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INTERNAL FRICTION IN A NICKEL BASE METALLIC GLASS

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Résumé - Les auteurs ont étudié le frottement interne et le module de torsion en fonction de température de l’alliage amorphe à base de nickel contenant Cr 6.6, Fe 2.65, Si 7.8, B 14, C 0.25 at.%. Sur les courbes FI on a trouvé deux maximums élevés et un plateau distinct; sur celles de G/T/ on a observé quelques extrénums. Ces phénomènes sont liés, entre autres, à la cristallisation du nickel et à la précipitation de certains composés intermétalliques.

Abstract - Studies were made of internal friction /IF/ and shear modulus as functions of temperature for a metallic glass on nickel matrix containing Cr 6.6, Fe 2.65, Si 7.8, B 14, C 0.25 at.%. On the IF curves were found two high maxima and a distinct plateau while on the G/T/ curves several extrema were observed. These effects are associated, inter alia, with the crystallization of a solid solution in nickel and the precipitation of certain intermetallic compounds.

I - INTRODUCTION

Interesting results have been obtained using the internal friction /IF/ method for the investigation of the thermal stability of glassy alloys. In the temperature range of thermal non-stability two structural effects are observed [1]: 1/crystallization, 2/structural relaxation, taking place at considerably lower temperatures. The first drastically changes the alloy properties while the second, though having no significant influence on the amorphous nature of the sample, has a marked effect on the characteristic physical and mechanical properties of the metallic glass. Both these effects may be observed by the IF method, the first particularly clearly.

It is normally taken /e.g.[1,2]/ that crystallization involves crystals nucleation and growth, while to attain the stable crystal state the sample may pass through certain metastable states. IF investigations of crystallization of amorphous alloys have been reported in [3-6] while a peak associated with crystallization was also observed in [7]. In the present paper the IF method was applied to study the crystallization process in a multicomponent glass on nickel matrix. This glass is used in engineering as a solder in hard soldering.
Tests were conducted on metallic glass in the form of ribbons of dimensions 50x5x0.04 mm$^3$ produced by the standard melt-spinning method [8]. In atomic percentages the alloy contained: Cr 6.6, Fe 2.65, Si 7.8, B 14, C 0.25 the remainder being nickel. Internal friction was determined using an inverted type KG pendulum as described in [9]. Measurements were made in vacuum $10^{-4}$ torr in the temperature range from 300-1000 K. Linear heating was applied at a rate of 2 K/min. The measurement frequency of the pendulum was about 0.5 Hz.

Due to very small thickness of the ribbon the vibrating system was liable to lateral vibrations causing perturbation in IF measurements. In order to eliminate these unwanted vibrations a slight external stress of about 10 MPa/1 kg/mm$^2$/ was applied to the sample. Measurement of internal friction at various values of applied external stress showed that even doubling this stress has no effect on the form of the $Q^{-1}/T$/ and $f^2/T$/ curves.

Measurements performed at room temperature indicated that $Q^{-1}$ is only slightly dependent on vibrations amplitude as compared with the magnitude of variations observable on the $Q^{-1}/T$/ curve /Fig.1 and e.g.Fig2/. For this reason the vibrations amplitude was taken to be the mean of extreme values and this value was constant throughout all the measurements made. Vibrations amplitude calculated according to the Saint-Venant theory, assuming free torsional deflection of a narrow rectangular section bar, was equal to $3\times10^{-5}$.

In the formula for shear modulus /equation (1) in [3]/ even at peak temperature the $/Q^{-1}/^2$ is less than 0.01 /e.g. Fig.2/ and thus very small in comparison with 4, therefore the form of the $f^2/T$/ curves was taken to represent adequately the modulus versus temperature relation $G/T$/.

In the temperature range for which IF measurements were made the tested alloy is not ferromagnetic and hence there cannot occur changes in the form of the $Q^{-1}/T$/ and $f^2/T$/ curves as observed for ferromagnetic metallic glasses [3].

As well as IF measurements, using a type TA-1 thermoanalyser from the firm Mettler the DTA curve was determined for an as-quenched sample applying a heating rate of 2 K/min.

The $Q^{-1}/T$/ and $f^2/T$/ curves were determined for more than a dozen samples, in the as-quenched state and after in situ heating at temperatures 680, 733, 820, 970 K for 0.5 hours. X-ray diffraction patterns were made and the Miller indices determined for all samples. In the
as-quenched state the samples were not crystalline.

III - RESULTS

On the curve of internal friction versus temperature determined for as-quenched samples two sharp maxima may be distinguished, occurring at temperatures $A'\approx 729^\circ K$ and $B'\approx 770^\circ K$, and a plateau $C$ in the interval 850-890 K /Fig.2/. On the shear modulus curve these maxima correspond to the minima $A'$, $B'$ occurring at the same temperatures, while the plateau $C$ is associated with an abrupt drop in modulus $C'$.

Fig.1. Relation $Q^{-1}$ versus amplitude; sample in as-quenched state, room temperature.

Fig.2. Curves $Q^{-1}/T$ and $f^2/T$ for an as-quenched sample /$f=0.54$ Hz at peak $A'$/.

Fig.3. DTA curve for an as-quenched sample; heating rate 2K/min, Cu standard.

Fig.4. $Q^{-1}/T$ curves for as-quenched sample determined for various frequencies /$f$ at peak $A'$/.
In the as-quenched state curves $G/T$ exhibit certain additional extrema at temperatures lower than that of peak A, i.e., a minimum at about 580 K and a maximum at about 670 K. Fig. 3 shows the DTA curve obtained for a sample in the as-quenched state. A sharp exothermal peak may be clearly observed at 753 K and an inflexion at about 835 K.

