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L. GRÁNÁSY
A. LOVAS
T. KEMÉNY

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L. Gránásy, A. Lovas, T. Kemény

Central Research Institute for Physics
H-1525 Budapest 114, P.O.B. 49, Hungary

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АННОТАЦИЯ

Исследовалось влияние изменяющихся условий получения на термические свойства металлических стекол $Fe_{83,4}B_{16,6}$ при помощи дифференциальной сканирующей калориметрии /DSC/. Ленты были получены методом быстрого охлаждения /melt spinning/ с изменением температуры расплава /T_M/ и скорости вращения цилиндра /v/, причем все остальные условия были постоянными. Отличие аморфных состояний образцов, полученных при различных условиях, наблюдалось через процесс кристаллизации, при котором измерялось выделяющееся тепло, начальная температура и кинетика перехода.

KIVONAT

A változó elõállitási körülmények termikus tulajdonságokra gyakorolt hatását vizsgáltuk $Fe_{83,4}B_{16,6}$ üvegfémeken differenciális pásztázó kalorimetria /DSC/ segitségével. A szalagokat "melt spinning" módszerrel állitottuk elõ az olvadékhõmérsékletet /T_m/ és a hengerfordulatszámot /v/ változtatva, miközben a többi elõállitási körülmény azonos volt. A különbözõ körülmények közt elõállitott minták üvegállapotai közti eltéréseket a kristályosodási folyamaton keresztül követtük, mérve a felszabaduló hõt, a kristályosodás kezdõ hõmérsékletét és átalakuláskinetikáját.

THE INFLUENCE OF THERMAL HISTORY ON THE PHYSICAL PROPERTIES OF Fe-B METALLIC GLASSES

L.Gránásy, A.Lovas, T.Kemény

Central Research Institute for Physics, Budapest,Hungary

ABSTRACT

The influence of casting conditions on the thermal properties of $Fe_{83.4}B_{16.4}$ metallic glasses has been investigated by differential scanning calorimetry /DSC/. The ribbons were prepared by melt spinning varying the melt temperature /T_m/ and the rotating speed /v/ of the roller, while other casting parameters were fixed. Differences in the glassy state of the samples produced under different circumstances were followed through the changes in their crystallization process, measuring its heat release, starting temperature and kinetics.

EXPERIMENTAL

a/ Sample preparation

The samples were produced by Liebermann-Graham technique [1], using a copper roller of 7.5 cm diameter. The casting temperature was measured by an optical pyrometer calibrated by the melting point of pure metals. The accuracy and reproducibility of melt temperature is about 20 K. Glassy state of the samples was verified by X-ray measurements.

b/ Calorimetry

A Perkin-Elmer DSC-2 calorimeter was used for the investigation of thermal properties. The kinetic parameters /thermal activation energy, E_a; frequency factor, k_o; Avrami-exponent, n/ were obtained from dynamic measurements made at different heating rates using Arrhenius-type thermal activation

$$k/T/ = k_o \exp /- \frac{E_a}{RT} / \qquad /1/$$

and Johnson-Mehl-Avrami type rate equation

$$\frac{dC}{dt} = k(T)(1 - C) [- \ln (1 - C)]^{\frac{n-1}{n}} \qquad /2/$$

where C is the crystalline fraction. Activation energy was determined with the method of Ozawa [2], while k_o and n by fitting Eq.2. to the measured dC/dt, C and T values [3].

RESULTS

Samples were produced both, at a fixed rotating speed /v = 6200 r.p.m. = "5" in the units of the figures/ varying the melt temperature and with fixed melt temperature /T_m=1300 C/ at different v values /which vary the quenching rate through the thickness, see *Fig. 1.*/

