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An improved capacitance method of measuring thermal expansion and magnetostriction of amorphous ribbons : application to FeNiCr metallic glasses

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Résumé. — On peut mesurer avec précision la magnétostriction et la dilatation thermique de rubans amorphes enroulés de façon à obtenir des cylindres creux rigides : les déformations sont mesurées selon l'axe du cylindre à l'aide d'un dilatomètre capacitif. Cette méthode, plus facile et fiable que celles qui ont été employées à ce jour, a été testée en étudiant les propriétés magnétoélastiques des alliages amorphes : $\text{Fe}_{40-x/2}\text{Ni}_{40-x/2}\text{Cr}_x\text{Mo}_2\text{B}_8\text{Si}_{10}$. Nous avons vérifié le caractère à un ion de la magnétostriction.

Abstract. — Magnetostriction and thermal expansion can be accurately measured on amorphous ribbons wound in order to get rigid hollow cylinders : the strains are measured along the cylinder axis by means of a capacitance dilatometer. This method, easier and more reliable than the conventional ones, has been tested by studying the magnetoelastic properties of amorphous $\text{Fe}_{40-x/2}\text{Ni}_{40-x/2}\text{Cr}_x\text{Mo}_2\text{B}_8\text{Si}_{10}$ alloys. The one-ion character of the magnetostriction has been verified.

1. The hollow cylinder method.

Various techniques have been developed these last ten years to measure the magnetostriction and the thermal expansion of amorphous alloys :

- the capacitance method gave good results when ten ribbons, each $30\text{ }\mu\text{m} \times 1.5\text{ mm} \times 3\text{ mm}$ were glued together in order to give a composite bulk sample [1]. But, there is a problem with the stiffness of the glue ; moreover, the magnetic field must rotate perfectly in the plane of the platelets in order to prevent the sample from tilting ;

- the strain gauge technique can be used provided that two or more ribbons are glued together in order to stiffen the sample [2, 3] ; the stiffness of the glue can alter the data in the case of thin ribbons ;

- other techniques are reviewed in [4].

Here we propose a new method for studying amorphous ribbons, which eliminates all the problems associated with the preparation of the samples and makes the experiment as easy as in the case of a bulk sample :

The as-prepared ribbon, e.g. 20 to 50 μm in thickness and 10 to 30 cm in length, is wound in order to give a hollow cylinder that is slipped into a copper ring which

prevents it from reeling off. The height of the cylinder is equal to the width of the ribbon, typically 2 to 10 mm, and its inner and outer diameters respectively $\approx 3.5\text{ mm}$ and 5 mm.

The stability and the rigidity of these samples are comparable with those of bulky cylinders : when cemented (loctite adhesive N° IS 495) onto the sample holder, they can no longer tilt under the influence of any mechanical or magnetic stress, which is a decisive advantage for measuring small forced magnetostriction. Last this is a non destructive method : due to the outstanding mechanical properties of the as-quenched metglasses, the ribbons are not deformed when extracted from the ring if they have not been annealed. The deformation of the samples is measured by means of a capacitance dilatometer [5] associated with coils and magnets working up to 10 kOe.

This method is suitable to accurately determine the thermal expansion of amorphous ribbons as we shall see in the next section. For magnetostriction experiments, the demagnetizing field cannot be derived easily due to the shape of the samples ; since magnetostriction is obtained by extrapolating the high magnetic field linear variation $\lambda(H)$ down to null internal field it

can be sometimes necessary to analyse the curves $\lambda(H)$ in connection with the magnetization curves $\sigma(H)$ as measured on the same samples when the forced magnetostriction is very large. In the present case, the values λ_s deduced from $\lambda(H)$ and from $\lambda(\sigma)$ are the same within $\pm 2\%$ as we shall discuss in section 3. For $x = 0$, the room temperature value of λ_s agrees with the values obtained by various methods for other $\text{Fe}_{40}\text{Ni}_{40}\text{M}_{20}$ glasses [4], where λ_s has been proved not to depend on the actual metalloid m .

2. The thermal expansion of some FeNiCr metallic glasses.

We have measured the thermal expansion of amorphous $\text{Fe}_{40-x/2}\text{Ni}_{40-x/2}\text{Cr}_x\text{Mo}_2\text{B}_8\text{Si}_{10}$ meltspun alloys prepared by Vacuumschmelze (Hanau). Their main magnetic properties have been already published [6]. We present in figure 1, the thermal expansion below room temperature of two alloys, namely $x = 0$ and 3.

Fig. 1. — Thermal expansion of two amorphous FeNiCr metallic glasses.

Our cooling rate was 1.5 degree per minute, and the thermal drift of the apparatus is equivalent to $(100 \pm 25) \text{ \AA}$ per degree, so the correction amounts to 8 % of α_T for the sample $x = 0$ (9.8 mm in length) and 40 % of α_T for the sample $x = 3$ (1.9 mm in length), at room temperature. The room temperature value $\alpha_T = 13 \times 10^{-6} \text{ K}^{-1}$ for $x = 0$ is somewhat larger than the value $10 \times 10^{-6} \text{ K}^{-1}$ found for the $\text{a-Fe}_{37.5}\text{Ni}_{37.5}\text{P}_{16}\text{B}_6\text{Al}_3$ alloy [7]. The difference might

arise from different quenching rates, as suggested by the work of Tyagi *et al.* [8] where the thermal expansion is shown to be very sensitive to any change in the amorphous state, or from the different metalloids entering in the alloys composition. In any case, these experiments have shown the feasibility of such measurements in a conventional dilatometer, and no buckling instability was observed contrary to the opinion given in [7] : the accuracy is good with the long samples (ribbon width 10 mm) while only qualitative information can be obtained with shorter samples due to the important correction ($100 \text{ \AA K}^{-1} \pm 10\%$) arising from thermal drifts in our dilatometer [5].

