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Explosive crystallisation of metal glasses based on Fe-B during pulsed laser heating. Experiment and modelling

O.V. Smolyakov [a], V.V. Girzhon [b], S.I. Mudry [c], Y.S. Nykyruy [c,*]

[a] Metal Physics Department, Zaporizhzhia National University, Zaporizhzhia, Ukraine
[b] Physical Materials Science Department, National University "Zaporizhzhia Polytechnic", Zaporizhzhia, Ukraine
[c] Physics of Metal Department, Ivan Franko National University of Lviv, 8 Kyrylo and Mefodiy, Lviv, Ukraine
* Corresponding e-mail address: yuliya.nykyruy@lnu.edu.ua
ORCID identifier: https://orcid.org/0000-0003-1760-8840 (S.I.M.);
https://orcid.org/0000-0001-5808-0857 (Y.S.N.)

ABSTRACT

Purpose: The structure evolution of amorphous metallic alloys during different kinds of thermal effects is an important problem of disordered systems physics. A precise evolutional model would allow predicting the formation of such a structural state, providing the necessary physical and mechanical alloy properties.

Design/methodology/approach: The paper is devoted to the problem of modelling the explosive crystallisation process in metal glasses induced by laser, supplemented by experimental results.

Findings: A theoretical model of laser-induced explosive crystallisation in metal glasses is proposed. A pulse laser heating method for the surface processing was developed, making it possible to obtain two-layer structures with an adjustable thickness of the amorphous crystalline layer.

Research limitations/implications: The proposed model is assumed to test and optimes for metal glasses of other chemical compositions.

Practical implications: A theoretical model of laser-induced explosive crystallisation in metal glasses allows for predicting and controlling structure changes to obtain the desired properties.

Originality/value: The investigation of structure changes at rapid heating of amorphous alloys by experimental methods is very limited in obtaining data and their interpretation. For that reason, combining the modelling with experimental measurements is proposed. The results of this work have value for a scientist in material science, physics and engineering, which use nonequilibrium physical processes to obtain new materials, including nanoscale systems.

Keywords: Laser-induced explosive crystallisation, Metal glass, Structure modelling

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METHODOLOGY OF RESEARCH, ANALYSIS AND MODELLING

1. Introduction

The structure evolution of amorphous metallic alloys during different kinds of thermal effects is an important problem of the physics of disordered systems. It can be explained by the fact that almost all material properties are sensitive to structural changes [1]. Therefore, a precise model of such changes would allow prediction of the formation of such a structure state, providing the necessary set of physical and mechanical alloy properties.

One of the most important questions of metal glass physics is the study of the transition from an amorphous to a crystalline state. Given different thermal conditions, the structure formation process in metal glasses can vary significantly, and crystallisation would result in quite different definitive structures [2]. Another valid question concerns the impact of heating speed on phase formation processes during metal glass crystallisation. Hence, this research aims to study structure-forming processes in metal glasses of the Fe-B system during pulsed laser heating.

2. Description of the approach

The research objects were the metal glass ribbons of composition $Fe_{80}Si_6B_{14}$, $Fe_{76}Si_{13}B_{11}$, and $Fe_{72}Ni_9Si_8B_{11}$, all obtained by the melt spinning technique. Crystallisation processes were studied using XRD after isothermal annealing and pulsed laser heating. Annealing was performed in a resistive heating furnace with different temperatures on a timescale of 30 minutes; laser heating was performed using a pulsed YAG laser (λ = 1.06 μm) in an argon atmosphere utilising different power densities. Crystallisation kinetics was described using conventional crystallisation theory [3].

3. Results and discussion

Metal glass crystallisation processes during different kinds of thermal influence were explored in numerous works [4-12]. In [13-22], in particular, crystallisation processes during laser heating were researched. Comparative analysis of crystallisation of the alloys $Fe_{80}Si_6B_{14}$, $Fe_{76}Si_{13}B_{11}$, and $Fe_{72}Ni_9Si_8B_{11}$ after annealing and pulsed laser heating is presented in [23,24]. That works show a multitude of crystallisation process particularities during laser heating: the change of crystallisation type from primary to eutectic (according to classification [25]), the formation of various types of borides as well as an α-solid solution with anomalous lattice parameters. This work is a continuation of research on crystallisation processes in these glasses during laser heating. One of the peculiarities is explosive crystallisation. It comes down to the fact that during laser heating, some samples stay X-ray amorphous or immediately crystallise after reaching certain values of power density q of laser radiation (e.g. alloy $Fe_{76}Si_{13}B_{11}$). In another case (alloy $Fe_{72}Ni_9Si_8B_{11}$) with the increase of q, the ribbon structure is in the two-phase state (amorphous matrix + α-solid solution). Still, after reaching a specific q value, it immediately crystallised.

