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Kinetics and mechanism of structural transformations of Fe$_{75}$Ni$_2$Si$_8$B$_{13}$C$_2$ amorphous alloy induced by thermal treatment

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ABSTRACT

The kinetics of structural transformations of the Fe$_{75}$Ni$_2$Si$_8$B$_{13}$C$_2$ amorphous alloy under non-isothermal conditions was studied. It was shown that the amorphous alloy was stable up to 723 K, when multi-step structural transformations began, involving formation of stable α-Fe(Si) and Fe$_2$B crystalline phases, and a metastable Fe$_{15}$Si$_3$B$_2$ phase. Their growth and the decomposition of the metastable phase into the two stable phases. The effects of structural transformations on the established kinetic model were discussed in detail.

1. Introduction

Metallic glasses are kinetically and thermodynamically metastable materials. They are usually stable at room temperature and transform to more stable crystal forms at higher temperatures [1]. Due to their excellent soft magnetic properties, such as high saturation magnetization, high permeability, low coercivity and loss, these materials are used in many different applications, such as power devices, information technology, magnetic sensors, anti-theft security systems [2,3]. The change in structure can lead to changes in their technologically important properties, such as the heat capacity, electrical resistivity, volume and magnetic properties [4–7]. This can, sometimes, improve their performance, but it can also deteriorate it, making them suitable for single-use only. This makes it important to study the kinetics of phase transformations induced by thermal treatment of amorphous alloys.

Our research of Fe-based amorphous alloys showed that, at high temperatures, thermally treated amorphous alloys undergo single- or multi-step processes of structural phase transformations. The kinetics of these processes was investigated in detail [8,9]. We also studied the structural transformations of the Fe$_{75}$Ni$_2$Si$_8$B$_{13}$C$_2$ amorphous alloy under non-isothermal conditions [10]. These studies showed that the Fe$_{75}$Ni$_2$Si$_8$B$_{13}$C$_2$ amorphous alloy is stable under the temperature of 723 K, when multi-step structural transformations begin. The primary crystallization already started at 723 K with formation of the Fe$_2$Si crystalline phase in the amorphous matrix. At higher temperatures (between 780 and 800 K) we detected the presence of the boron–iron–silicon phase (B$_2$Fe$_{15}$Si$_3$) as well as the iron–boron (Fe$_2$B) phase. Above 870 K only two phases, Fe$_2$Si and Fe$_2$B, were detected. The present paper is concerned with the study the non-isothermal mechanism and kinetics of multi-step processes of structural transformations of Fe$_{75}$Ni$_2$Si$_8$B$_{13}$C$_2$ amorphous alloy in 293–1273 K temperature range, with particular emphasis on correlation of structural transformations and the kinetic parameters.

2. Experimental procedure

2.1. Materials and techniques

The ribbon-shaped samples of Fe$_{75}$Ni$_2$Si$_8$B$_{13}$C$_2$ amorphous alloy (2 cm wide and 35 μm thick) were obtained using the standard procedure of rapid quenching of the melt on a rotating disc (melt-spinning method).

The thermal stability of the alloy as well as its structural transformations were investigated by the differential scanning calorimetry (DSC) in a nitrogen atmosphere using a DSC-50 analyzer (Shimadzu, Japan). In this case, samples weighing several milligrams were heated in the DSC cell from room temperature...
to 973 K in a stream of nitrogen with a flow rate of 20 ml min\(^{-1}\) at different heating rates. The overlapping exo-peak was resolved using Peakfit software version 4.11 [11,12]. The peak resolution was conducted using a combination of Gaussian and Lorentzian functions. Only the fitted peaks with correlation factor \(R^2 > 0.995\) were accepted as a good fit.

### 2.2. Solid-state kinetic analysis

All kinetic analyses of solid-state transformations are based on a single-step kinetic equation:

\[
\frac{dx}{dt} = k(T)f(\alpha) \tag{1}
\]

where \(k(T)\) is the rate constant, \(t\) is the time, \(T\) is the temperature, \(\alpha\) is the fractional extent of reaction (rate conversion), and \(f(\alpha)\) is a conversion function (kinetic model) which depends on the particular reaction model.