3. The magnetostriction of some FeNiCr metallic glasses.

We have studied the field and temperature dependence of the magnetostriction of amorphous $\text{Fe}_{40-x/2}\text{Ni}_{40-x/2}\text{Cr}_x\text{Mo}_2\text{B}_8\text{Si}_{10}$ with $x = 0, 1, 3, 4$ and 5 below room temperature.

As it is not easy to accurately determine the external magnetic field for which the internal field is null,

Fig. 2. — The magnetostriction of an amorphous FeNiCr alloy as a function of its magnetization : $\lambda_{\parallel}$ and $\lambda_{\perp}$ are measured with the magnetic field lying respectively along the measuring direction and in the plane perpendicular to this direction. σ is given in emu g^{-1} units. Full line : $\lambda_{\parallel} = (2 \sigma^2 + 90 \sigma) \times 10^{-9}$. Inset : the same magnetostriction data plotted against the applied magnetic field.

the determination of the spontaneous magnetostriction was first performed by plotting the magnetostrictive strains λ against the magnetization measured at the same temperature in the same condition, i.e. parallel to the cylinder axis for $\lambda_{\parallel}$ and then perpendicular for $\lambda_{\perp}$ (which was verified to be isotropic within $\pm 2\%$ of λ_s). Figure 2 gives an example of such a drawing : λ follows a regular curve during the magnetization process, e.g. $\lambda_{\parallel} = (2\sigma^2 + 90\sigma) \times 10^{-9}$ in the present case, and the slope changes drastically above saturation thus giving a good definition of $\lambda_{\parallel}$ and $\lambda_{\perp}$. We derive $\lambda_s = \frac{2}{3}(\lambda_{\parallel} - \lambda_{\perp})$. This is the same value as that derived by extrapolating the high field linear dependences of $\lambda_{\parallel}$ and $\lambda_{\perp}$ down to the value of the field indicated by a hatched line in the inset of figure 2, i.e. the value obtained by intersecting the two linear parts of the curve $\lambda_{\parallel}(H)$ respectively below and above the saturation : this definition holds true for samples $x = 3, 4$ and 5 which have the same dimensions. For samples $x = 0$ and 1 , the saturation was achieved in smaller fields along the axis than perpendicular to this axis.

The thermal variations of λ_s are given for the five samples in figure 3, and fitted by an $\hat{I}_{5/2} |\mathcal{L}^{-1}(m)|$ law, the well-known one-ion law established by Callen [9] where m is the reduced magnetization, $\mathcal{L}$ the Langevin function and $\hat{I}_{5/2}$ the modified hyperbolic function of degree 2. The only problem was

Fig. 3. — The thermal variations of λ_s through the amorphous FeNiCr series. Full circles : experiment. Full line : the one-ion model prediction. Open circles : the second and third sets of experimental data with the sample $x = 5$ (see text).

encountered with the sample $x = 5$: the first three closed circles (300, 250 and 200 K) seemed correct, but a shift of the curve was observed at lower temperatures, where too high values were observed. A new thermal variation performed after having unglued, cleaned and glued again the sample confirmed the new behaviour with a good accuracy : the thermal variation (open circles) is somewhat more pronounced than predicted by the one-ion model. This unexplained behaviour was observed only with the $x = 5$ sample which was cemented with « Saureisen 29 » cement and cleaned with NH_4OH , while the other four samples were glued with loctite and cleaned with acetone.

The magnetostriction decreases linearly with increasing chromium content, x , as shown in figure 4, a behaviour typical of the one-ion model of magnetostriction. It may be noticed here that, within the experimental uncertainties, the x dependence could as well be consistent with the σ_s^2 dependence of the magnetostriction established by a number of different authors at room temperature, but it deviates markedly from this behaviour at 4.2 K.

Fig. 4. — The Cr concentration dependence of the magnetostriction. Full line : the one-ion model. Hatched line : the σ_s^2 dependence of the magnetostriction. Open circles : the latter sets of experimental data for $x = 5$ (see text).

The forced magnetostriction was measured above technical saturation, and found to be $\sim 2 \times 10^{-10} \text{ Oe}^{-1}$ at liquid helium for all samples, and nearly tempe-

perature independent up to 300 K, except for the sample $x = 5$ due to the approach of the Curie temperature, $T_c = 425$ K after [6]. We give in figure 5 the thermal variation of $\partial\lambda/\partial H$ for this latter sample, and observe

Fig. 5. — The thermal variation of the forced magnetostriction of an amorphous FeNiCr alloy.

a λ -like curve which peaks at 395 K which is somewhat lower than the Curie point derived from magnetization data [6]. This is perhaps an indication that the structure was not the same during the last magnetostriction measurements as during the first one, a fact which could explain the anomalies observed in the thermal variation of λ_s (a faster decrease with temperature is consistent with a lower T_c).

Above 416 K, the magnetostriction was perfectly isotropic, i.e. $\lambda_s \equiv 0$, and the quadratic law was verified up to 10 kOe at 440 K ($\partial\lambda/\partial H$ was taken for an applied field of 10 kOe above T_c , in figure 4).

As a conclusion, the hollow cylinder method provides reliable data on magnetostriction and thermal expansion of amorphous ribbons. It is especially recommended when non-destructive tests are needed. This straightforward method has been checked by testing the one-ion character of the magnetostriction of a series of amorphous FeNiCr metallic glasses.

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