Considering a rather significant change in the kinetic characteristics of crystallisation during laser heating, the following should be considered. The area of the sample affected by laser heating only absorbed about 1 J of pulse energy, due to the reflectance of the metallic surface [22,24]. Using the known quantity of heat produced during crystallisation from the liquid state, approximate quantities of heat produced in this case were computed. The results show that the irradiated area of the sample released close to 1 J of heat energy during crystallisation. The amount of heat produced in a time frame of a laser pulse could cause significant heating of the area affected by the laser beam. According to [26], an increase in temperature of a few tens of degrees could increase the nucleation rate in metal glasses by 2-3 orders of magnitude. This can explain the rapid change of crystallisation characteristics during laser heating with the critical q mentioned above. It can be assumed that in these conditions, the temperature gets to a point when crystallisation heating begins to dominate over the heat removal from the affected area. Then crystallisation will occur with an increase in temperature and velocity, therefore taking explosive characteristics [27]. At lower power density, q heat removal dominates heat production caused by crystallisation. This causes temperature reduction in the affected area and subsequent inhibition of crystallisation processes.

Thus, summarising and taking into account the results of the work [23], it is possible to conclude that the distinguishing feature of explosive crystallisation is the rapid transformation to a crystalline state from either completely amorphous or partially crystalline during a slight increase of applied power density (few MW/m^2).

Considering how diffusion-related processes define the crystallisation rate, we can presume that the effect of chemical stratification should be minimal during explosive crystallisation. It is experimentally proven that the intensity of the diffraction peaks caused by the phases Fe_2B and α-(Fe, Ni) after laser heating of the alloy $Fe_{72}Ni_9Si_8B_{11}$ was significantly lower than after isothermal annealing [24]. Then it can be assumed that the crystallisation rate becomes sufficient for the process to occur using the explosive mechanism as a result of the formation of a sufficiently large amount of the over-saturated α- solid solution. An insignificant amount of the phases Fe_2B and γ-(Fe, Ni) requires significant chemical stratification, therefore does not affect crystallisation by a large margin. A similar reduction of diffraction peak intensities of the phase $(Fe, Si)_2B$ after laser heating (compared to isothermal annealing) was also noticed in the alloy $Fe_{76}Si_{13}B_{11}$ [20]. Such similarity in the crystallisation processes of alloys $Fe_{72}Ni_9Si_8B_{11}$ and $Fe_{76}Si_{13}B_{11}$ verifies the assumption about the significant impact of the over-saturation of the α-solid solution with silicon on the crystallisation rate during laser heating. The same conclusion can be made by comparing these results with the results in [19], which are dedicated to researching the crystallisation processes of Fe-B alloys. It is known that silicon is highly soluble in α-iron [28], and when compounded with boron, it efficiently enhances iron's tendency to amorphised, as well as raises its crystallisation temperature [29].

On the other hand, an increase in silicon concentration allows reducing the concentration of boron, the solubility of which is lower, without a dramatic impact on thermal stability. Hence, replacing not very soluble boron with easily soluble silicon coupled with a shift crystallisation temperature higher, where the nucleation rate is greater, would benefit the explosive crystallisation. Therefore, replacing boron with silicon reduces the limiting impact of diffusion on the crystallisation processes that happen during laser heating in conditions of high cooling rates (~10^4 K/s).

For purposes of theoretical proving of the possibility of explosive crystallisation incident during pulsed laser heating, the modelling of polymorphic crystallisation of alloys similar to $Fe_{75}(B, Si)_{25}$ was conducted. The formulas of the classical theory were used to describe the kinetics of the process [5]:

$$X(t) = 1 - exp(-\frac{\pi}{3} I U^3 t^4) \quad (1)$$

where I and U – are the nucleation rate and crystal growth rate, respectively; X – the crystalline fraction of the crystalline volume; t – is the time passed since the beginning of the heating. To calculate the rate of homogenous nucleation and diffusion-controlled crystal growth, the following formulas were used [1,28,29]:

$$I(T) = \frac{N_0 D}{a_0^2} exp\left(-\frac{16\pi\sigma^3 V_m}{3kT\Delta G^2}\right) \quad (2)$$

$$U(T) = \frac{D}{a_0}\left[1 - exp\left(-\frac{\Delta G}{RT}\right)\right] \quad (3)$$

where N_0 – is the number of atoms in the unit of volume; a_0 – is the length of the diffusion jump (average atom diameter), D – diffusion coefficient, T – temperature, k – Boltzmann constant, σ – specific free energy of crystal boundary, V_m – a molar volume of the amorphous phase, ΔG – the difference between free energies of the amorphous and crystalline phases.