The temperature dependence of the rate conversion is introduced by replacing \(k(T)\) with the Arrhenius equation, which gives the relation:

\[
\frac{dx}{dt} = A \exp\left(-\frac{E}{RT}\right)f(\alpha) \tag{2}
\]

where \(A\) (pre-exponential factor) and \(E\) (activation energy) are the Arrhenius parameters and \(R\) is the gas constant.

Kinetic description of solid state transformations usually includes a kinetic triplet, involving Arrhenius parameters (activation energy, \(E\) and pre-exponential factor, \(A\)) as well as an algebraic expression of the conversion function, \(f(\alpha)\) describing the dependence of the reaction rate on the conversion degree, \(\alpha\).

In solid state reactions, the constant value of activation energy can be expected only for a single-step reaction and \(E\) in Eq. (2) becomes an apparent quantity (\(E_a\)), based on a quasi-single-step reaction. In non-isothermal measurements at constant heating rate, \(\beta\), Eq. (2) transforms to:

\[
\beta \frac{dx}{dt} = A \exp\left(-\frac{E_a}{RT}\right)f(\alpha) \tag{3}
\]

where \(dx/dt = \beta \cdot dx/dt\).

The rate conversion \(dx/dt\) is proportional to the measured specific heat flow \(\Phi\), normalized per sample mass (\(W g^{-1}\)):

\[
\frac{dx}{dt} = \frac{\Phi}{\Delta H} \tag{4}
\]

where \(\Delta H\) corresponds to the total enthalpy change associated with the solid-state transformations. The fractional extent of reaction \(\alpha\) can easily be obtained by partial integration of non-isothermal thermal analysis curve.

### 3. Results and discussion

#### 3.1. Thermal behavior and structural transformations induced by thermal treatment

As-prepared Fe\(_{75}\)Ni\(_{25}\)Si\(_8\)B\(_{13}\)C\(_2\) alloy is stable up to a temperature of 723 K when the multi-step crystallization begins, exhibiting overlapping crystallization peaks in DSC (Fig. 1), between 790 and 900 K, depending on the heating rate [10].

The appearance of such overlapping peaks in the process of crystallization of amorphous alloys could be provoked by several stages of crystallization of compounds involving different constituents, produced during a reaction, or by crystallization of compounds involving same constituents in several different stoichiometries. The overlapping peaks can also indicate that the nucleation and growth of crystals take place simultaneously. In overlapping peaks, there are intervals where the experimental data corresponds only to the summed values of multiple individual steps, rather than to any individual step in particular. If the overlap is negligible (the second step begins as first one is almost finished) this can be ignored, but when the degree of overlap is significant (in simultaneous steps) it is necessary to resolve the complex peaks.

The resolution of the complex peaks yielded two well separated peaks for heating rate 5 K min\(^{-1}\) and three for the higher heating rates (Fig. 2).

Fig. 2a and b shows the experimental (dashed lines) and resolved DSC curves (solid lines) at heating rates 5 and 10 K min\(^{-1}\), respectively. The changes in intensities and characteristic temperatures with the heating rate indicate that the heating rate has a significant influence on the crystallization process. As the heating rate is increased, the intensities of all DSC peaks increase and they shift to higher temperatures, indicating thermal activation of the observed steps of the crystallization process. The ratios of peak intensities and the intervals between the peaks change with the increasing heating rate, as the activation energies of individual crystallization steps are different.

The overall activation energy of different crystallization steps, as well as the pre-exponential factors under linear heating conditions, were determined by the Kissinger’s, as well as the Ozawa’s peak methods, based on the dependence of exothermic peak temperature \(T_p\) on heating rate \(\beta\) [13,14]. Table 1. When compared to our previous study of this system, conducted using Kissinger’s and Ozawa’s methods on unresolved peaks [10], the values obtained for resolved peaks are slightly lower, but they are within the range of experimental error of those data. The resolution of peaks, however, resulted in much lower experimental error, when compared to analysis of unresolved peaks.

