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STRUCTURAL RELAXATION AND CRYSTALLIZATION OF FeB AND NiP METALLIC GLASSES FOLLOWED BY INTERNAL FRICTION AND MODULUS MEASUREMENTS

S. Kiss, G. Posgay, I.Z. Harangozó and F.J. Kedves

Institute for Applied Physics, Kossuth L. University, H-4010 Debrecen, Hungary

Abstract.- The dynamic elastic modulus and internal friction of metallic glasses was measured by a torsional pendulum with low inertial momentum and low external damping.

The internal friction measured during heating increases rapidly above 100°C, and near the crystallization temperature it decreases suddenly. At the same time a modulus jump in one or two steps was found during crystallization in a narrow temperature range. Isothermal measurements in the crystallization range show that an incubation time is needed and the velocity of this process depends on temperature as well. A large continuous increase in the dynamic elastic modulus was observed far below the crystallization temperature in a wide temperature range above 100°C. This is probably caused by a structural relaxation process in the amorphous state. No relaxation peak above room temperature was found neither in glassy nor in crystallized state.

1. Experiment.— A new internal friction /i.f./ apparatus with low inertia momentum was constructed in order to measure the thin metallic glass samples having internal stresses too in the frequency range of torsional vibrations of 0.1 - 1 Hz. The torsional pendulum is built into vacuum vessel under pressure of 10^-2 Pa, and the temperatures can be varied by means of an internal furnace up to 600°C. To avoid the electrodynamic attenuation of the induction detector the amplitude of vibration was indicated by a Sfram type light following galvanometer using the reflected laser light from the mirror mounted onto the pendulum.

The NiP samples of 19.4 at%P content were produced by an electroless deposition technique and the specimens were cut from a 40/μm thick sheet. The FeB samples of 17 and 20 at%B content were made by melt quenching technique. The specimens were about 30/μm thick, 1 mm wide and about 30-50 mm long.

2. Results and discussion.— The i.f. and dynamic modulus /d.m./ spectra and the kinetic behaviour had a couple of similar features
for both types of metallic glasses /Fig. 1-3/. The internal attenuation of the materials in amorphous condition at room temperature /RT/ and at higher temperatures is considerably higher than in crystalline state. The i.f. of FeB materials at RT was always between 40 and $90 \times 10^{-4}$. This value decreased applying axial mechanical stress which can be attributed to an ordering caused by the stress [1]. Concerning amorphous and crystalline state this is in agreement with the statement that the i.f. of more disordered structure is higher.
In both types of materials the i.f. measured at heating rate of about 2°C/min starts to increase approximately exponentially at relatively low temperatures (at about 100°C) and d.m. increases differently. Differences in the behaviour of various samples begin only 100°C below the crystallization temperature /$T_x$/ but they have similar behaviours too since in this range the rapid increase of i.f. ceases and the d.m. has also decreasing tendency. At $T_x$ in all cases the d.m. jumps up abruptly and the i.f. decreases significantly. At higher temperatures the common behaviour was found: the d.m. decreases the i.f. increases approximately exponentially. The i.f. and d.m. spectra in crystalline state are smooth, the i.f. runs always below and the d.m. above the curves of the amorphous condition. The d.m. of crystalline state at RT is at least 30-40% higher than that of amorphous but one can find much higher differences too.

The strong increase in d.m. and decrease in i.f. caused by crystallization appear in the FeB metallic glasses for both composition. The crystallization of Fe$_{80}$B$_{20}$ takes place in one step between 400 and 420°C for a given /2°C/min/ heating rate. The Fe$_{83}$B$_{17}$ samples have two crystallization steps between 380 and 440°C. The amount of increase in d.m. is 15% in the first step up to about 400°C and further 40% in the second one. The i.f. decreases in one or two steps from 600 x $10^{-4}$ to about 300 x $10^{-4}$. It is known that there is a two step crystallization in FeB hypoeutectic alloys, when at first α-Fe then Fe$_3$B precipitates. In case of higher B content one can expect the precipitation of the two crystalline phase in a single step. The composition of Fe$_{83}$B$_{17}$ lies near to the eutectic one so with respect to $T_x$ too our results are in good agreement with previous data obtained by means of different methods [2,3,4].

The NiP crystallizes in one step between 300 and 310°C measured using 2°C/min heating rate. The behaviour of the d.m. and i.f. is similar to that of FeB. Above $T_x$ the d.m. decreased further characteristic to more stable structures and a further increase in the d.m. was found at about 400°C. This can be attributed to the transformation of the Ni$_7$P$_3$+Ni crystalline phase formed earlier to the Ni$_3$P+Ni crystalline phase [5].

In order to find out the time parameters of the crystallization isothermal measurements were also made. It was observed already 30 - 40°C below $T_x$ determined using relatively high heating rates that a d.m. increase and i.f. decrease takes place within several hours caused by crystallization. The temperature dependence of the rate of crystallization can be seen in Fig. 4. The crystallization was almost
completed within time intervals indicated in this figure. This is confirmed by the fact that further heat treatments at higher temperatures caused only several per cent increases in the d.m. The amount of d.m. increase /about 35%/ agrees well with those found during continuous heating and the time needed for crystallization is also the same as was observed using resistance measurements [6].

The rate of crystallization increases on the curve belonging to 365°C above 45 min from the beginning /Fig. 4/. The same phenomena can be seen more expressed in the case of NiP samples where this change /Fig. 5b/ occurred after 1 hr at 280°C and after 2 hrs at 274°C.

This shows that before crystallization a certain time of incubation is needed which is already known from calorimetric measurements [7]. The i.f. in both types of materials decreased at the beginning of crystallization quickly enough. After the incubation time elapsed the i.f. increased slightly or the rate of decrease diminished what can be interpreted by the fact that the process of crystallization itself gives a contribution to the dissipation.

It has to draw attention to the fact that d.m. increases can be found for both types of materials below \(T_x\). This increase could be
different and rarely significant for different samples. In order to investigate this behaviour cycles of heating up, holding for one hour and cooling down were made with increasing temperatures. The results gained on NiP are summarized in Fig. 6. It can be seen that a holding for one hour at 50°C causes an increase in d.m. and decrease in i.f. and this effect is more and more significant at temperatures 75, 120 and 200°C. The results can be interpreted by assuming ordering or structural relaxation which can take place far below $T_x$ and
can cause d.m. increase and i.f. decrease. During intervals of constant temperatures the d.m. increased approximating the equilibrium value and the i.f. decreased exponentially /Fig. 5a/. It can be seen in Fig. 6, that the d.m. increased 75% from the RT value up to starting the crystallization at 280°C and during this process further 55% increase was observed. We can conclude inspecting the i.f. and d.m. curves of Fig. 6, that no relaxation peak occurred within the temperature range investigated for the studied materials in the amorphous and different crystalline state.

References