The values of thermodynamic motive force were calculated using Thomson-Spaepen approximation [30], developed for metallic alloys:

$$\Delta G(T) = \frac{2\Delta H_m T(T_m - T)}{T_m(T_m + T)}, \quad (4)$$

where ΔH_m and T_m – are the heat and melting point, respectively:

$$D = D_0\, exp\left(\frac{-E}{RT}\right), \quad (5)$$

where E – is the activation energy.

For the numerical calculation of the fraction of the crystallised volume $\Delta X(t_i)$, the time-differentiated formula (1) was used:

$$\Delta X(t_i) = \frac{4\pi}{3} I(t_i) U^3(t_i) t_i^3\, exp\left(-\frac{\pi}{3} I(t_i) U(t_i)^3 t_i^4\right) \Delta t;$$
$$X = \sum \Delta X(t_i), \quad (6)$$

where $\Delta t = 10^{-6}$ s, $t_i = t_{i-1} + \Delta t$.

In formula (6), the temperature changes due to the heat release during crystallisation of the volume $\Delta X(t_i)$ and heat transfer to the environment have been taken into account in the calculations. In the next step (calculation of $\Delta X(t_{i+1})$), formulas (2) ... (5) were used, taking into account the already changed temperature. The quantities included in the formula (2) ... (5) were taken from works [31,32] for crystallisation of Fe_3B phase: T_m = 1424 K; ΔH_m = 14.2 kJ/mol; E = 243.65 kJ/mol; D_0 = 0.615 m^2/s; a_0 = 0.23 nm; σ= 0.234 J/m^2; V_m = $6.6 \cdot 10^{-6}$ m^3/mol. The heat capacity and the density of the alloy at constant temperature were chosen constant.

In the simulation of the crystallisation process, it was considered that the amorphous ribbon with a thickness of 30 µm was heated by a non-focused laser beam with a cross-sectional area of 10^{-4} m^2 instantaneously to a certain temperature T_m. The temperature changed only due to processes of crystallisation and radiative heat losses from both surfaces of the ribbon. The radiation power density was estimated from the formula:

$$q = A\sigma_0(T^4 - T_0^4), \quad (7)$$

where σ_0 – Stefan-Boltzmann constant; T_0 – room temperature; A – radiation coefficient, magnitude of which is considered equal to 0.2.

The heat transfer on the boundary of the ribbon-air surface was neglected since the contribution of this process to the ribbon temperature change was an order of magnitude lower than the radiation. To simplify the calculations, it was considered that the ribbon temperature was the same throughout the volume.

According to calculations, given starting temperature T ≤ 907 K, only partial crystallisation took place (Fig. 1). At T_s = 907 K, the part of crystallised volume has not exceeded 12%, and at T_s = 906 K, this part was reduced to 4%. An increase in starting temperature of 1 degree caused a significant change in crystallisation kinetics (Fig. 2). After approximately 1 ms, a rapid spike in temperature was observed, caused by a significant excess of the amount of heat generated compared to the amount of heat transferred to the surrounding space. Subsequently, the temperature was steadily dropping. Almost complete crystallisation has occurred (the crystallised volume part was equal to 96%). Thus, the proposed model confirms that explosive crystallisation of amorphous metal ribbons under pulse heating using a millisecond laser may occur under certain conditions.

Subsequently, the effect of the diffusion constants E and D_0, which may change during the alloying of Fe-B systems, on the minimum initial temperature T_s, at which explosive crystallisation begins, was investigated (Tab. 1).

The analysis of calculation data made it possible to obtain an empirical criterion for explosive crystallisation for a system $Fe_{75}(B, Si)_{25}$:

$$E < (270 + 8.5 \ln D_0) \text{ kJ/mol} \quad (8)$$

If condition (8) is not satisfied, then, under given heat transfer conditions, the amorphous ribbon will crystallise only partially or, practically, will not crystallise at pulsed laser heating to any temperature. We introduce a limit on the temperature value of explosive crystallisation, for example, T_e 900 K. Then the criterion of explosive crystallisation will have the form (9) and will somewhat differ from the expression (8), the data for calculations of which were taken from [32].

$$E < (246 + 7{,}3 \ln D_0) \text{ kJ/mol} \quad (9)$$

Let's assume that for other amorphous alloys, the criterion of explosive crystallisation is similar to (8). Then in a more general way, the criterion of explosive crystallisation can be written as follows:

$$E < (E_0 + A \ln D_0) \text{ kJ/mol} \quad (10)$$

Here parameters E_0 and A depend on the alloy properties, such as T_m, ΔH_m, σ, V_m, ribbon thickness, etc.