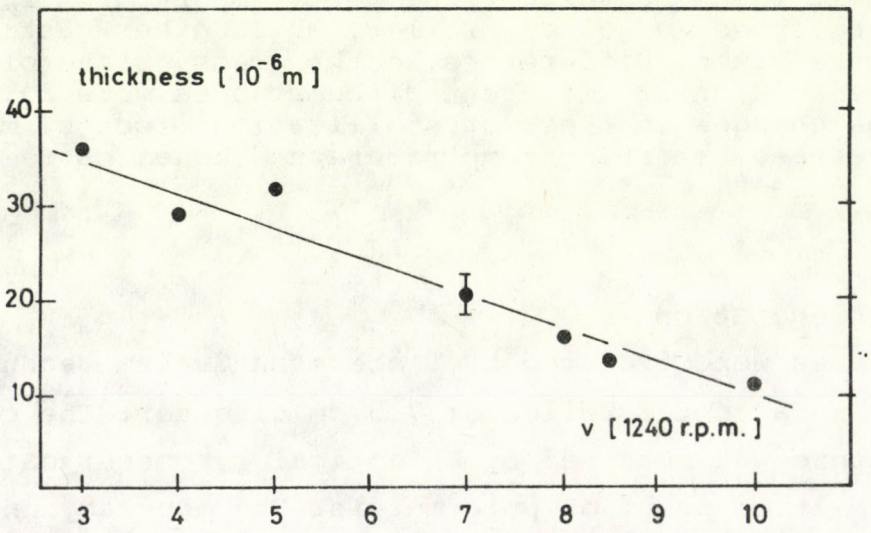

Fig.1. The thickness of the samples vs. rotating speed

Variation of the melt temperature in the investigated range does not yield any observable changes in the measured parameters i.e. the energy and the temperature of the crystallization and the DSC curves themselves are identical within the experimental error.

There are changes in the investigated properties, however, due to the different rotating speeds. The crystallization energy is independent of the rotating speed */see Fig.2./* which is in good accordance with the X-ray measurements, showing that the following differences are not due to the partial crystallization.

