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Crystallization and mechanical properties of some Fe-Ni base metallic glass

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Résumé. — Le processus de cristallisation a été déterminé en fonction du temps et de la température pour le verre métallique $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$. L'énergie d'activation du processus de cristallisation a été trouvée égale à 4,5 eV/at. en utilisant l'équation d'Arrhénius. Les mesures de microdureté ont permis d'étudier la stabilité de cet alliage dans une gamme de température allant de 20 °C à 450 °C. Les essais de traction ont été faits en fonction du temps et de la température. Le début de la cristallisation a été déterminé en utilisant la méthode des moindres carrés.

Abstract. — Crystallization process has been determined as a function of time and temperature for the metallic glass $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$. The activation energy for the crystallization process was found to be 4.5 eV using an Arrhénius type equation. The stability of the studied alloy on annealing in the range 20 to 450 °C has been investigated by microhardness measurements. Also tensile tests were made as a function of time and temperature. The onset of crystallization of the same metallic glass has been determined numerically.

1. Introduction. — In the glassy state, metals have some remarkable properties that promise to be quite advantageous in a variety of applications. One of these properties is a great strength which, for certain alloys, can exceed three times that of stainless steel. Furthermore, unlike strong and hard crystalline metals, which are normally very brittle, metallic glass exhibit a high plasticity. These substances are produced by a very rapid quenching from the liquid state [1-6], for instance by propelling small high velocity globules onto cold surfaces, a technique known as splat quenching. More recent methods [7-9] involve the injection of molten material between cold counter-rotating drums, which made it possible to initiate modern and quantitative investigations on the various properties of amorphous metals due to the economical mass production of good quality amorphous ribbons. Recently a number of researchers have been showing a considerable interest in the characteristics such as the mechanical strength and the hardness of amorphous ribbon specimen produced by a continuous rapid quenching method [10].

In 1977, Luborsky [11] investigated the crystallization of some Fe-Ni metallic glasses ($\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$, $\text{Fe}_{40}\text{Ni}_{40}\text{B}_{20}$ and $\text{Fe}_{80}\text{B}_{20}$). The onset of crystallization was found to be a thermally activated process;

the activation energies, ΔE , for the three alloys were 3.9 eV, 3.0 eV and 2.1 eV respectively. He observed experimentally that the activation energy for the initiation of crystallization increases with the number of different atomic species in the amorphous alloy.

Scott [12], in 1978, studied the crystallization kinetics of two commercial Fe-Ni metallic glasses near their glass transition point. He estimated the activation energies for the crystallization process from the time to 50 % transformation are close to 100 kcal.mol⁻¹ which is close to those measured for viscous flow.

In 1979, it was proved experimentally that the first crystallization of $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$ metglass is a two stage process [13], called MSI and MSII, rather than one stage as described by Chang and Sastri [14]. Furthermore, the stability of Fe-Ni base metallic glasses on annealing in the range 30 to 1 000 °C was investigated by microhardness measurements [15]. The authors concluded that the microhardness increases with increase of Fe as well as B content in samples that cover a wide range of composition, which indicate the possible similarity of the structural changes on heating all amorphous Fe-Ni base alloys.

Recently [16], it was found that the amorphous alloy $\text{Fe}_{40}\text{Ni}_{40}\text{P}_{14}\text{B}_6$ crystallizes by a eutectic mechanism to an austenitic and a body-centred tetragonal phase. The purpose of this article is to review our experimental results on the $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$ amor-

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phous alloy. The selected topics are listed as follows :

- the beginning of crystallization of the $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$ metallic glasses ;
- the measurements of the mechanical properties of the same alloy.

2. Experimental. — The amorphous alloy $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$ ribbons were purchased from Allied Chemical Metglass — New Jersey 07960 (1978 at Saclay 21^e Colloque de Métallurgie). The amorphous condition of the as-received material was checked by X-ray diffractograms [17]. These ribbons (of about 13 mm in width and 0.07 mm in thickness) were cut into ribbons (of about 0.7 mm in width and the gauge length 5 mm) for tensile tests and into pieces of 10 mm for microhardness measurements. The heat-treatment consisted of an isothermal annealing in air in the temperature range 20 to 450 °C. This temperature range was divided into periods and the samples were successively annealed at each chosen temperature for 15, 30 and 60 min. After each annealing step, the measurements were performed. The microhardness data were obtained using a Shimadzu type M equipment with 1 000 gm Load. During heat-treatment, the sample become very brittle [18] and the measurements were performed carefully.

3. Results and discussion. — The crystallization temperatures were determined by differential scanning calorimetry at heating rate of 10 K/min. (Fig. 1). It was found that the time to initiate crystallization is a function of the reciprocal of the absolute temperature as shown in figure 2. The onset of crystallization can be described by an Arrhenius type equation for a thermally activated process

$$t = t_0 \exp(\Delta E/KT)$$

Fig. 1. — DSC scan of the as-received sample.