Based on this, using (9), it is quite easy to explain the experimentally established fact of explosive crystallisation under the influence of pulsed laser heating in the alloy $Fe_{76}Si_{13}B_{11}$ and the impossibility of a complete crystallisation in the alloy $Fe_{80}Si_6B_{14}$.

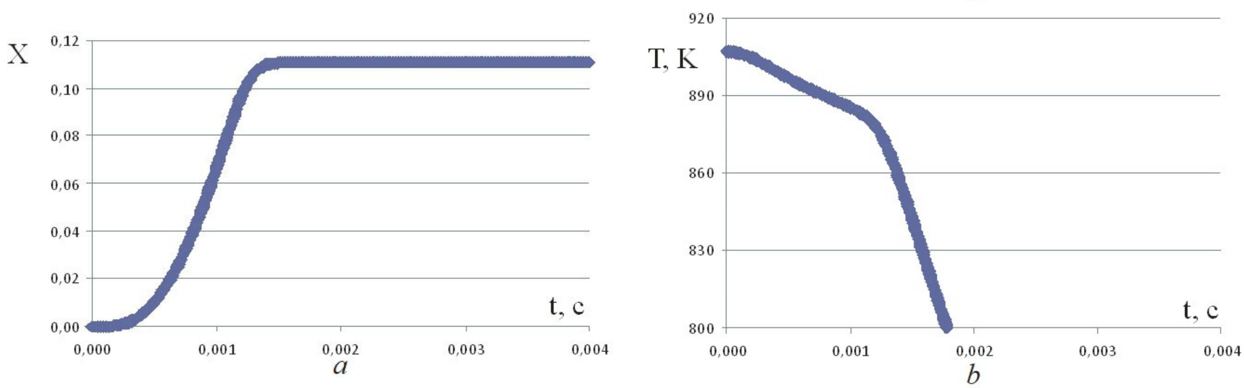

Fig. 1. Modeling of the crystallisation process of an amorphous metal alloy with pulsed heating to a temperature of 907 K: a) the dependence of the crystallised volume part on time; b) temperature change in the process of crystallisation

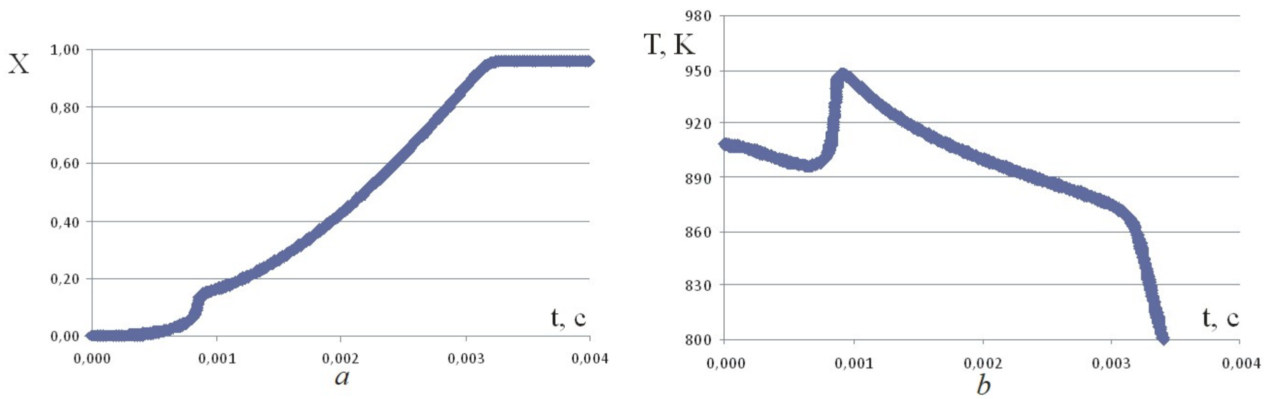

Fig. 2. Modeling of the crystallisation process of an amorphous metal alloy with pulsed heating to a temperature of 908 K: a) the dependence of the crystallised volume part on time; b) temperature change in the process of crystallisation

Table1.
Dependence of the temperature of explosive crystallisation on the parameters of the diffusion coefficient