Measurements of $Q^{-1}/T$ curves made for three vibration frequencies showed that the peaks do not shift with $f$ /Fig. 4/, however a marked fall in peak heights with increasing measurement frequency may be observed.

The effect of heating in the temperature range 680-970 K on the form of curves $Q^{-1}/T$ and $f^2/T$ is presented on Fig. 5. Heating the samples at 680 K, i.e., at a temperature lower than that of maximum A, does not affect temperature of occurrence and height of peaks A and B, but the low temperature side of maximum A is shifted towards higher temperatures so that the whole peak is narrowed. After heating at 733 K, i.e., only 4 K above the temperature of peak A, peak A is seen to disappear while peak B remains unchanged. Heating at 820 K, i.e., above the temperature of peak B, causes both peaks A and B to disappear. However, the plateau persists and traces are still found even after heating at 970 K /0.5 hours/. The plateau occurs against a sharp rising background and

Fig. 5. $Q^{-1}/T$ curves and normalized $f^2/f_o^2$ for samples in as-quenched state-1, and after heating at temperatures: 2-680K/0.5h, 3-733K/0.5h, 4-820K /0.5h, 5-970K/0.5h; $f_o$-frequency at room temperature.

Fig. 6. Height of peak A plotted against $f^{-1/2}$. 
above the plateau temperature the form of the $Q^{-1}/T$ curve is virtually identical for all samples. Considering the curve $G/T$, after heating at 680 K the extrema at 580 K and 670 K disappear, after 733 K the minimum A' also disappears and after 820 K minimum B' disappears as well, there remains only the drop in modulus $C'$, associated with the plateau C. Heating at 970 K smooths out the $f^2/T$ curve completely, although the plateau on $Q^{-1}/T$ remains.

IV - DISCUSSION

From the tests conducted it was ascertained that on the $Q^{-1}/T$ curves there occur two high peaks and also a small plateau /Fig. 2, 4, 5/, which are accompanied on the $G/T$ curve by distinct minima. For as-quenched samples two additional extrema are found on the $G/T$ curve, i.e. a minimum at 580 K and a maximum at 670 K. The temperatures of the peaks and the plateau are very stable and show no change even after heating which causes the disappearance of certain of these extrema /Fig. 5/. The peak heights also remain constant for the same vibrations frequency. Peaks A and B correspond to the exothermal maximum on the DTA curve, while the plateau appears to be associated with a small inflexion occurring at about 835 K. It is noteworthy that the temperature of the beginning of the maximum is in good agreement with the temperature of peak A, i.e. 729 K.

Similar curves of $Q^{-1}/T$ and $G/T$ were reported in [3-7] for other glassy alloys, two phase change peaks being found to occur for Fe$_{80}$P$_{13}$C$_7$, Fe$_{50}$Ni$_{33}$P$_{12}$C$_5$ [3], Fe$_{83}$P$_{17}$ [4], Pd$_{80}$Si$_{20}$ [5].

Studies on the influence of pendulum vibrations frequency indicated that the temperatures of occurrence of the peaks and the plateau are independent of f /in the range 0.27-0.63 Hz/ while the heights of peaks A and B decrease with increasing f. The first characteristic confirms definitively that the observed maxima and the plateau are not associated with migrational relaxation processes but with phase changes.

On Fig. 6 is presented the relation showing heights of A - peaks $Q_{\text{max}}^{-1} - Q_{b}^{-1}$ versus $1/\sqrt{f}$; these values were found by deducting the processes B and C and the high-temperature background which remained after heating at 733 K, from the $Q^{-1}/T$ curve /Fig. 4/. The curve obtained is approximately a straight line passing through the origin of the co-ordination system. The same result was reported in [6] for phase transformation peaks in a Fe$_{0.43}$Ni$_{0.47}$B$_{0.06}$P$_{0.04}$ alloy at acoustic frequencies. These results are in agreement with the Krivoglaz IF theory of phase changes of the first-kind [8]. An approximate relation of peak height versus vibrations frequency is given in [5].
Heating in the temperature range 680-970 K has a very marked effect on the form of curves $Q^{-1}/T$ and $G/T$. By careful choice of heating temperature peak A or peaks A and B can be removed, and also quite probably A,B,C. X-ray observations showed that after heating at 740 K reflexes originating from a crystalline solid solution in nickel appear, and above 790 K peaks associated with certain intermetallic compounds are additionally observed. Applying the high temperature X-ray structure analysis these results were confirmed and it was shown that the maximum B is associated with precipitation of $\text{Ni}_2\text{B}$ and probably also $\text{Ni}_3\text{Si}_2$, while the plateau C is associated with the precipitation of $\text{Fe}_{4.5}\text{Ni}_{18.5}\text{B}_6$ and probably also $\text{Fe}_3\text{SiB}$ [11].

The influence on the $Q^{-1}/T$ and $G/T$ relations of heating or of successive measurements to ever higher temperatures was also studied in [3-5]. In the main the same results were obtained.

It is noteworthy that the extrema occurring on the $G/T$ curve below the temperature of peak A which disappear after heating at 680 K, have recently been reported to occur also on curves determined for as-quenched samples of alloy $\text{Co}_{75}\text{Si}_{10}\text{B}_{15}$ [12]. A similar effect was observed for certain $G/T$ curves determined by Soshiroda et al. [3].

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