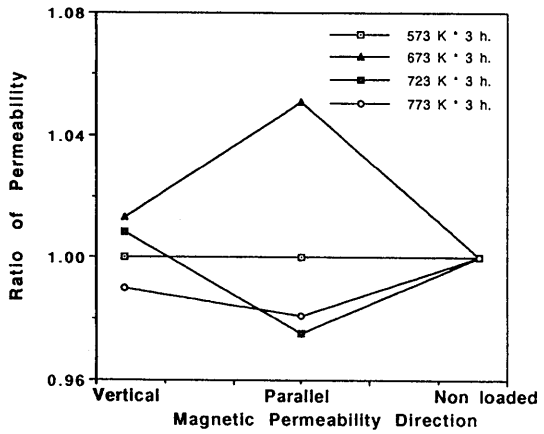

Fig. 6 Ratio of permeability between Vertical or Parallel to Stress Direction and Non-loaded samples

IV. Discussion

(a) Relation between Permeability and Excited Voltage

In MPR method, electromagnetic mutual induction was the major factor for the Excited Voltage. According to electromagnetic equation (1), magnetic flux dominates the electromagnetic interaction between two coils. Coefficient of L12 is called electromagnetic mutual inductance.

$$V_e = L_{12} \frac{dI_s}{dt} \cdots \quad (1)$$

L_{12} ··· electromagnetic mutual inductance
V_e ··· excited voltage
I_s ··· source current

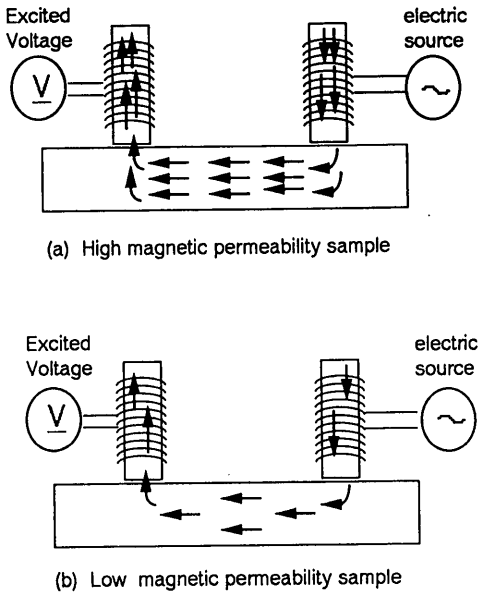

Fig. 7 Schematic Relation between Magnetic Permeability and Magnetic Flux Density for Voltage Excitation

Excited Voltage is proportional to magnetic flux density, and magnetic flux density is proportional to magnetic permeability. Therefore Excited Voltage is proportional to the magnetic permeability. In MPR method, magnetic circuit was composed of three parts; E-shaped ferrite core, contacted sample and the gap between the core and the sample. Since, in each measurement, condition of the ferrite core and the gap were kept constant, we think Excited Voltage was proportional to magnetic permeability of the sample. Magnetic field, which was caused by the first coil, was so small that this permeability is nearly equal to initial permeability. From these reasons, Excited Voltage of MPR measurement and initial permeability of VSM measurement, in Fig.5, showed same tendency. Since decision of initial permeability from tangent line at the origin of VSM curve contains certain error, and Excited Voltage involves non-linearity effect of permeability, some unavoidable difference between both methods existed.

(b) Crystallization and Permeability

Magnetic permeability was closely related with magnetic domain movement, which was induced from rotation of magnetization direction and movement of magnetic domain wall. Ease of these factors, magnetization rotation and magnetic domain wall movement, makes magnetic permeability high. Magnetic anisotropy, that involves crystal anisotropy and shape anisotropy, interferes magnetic domain

movement. High magnetic saturation assists increase of permeability. Relation among these factors is shown in equation (2)[4].

$$\mu_i \propto \frac{I_s^2}{\mu_0 K} \cdots \quad \text{Magnetization Rotation}$$

$$\mu_i \propto \frac{I_s}{\sqrt{K}} \cdots \quad \text{Magnetic Domain Wall Movement} \qquad (2)$$

μ_i ··· initial magnetic permeability
I_s ··· magnetic saturation
μ_0 ··· space permeability
K ··· magnetic anisotropy

Crystal magnetic anisotropy and shape magnetic anisotropy are instinct properties of crystals, but amorphous materials are free from these anisotropies. That is why, ferro-amorphous metal shows high magnetic permeability, comparing with crystalline materials.

In our investigation, X-ray Powder Diffraction measurement showed, after above 723K heat treatment, amorphous samples were crystallized into α-Fe. And in the same samples, Excited Voltage of MPR measurement and magnetic permeability of VSM measurement showed more than one fifth of decreasing. According to these results, we conclude that phase transition by crystallization increase magnetic anisotropy, and caused the large decrease of permeability and Excited Voltage.