Fig. 2. — The temperature dependance of the relaxation time, the individual relaxation times are taken as varying with temperature in an arrhenian manner, as : $t = t_0 \exp(\Delta E/KT)$ [21].

with the constant t_0 . The activation energy for the initiation of crystallization was calculated and it was found to be 4.5 eV.

Tensile properties of the investigated amorphous alloy was studied after different times at different temperatures. The load-elongation curves were not straight and the deviation from the linearly existed well below the fracture point as illustrated in figures 3, 4 and 5. An experimental correlation was found, showing that the load *versus* elongation curves rise

Fig. 3. — Load-elongation curve at different temperatures for annealing time 15 min.

Fig. 4. — Load-elongation curve at different temperatures for annealing time 30 min.

Fig. 5. — Load-elongation curve at different temperatures for annealing time 60 min.

steeper and steeper with increasing temperature up to 100 °C, and after that they steepness decreases with increasing temperatures, for all values of elongation. It is interesting to note that, in the alloy studied, at the same temperature and at different times, it is also indicated that the maximum load and elongation were decreased. The results indicate that a complete view of the mechanical properties depend on crystal-

lization. This tendency is quite similar to the behaviour of $\text{Fe}_{55}\text{Pb}_{35}\text{P}_{10}$ [19, 20].

From the microhardness measurements, it is well established that the maximum hardness H_v obtained is 1 922 kg/mm^2 after 15 min. at 100 °C. With the increase in temperature, the hardness decreases as shown in figure 6a and from the figures (6a, 6b), it is concluded that the hardness is a function of temperature and time. From the curves, it is noticed that at temperatures between room temperature and 100 °C there is a gradual increase in hardness due to the effects of structural relaxation, decreasing somewhat between 100 °C and 150 °C. From 150 °C to 210 °C there is a rapid and abrupt decrease. But above 210 °C there is a gradual decrease up to 450 °C where crystallization is essentially complete. The measured hardness values are listed in table Ia, Ib and Ic along with corresponding yield stress.

Table Ia.

— Time of annealing 15 min.

Temperature T °C	Hardness Hv Kg/mm ²	yield stress oy Kg/mm ²	Hv/oy
20	1715	470	3.65
50	1811	482	3.76
100	1922	560	3.43
150	1918.5	528	3.63
210	1786	450	3.97
270	1681	384	4.38
330	1639	204	8.03
405	1607	172	9.34
450	1582	76	20.82

Table Ib.

— Time of annealing 30 min.

Temperature T °C	Hardness Hv Kg/mm ²	Yield stress oy Kg/mm ²	Hv/oy
20	1715	470	3.65
50	1750	480	3.65
100	1786	510	3.50
150	1782	500	3.56
210	1703	440	3.87
270	1639	330	4.97
330	1605	212	7.57
390	1572	176	8.93
450	1554	72	21.58

Table Ic.

— Time of annealing 60 min.

Temperature T °C	Hardness Hv Kg/mm ²	yield stress oy Kg/mm ²	Hv/oy
20	1715	470	3.65
50	1726	502	3.44
100	1734	546	3.18
150	1730	544	3.18
210	1644	430	3.82
260	1604	200	8.02
345	1564	148	10.57
430	1544	60	25.73

Fig. 6. — a) Microhardness versus temperature at different annealing time.

Fig. 6. — b) Microhardness versus time at different temperatures.

The metastable crystallization phase was determined using Fortran V Programmes developed for this purpose of this work. From the results of the least square fitting for the experimental data of the microhardness measurements it is found that the temperature to initiate crystallization is at 100 $^{\circ}\text{C}$ (Fig. 7).

Fig. 7. — Microhardness versus temperatures, after the results of least square fitting data of the microhardness measurements.

4. Conclusion. — From the results, we conclude that :

— The activation energy for the initiation of crystallization was found to be 4.5 eV which is more higher than the values described by Luborsky [11]. This indicates that the studied alloy more stable than those studied by him.

From the experimental results (microhardness measurements), it could be concluded that the beginning of nucleation is at 100 $^{\circ}\text{C}$ and the end of nucleation is at 150 $^{\circ}\text{C}$. These two maxima indicate possible structure changes in the amorphous as well as in the crystalline states.

— It is interesting to compare our results with that of Von Heimendahl and G. Maussner [13] on the Fe-Ni based amorphous alloy $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$. It is determined that, this alloy exhibits one stage process of crystallization rather than two stages as described by them. So it behaves the same characteristics as reported by Chang and Sastri [14].

— Judging from the load-elongation curves, it may be assumed that existence of correlation between the onset of crystallization, where a periodical atomic arrangement embrittled $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$ amorphous alloy, and temperature.

— It is also observed that $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$ metallic glasses exhibit a ratio of hardness to yield stress of ~ 3.5 .

— Finally, disadvantage is that $\text{Fe}_{32}\text{Ni}_{36}\text{Cr}_{14}\text{P}_{12}\text{B}_6$ metallic glasses has limited thermal stability, i.e. at a

few hundred degrees centigrade, it crystallizes and drastically changes its properties.

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