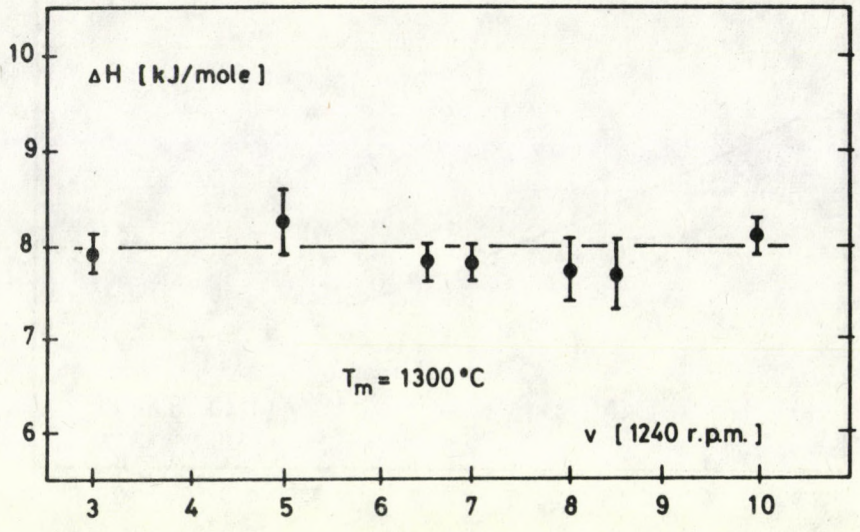

Fig. 2. Crystallization energy vs. rotating speed at fixed melt temperature

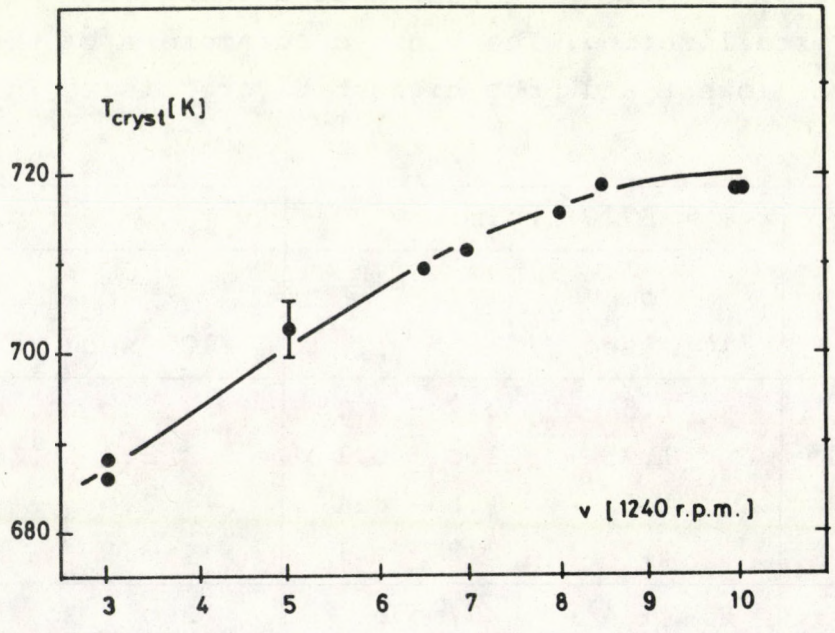

Fig. 3. Crystallization temperature vs. rotating speed

The starting temperature of crystallization determined from 20 K/min. DSC measurements increases */Fig. 3/* and the half-width of the crystallization peak decreases */Fig. 4/* with

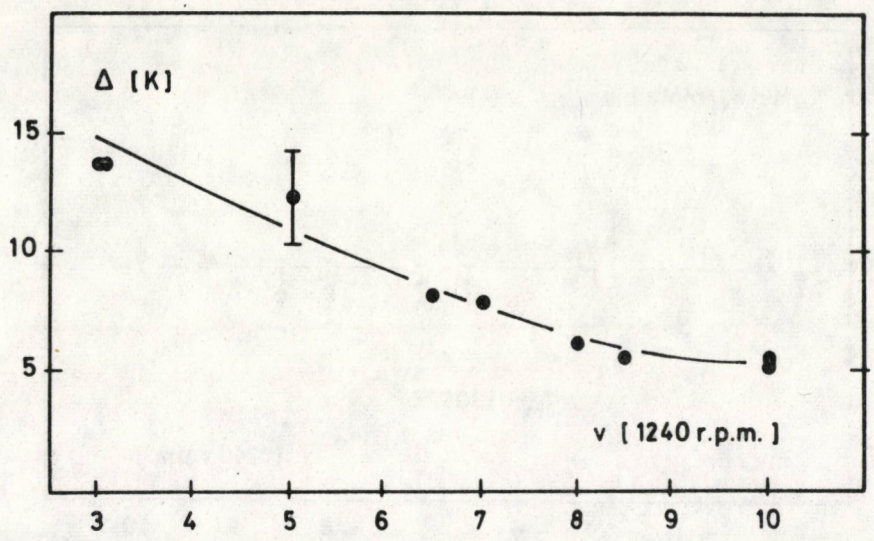

Fig. 4. Half-width of the crystallization peak vs. rotating speed

increasing v. This dependence indicates a change in the kinetics of the crystallization. The kinetic parameters of the samples made at the lowest and the highest v are listed in Table 1.

Table 1

heating rate (K/min)	v = 3720 r.p.m. /="3"/ k_o /10^{17} sec^{-1}/	n	v = 12400 r.p.m./="10"/ k_o /10^{19} sec^{-1}/	n
2.5	1.5	2.2 ± o.15	2.o	4.2 ± o.4
5	1.75	2.o ± o.1	2.4	6.5 ± o.3
1o	1.5	1.8 ± o.1	3.o	13 ± 2
2o	1.7	2.2 ± o.1	3.9	16 ± 5
E_a	254 ± 4	kJ/mole	29o ± 2o	kJ/mole

The samples were heat treated for 8 hours at 300 C. No significant changes were observed in the crystallization of the sample made at low rotating speed, however the crystallization peak of that made at high v grew slightly wider */Fig. 5./*. Crystallization energies remained identical in both cases.

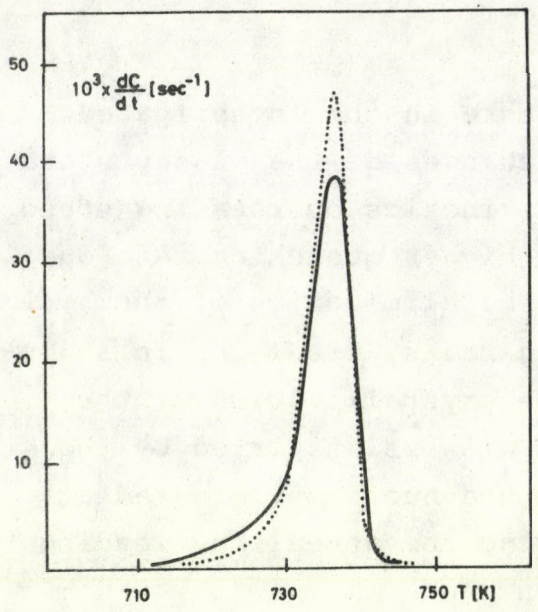

Fig. 5.
The effect of heat treatment on the shape of the crystallization peak, observed by 20 K/min DSC measurements. Dotted line stands for the as-quenched and the solid one for the heat treated sample