D_0, m²/s	*E*, kJ/mol	T_B, K	Commentary
0.1	243.65	1132	Temperature spike does not exceed starting temperature (Fig. 3)
0.2		975	
0.3		946	
0.4		929	
0.5		918	
1.0		886	
2.0		860	
5.0		829	
0.615	210.0	741	
	220.0	785	
	230.0	832	
	240.0	886	
	250.0	952	
	260.0	-	Only partial crystallisation
	270.0	-	Part of the crystallised volume does not exceed 0.1%

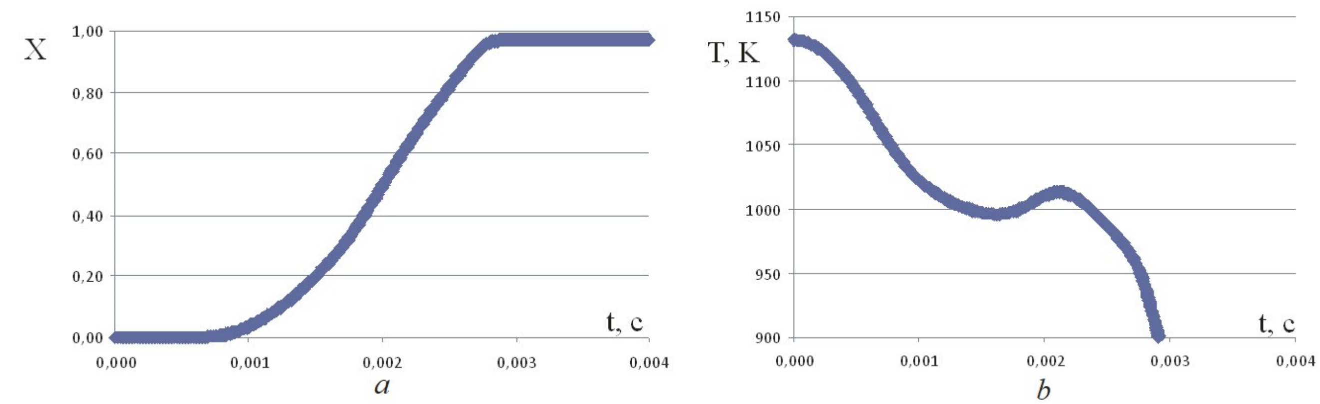

Fig. 3. Simulation of the crystallisation process of an amorphous metal ribbon at pulsed heating to temperature 1132 K at E = 243.65 kJ/mol and D_0 = 0.1 m²/s: a) the dependence of the crystallised volume part on time; b) temperature change in the process of crystallisation

According to the modelling results, the crystallisation of metal glass under the influence of pulsed heating can occur completely – by explosive mechanism or only partially, which does not correspond to reality. For example, the crystallisation of classical alloys Fe-B and $Co_{68}Fe_4Cr_4Si_{13}B_{11}$ occurs "gradually" with increasing density of laser radiation from a certain minimum to a certain maximum. This contradiction can be explained by the fact that the model does not consider the presence of "frozen" crystallisation centres, which can significantly affect the kinetics of crystallisation [32,33,34] and the possibility of phase transformations in the crystalline state. Indicated factors and assumptions about instant heating of the entire volume of amorphous ribbon also limit the possibility of using a model for analysing the crystallisation of glasses based on a transition metal metalloid system under the influence of pulsed lasers. Therefore, the model needs further optimisation and refinement.

4. Conclusions

1. Fast laser heating due to short pulse duration allows to reach temperatures on the $Fe_{80}Si_6B_{14}$ alloy surface, which exceed the temperature of the peritectic reaction $Fe_2B \Leftrightarrow FeB + Fe$, and fix the amorphous-crystalline state of the alloy «amorphous matrix + α-(Fe, Si) + FeB», which is impossible to achieve during slow heating or isothermal annealing.
2. The formation of a supersaturated solid solution based on iron in conditions of pulsed laser heating of

$Fe_{76}Si_{13}B_{11}$ and $Fe_{72}Ni_9Si_8B_{11}$ amorphous alloys reduces the role of diffusion separation during crystallisation, which provides the opportunity for an explosive crystallisation mechanism.
3. Pulse laser heating of the surface of the $Fe_{72}Ni_9Si_8B_{11}$ ribbon with the cooling of the opposite surface with water makes it possible to obtain two-layer structures of the type «amorphous matrix + α-(Fe, Si) – amorphous matrix» with an adjustable thickness of the amorphous crystalline layer.

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