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QUENCHING EFFECTS IN A Fe₄₀Ni₄₀P₁₄B₆ AMORPHOUS ALLOY STUDIED BY MAGNETIC ANISOTROPY MEASUREMENTS

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Abstract.—Kinetics for the establishment of the magnetic anisotropy has been found to be a reversible function of the equilibrium structural state quenched in from different temperatures (activation energy: 0.28 ± 0.02 eV)

Introduction

Amorphous metallic alloys quenched from the melt are known to be in an out-of-equilibrium state and to show a structure change when annealed at temperatures for below the crystallization temperature, \( T_x \).

This relaxation affects most of the physical properties [1-6], and must be considered as due to variations of topological (TSRO) and/or chemical (CSRO) short range order [7]. Physical properties depend more or less on either of these orders. At least in some cases [8-10], it is obviously not a matter of crystallization first step, but rather an evolution of the amorphous phase towards a metastable equilibrium state. So, a fundamental question is asked about the possible reversibility of this state along with the anneal temperature. Such a reversibility at least for one part has been established for Curie temperature \( T_C \) [7, 11], electrical resistivity [12] and magnetic anisotropy [13-18]; the latter one varies also reversibly as a function of direction of the applied magnetic field.

These three properties seem to reflect mainly CSRO [7, 19].

Things are quite different in our recent work [20]: we have shown that, in a Fe₄₀Ni₃₉Mo₂B₁₈ alloy subjected to a thermomagnetic treatment at a temperature \( T_A \), the induced anisotropy establishment rate depends reversibly on the temperature \( T_Q \) from that the sample was quenched down. Now the CSRO establishment rate is related to the atomic mobility, which, in turn, can be assumed to be influenced by TSRO [7]. Then, at least a part of the TSRO-type structural relaxation of an amorphous alloy behaves as a reversible function of temperature. This paper is a first quantitative study of this phenomenon.

Experiments

We want to compare kinetics of uniaxial magnetic anisotropy development as a function of the quench temperature, \( T_Q \). Such kinetics are measured during an isothermal thermomagnetic treatment (TMT) at a fixed temperature, \( T_A = 160^\circ C \), after the samples were quenched down from different temperatures, \( T_Q \).

We have used the Fe₄₀Ni₄₀P₁₄B₆ alloy supplied by the Allied Chemical Corporation (Metglass 2826) in the shape of a 50 μm thick ribbon prepared by melt spinning. Samples are disks of 7 mm diameter, cut in the ribbon. They are first annealed 30 min at 360°C, say about 15°C below the crystallization temperature. Such a treatment results in an anneal of the internal stresses produced during manufacturing, and in a large structural relaxation, as reported elsewhere [8, 9]. Then, provided that the further annealing temperature is not higher than 350°C, all the observed phenomena are found reversible.

Magnetic anisotropy measurements, as well as TMT, are carried out in an automatic torque magnetometer. The magnetic field applied during anneals or measurements is 0.34 T. All the measurements are obtained at a reference temperature (30°C), with an accuracy of ± 0.3 °C.

Results

1 - Determination of the asymptotic value of the anisotropy energy

At a first time, it was necessary to check that the anisotropy energy asymptotic value, induced at 160°C, was not depending on the quenching temperature: the method has already been described [20]: it consists in performing successively on a sample:

a) a long TMT at the temperature \( T_Q \), followed by a quench; the direction of the applied magnetic field during the TMT is roughly at 45° from the reference direction;
b) a TMT at the temperature $T_A = 160°C$, with the magnetic field applied along the reference direction. As in [20], the magnetic anisotropy evolution is plotted, during this treatment, in a diagram [21] where the figurative point coordinates are the two first coefficients, $A_2$ and $B_2$, appearing in the Fourier series development of the anisotropy torque (Fig. 1).

For the Fe$_{50}$Ni$_{40}$P$_{14}$B$_6$ alloy the possible range $40 \leq T \leq 140°C$ of the $T$ temperatures is much narrower than for the Fe$_{40}$Ni$_{38}$Mo$_{4}$B$_{18}$ alloy [20], because the Curie temperature, $T_c$, is lower (290°C). Thus, if $T_Q > T_c$, the figurative point moves on the $0 A_2$ axis, and if $T < 260°C$, the times required for the TMT (a) are prohibitive: for instance, Fig. 1 shows that a TMT of 22 hours at 260°C is not long enough for the alloy reaching its equilibrium magnetic anisotropy. So we have only considered two values of $T_Q$: 260°C and 275°C; the induced anisotropy asymptotic value, obtained at 160°C, has been found the same $K_u = 440 \pm 10 \text{ J.m}^{-2}$. Therefore this value is not depending on the quenching temperature.

2 - Kinetics of magnetic anisotropy establishment

For this study, each successive experiment, carried out on a same sample, includes three steps:

a) an anneal of 30 min at 340°C, rubbing out the anisotropy induced by the foregoing experiment

b) an anneal of time $t_0$, at the temperature $T_Q$, followed by a quench, without magnetic field

c) a TMT at the temperature $T_A (160°)$, during which the establishment of magnetic anisotropy is measured; for the sake of symmetry, only the coefficient $A_2$ changes [20]. Its variations, as a function of time, can be reported in a reduced form:

$$c(t) = \frac{A_2(t) - A_2(0)}{K_u}$$

Fig. 2 shows, as examples, a few curves $c(t)$.

Fig. 1 - Evolution of the uniaxial magnetic anisotropy energy during an isothermal TMT at 160°C. Figurative point coordinates are the two first coefficients of the anisotropy torque Fourier series development.