DISCUSSION

It is shown that crystallization of $Fe_{1-x}B_x$ $0.12<x<0.25$ metallic glasses is a combination of two processes, namely α-Fe and Fe_3B precipitation [4]. Therefore DSC measurements give apparent kinetic parameters, appearing as the average of those of the simultaneous processes. Earlier works [5, 6] studiing these processes separately mention nearly n = 1.5 for α-Fe and about n = 3 for Fe_3B precipitation which are the special values valid for zero nucleation rate. Transformation kinetics generally depends upon the heating rate of the experiment [7], the only realistic exception is the case of zero nucleation rate.

Taking these facts into account the heating rate independence and also the value of n observed on sample made at v = 3720 r.p.m. suggest that crystallization takes place with the growth of the quenched-in nuclei in this case. The Avrami-exponent, n of the sample produced at high rotating speed /v = 12400 r.p.m./ is significantly higher and shows a marked dependence on the heating rate. It indicates the role of nucleation in the crystallization process.

CONCLUSIONS

Variation of the melt temperature in the investigated range does not yield any apparent changes of the glassy state.

Dependence of crystallization kinetics on rotating speed can be interpreted as follows: The higher quenching rate due to higher rotating speed leads to the elimination of the nuclei which were present at low quenching rates, resulting in an increasing nucleation rate during the crystallization of the more rapidly quenched sample. This wiew is supported by the results on heat treatment: Some of the nuclei eliminated at high quenching rate may appear during the annealing, leading to a somewhat wider crystallization peak.

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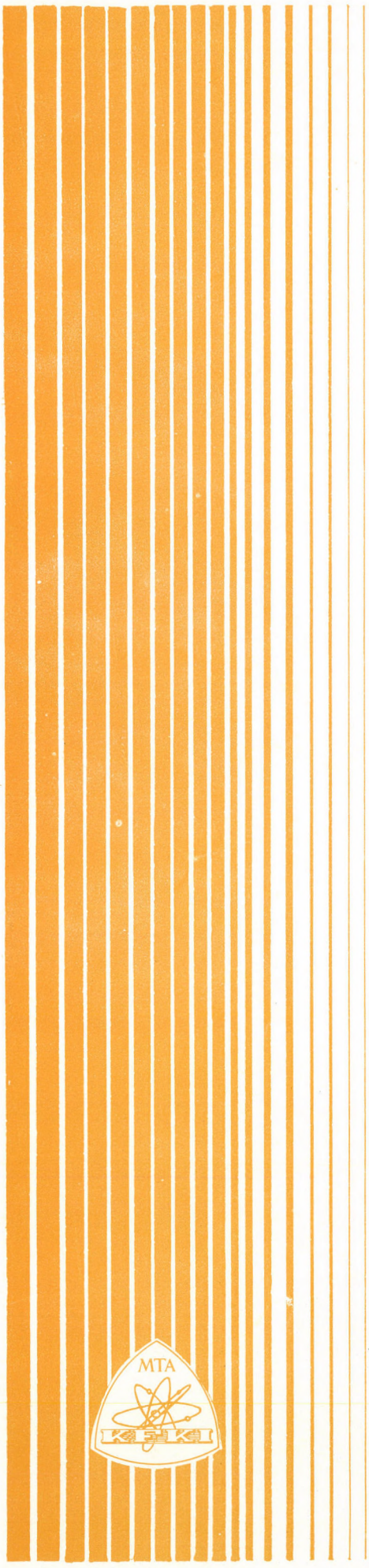

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Felelős kiadó: Tompa Kálmán
Szakmai lektor: Hargitai Csaba
Nyelvi lektor: Hargitai Csaba
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