In the heat treatment from 723K to 773K, according to X-ray measurement, further crystallization from amorphous phase to α-Fe was progressed. Further decrease of initial permeability and increase of coercive magnetization were explained in the crystallization of the sample. According to VSM measurement, in 573K heat treated sample, increase of initial permeability and magnetic saturation were observed. This increase is regarded as the result of structural relaxation of amorphous material during the heat treatment.

(c) Effect of Applied Stress and Anisotropy of Permeability

As shown in Fig.5, most significant anisotropy of permeability was observed in the sample heat treated at 673K, which was in amorphous state from X-ray measurement. Significant anisotropies were not observed in the other amorphous and crystallized samples. Result of anisotropic permeability implies an atomic anisotropic structural change in amorphous state. Since the anisotropy emerged at near the glass transition temperature, this structural change is induced from the uniform plastic deformation with viscosity flow of amorphous material. In some amorphous metals and conventional glasses, at near glass transition temperature and above plastic transition temperature, uniform plastic deformation has been observed with high viscosity flow. In this ferro-amorphous metal, since its glass forming ability is weak, plastic deformation with high viscosity flow did not undergo.

Ferro-amorphous metal, however, has high permeability and this permeability is sensitive to its atomic structure. Therefore, during stress application process above plastic transition temperature, small anisotropic atomic structural change occurs. And it can be detected as permeability difference.

As Schematically explained in Fig.7, during uniaxial viscosity flow process, atomic structure was rearranged and some iron atoms arrayed along the stress direction. The anisotropic rearrangement increases magnetic permeability. However, in perpendicular to the stress direction, this structural rearrangement is not sufficiently changed, and it doesn't affect permeability. During crystallization process, atomic diffusion dominates phase transition and stress application doesn't mainly affect the atomic structural rearrangement. From this reason, crystallized samples didn't show significant anisotropic magnetic permeabilities.

To clarify the relation between this uniaxial stress effect and atomic rearrangement precisely, further study such as atomic radial distribution measurement by EXAFS and more sensitive measurement such as differential magnetic permeability method[6] (Fig.8) are expected to analyze small induced anisotropy.

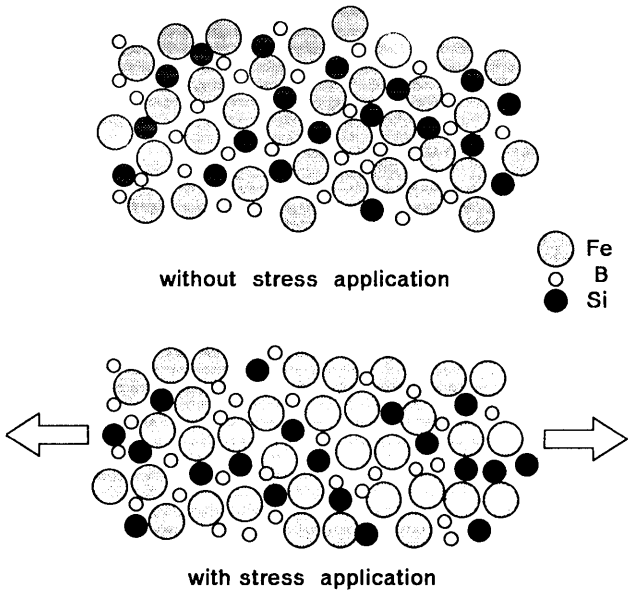

Fig. 8 Anisotropic Atomic Rearrangement with Uniaxial Stress Application

V Conclusion

$Fe_{78.0}$-$Si_{9.0}$-$B_{13.0}$ (at%) amorphous metal which is usually thought to be isotropic state of solid shows more than 5% anisotropic magnetic permeability under 5.9 MPa uniaxial stress application at 673K for 3 hours. The anisotropy was induced by atomic rearrangement accompanied with

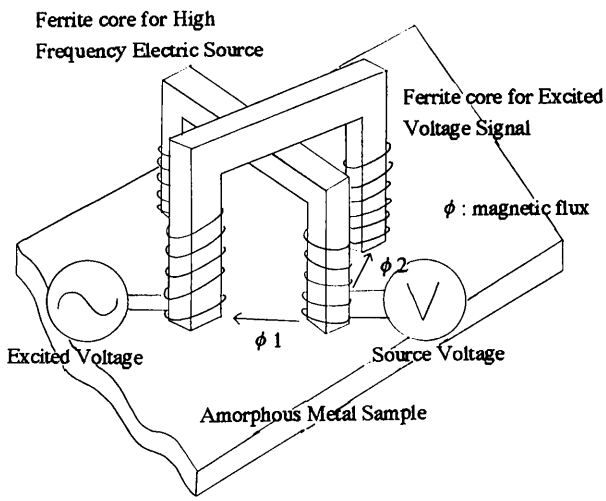

Fig. 9 Schematic Figure of Differential Magnetic Permeability Resonance Method

viscosity flow in amorphous solid. Other samples which was heat treated at 573, 723, 773K for 3 hours, did not show significant anisotropy. The amorphous which was heat treated at 723, 773K for 3 hours was crystallized into α-Fe and shows decrease of initial permeability and increase of coercive magnetization.

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