Fig. 2 - Some kinetics of the induced magnetic anisotropy at a fixed temperature, $T_A = 160°C$. The initial state was obtained by a 2 hours anneal at $T_Q$, followed by a quench.

It appears clearly that, keeping all the other parameters the same, the kinetic is as faster as $T_Q$ is higher. But these curves do not deduce exactly from each other by translation: their difference decreases a little as the anneal at the temperature $T_A$ continues; that shows that the enhancement due to the quench disappears gradually; that agrees well with the qualitative results obtained in the Fe$_{40}$Ni$_{38}$Mo$_{4}$B$_{18}$ alloy, by means of isochronal TMT [20].

The Arrhenius plot of fig. 3 reports the time for a fixed value of $c$ (0.03 or 0.06), as a function of $1/T_Q$. Most of the experiments have been
performed with an annealing time, at the temperature $T_Q$, equal to 2 hours, and the temperatures $T_Q$

have been chosen in a dispersive order. In order to appreciate the effect of the time $t_o$, two experiments have been made with an annealing time equal to 18 hours, the first one with a high quenching temperature, $T_Q = 310^\circ C$, the other one with a low quenching temperature, $T_Q = 180^\circ C$.

Discussion

Fig. 2 and 3 bear out what had been qualitatively shown in our previous paper [20], say that the atomic mobility in an amorphous alloy contains a component, related to the alloy structure, the value of which is a reversible function of temperature. Fig. 3 shows that this term obeys approximately an Arrhenius law for temperatures above $250^\circ C$, and presents a saturation effect at lower temperatures. We think that such a saturation occurs because, at temperatures below $250^\circ C$, the annealing time ($t_o = 2$ h) used at the temperature $T_Q$ is not large enough for the alloy reaching a structural equilibrium. This hypothesis is reinforced by considering the two points obtained for $t_o = 18$ hours: a time constant increase is observed, weak for $T_Q = 310^\circ C$, and quite larger for $T_Q = 180^\circ C$, because in this latter case the approach towards equilibrium spreads over a larger scale of time. Then, for $T_Q > 250^\circ C$, the slopes of the straight lines can be thought close to the equilibrium values, but it appears that the kinetics of coming-back towards equilibrium, as a function of temperature, could be very interesting to study.

As in [20] the experimental curve $\sigma(t)$ can be shown giving a very wide time constant spectrum, and its strong curvature immediately at the origin prevents from using the 'initial slope, as it was made for crystalline alloys [22]. Then we have preferred to put on fig. 3 the values of $t$ corresponding to $\sigma(t) = C (C = 0.03$ and $0.06)$. The slope of the straight line can be seen decreasing weakly with $C$. The extrapolation to $C = 0$ gives $0.28 \pm 0.02$ eV. We will keep this value as being the activation energy, $E^f$, of the atomic mobility component, connected to the structure.

With regard to the total activation energy of the atomic mobility in this alloy, it has been found to be about $1.7$ eV [12, 18, 16]. But, by the present work, in the temperature range $140-240^\circ C$, the time for reaching the structural equilibrium is shown to be quite similar to the experiment time in [12, 18]. As a result, the value $1.7$ eV does not correspond to a complete structural equilibrium at each temperature; it is comprised between both values to be expected, the first one at constant structural state $E^m$, and the second one at equilibrium structural state $E^d$, so that $E^m < 1.7$ eV < $E^d$. Then taking in account the obvious relationship $E^m = E^d - E^f$, $E^m$ is concluded to be worth $1.4$ to $1.7$ eV.

Two models have been proposed to describe the atomic mobility in amorphous materials:

a) In the "distributed free volume" model, of Cohen and Turnbull [23], an atomic jump can occur when, because of thermal fluctuations, a free space appears near the considered atom, of a size larger than a critical value $v^*$. At constant volume, the
atomic mobility is shown to vary as $T^{1/2}$, that corresponds to a thermal activation of about 0.02 eV at 200°C, a value very less than $E^\circ$ as deduced here. But the model can be taken into consideration if it is assumed that the atom must jump over a potential barrier to move away [24].

b) Ramachandrarao et al [25-26] have suggested that the thermal agitation could create holes in amorphous alloys, as it does vacancies in crystals. Such holes well localized would have a size of a fraction of an atomic volume. Then $E^\circ$ and $E^\circ$ as defined above are respectively equal to the formation and migration energies of these holes. Using data on viscosity, or dilatation coefficient gap at the glass temperature transition $T_g$, the authors [25] have been able to give an estimation of $E^\circ$ for several alloys; $E^\circ$ was shown to vary linearly with $T_g$. For the Fe$_{40}$Ni$_{40}$P$_{14}$B$_6$ alloy, with a value of $T_g$ taken between 658 and 678 K [27,28], the Ramachandrarao relationship gives $E^\circ = 0.33$ to 0.34 eV, of the order of magnitude found in this work.

Then the experimental values for activation energy can be explained as well by the model of distributed free volume with potential barrier as by the localized holes one.

Conclusion

The study of the Fe$_{40}$Ni$_{40}$P$_{14}$B$_6$ amorphous alloy has enabled us to separate, in the atomic mobility, the component depending on the structural state. Its equilibrium value has been shown to vary reversibly as a function of temperature, with an activation energy of 0.28 ± 0.02 eV. Such a value can be well explained by the model of localized holes, but the model of distributed free volume cannot be ruled out.

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