The high values of the apparent activation energy of crystallization of amorphous alloy indicate primarily the high complexity of these processes, as well as the fact that a lot of atoms are involved in an elementary step of structural reorganization. The errors were determined as a root-square deviation multiplied on Student’s coefficient for the probability of 0.95.

#### 3.2. Determination of kinetic triplets

In order to establish kinetic description of the crystallization process, we applied “the model free”, as well as “the model fitting” method. The fraction extent of reaction, \(\alpha\), at any temperature, \(T\), for all crystallization steps was obtained as \(\alpha = S/T\), where \(S\) is the total area of the exothermic curve between the temperature \(T_s\), where the crystallization is just beginning, and the temperature \(T_c\), where the crystallization is completed. \(S\) is the area between the initial...
temperature and a generic temperature, $T$, ranging between $T_i$ and $T_f$.

### 3.2.1. Model free approach

The model-free approach, also known as “isoconversion method”, requires determination of the temperature $T_\alpha$, at which an equivalent stage of the reaction occurs for various heating rates. The widely accepted procedure, giving the influence of fractional extent of reaction, $\alpha$, on the values of kinetic parameters, is the Flynn–Wall–Ozawa’s (FWO) method \[15,16\] in the form:

$$\ln \beta = \ln \left( \frac{AE_{\alpha,\alpha}}{Rg(\alpha)} \right) - 1.0518 \frac{E_{\alpha,\alpha}}{RT_\alpha}$$

(5)

where $g(\alpha)$ is the integral form of the conversion function $f(\alpha)$, and defined as $g(\alpha) = \int (d\alpha)/(f(\alpha))$.

In accordance with Eq. (5), the dependence of $\ln \beta$ on $1/T$ allows us to determine apparent activation energy, even without the knowledge of the correct conversion function. The values of the apparent activation energies, calculated from the slope of this dependence, and intercepts, are shown in Fig. 2 as a function of $\ln A$.

### 3.2.2. Model fitting approach

Taking into account that the first two crystallization steps can be treated as single step processes, we used model fitting method, applying the Coats–Redfern method [18] for different forms of conversion functions for solid state reactions, to determine the kinetic triplets for the first two crystallization steps. The criterion for choosing the right conversion function was the linearity correlation factor, $R^2$, of dependence $f(\alpha)=f(1/T)$ at different heating rates for individual steps, Table 2.

Although all correlation coefficients $R^2$ (Table 2) are very close to 1, the Arrhenius parameters for applied heating rates are highly variable, exhibiting strong dependence on the selected conversion function. This means that, under non-isothermal conditions, $\alpha=f(T)$ curves and Coats–Redfern’s method do not permit us to determine the true kinetic parameters as well as the correct conversion function. This is due to the fact that kinetic curves contain information about the temperature and conversion components in non-separate form. In order to find the true conversion functions for first and second step of crystallization, we have chosen the values of apparent activation energies determined using Kissinger’s method and applied some additional criteria, such as the independence of the activation parameters on the heating rate [19], master plot method [20] and analysis of two new functions defined by Málek [21–23].

In order to determine which of the conversion functions presented in Table 2 best corresponds to the first and the second crystallization step, we first applied the criterion of the independence of activation parameters on the heating rates on the conversion functions. According to this criterion [19], for the correct proposed conversion function, the following should be true:

$$\ln \left( \frac{g_\alpha(x)}{T^2} \right) = \ln \left( \frac{AR}{E_a} \right) - \frac{E_a}{RT}$$

(6)

where $g_\alpha(x)$ is the integral form of kinetic model, defined as:

$$g_\alpha(x) = \int_0^\alpha \frac{d\alpha}{f(\alpha)} - \frac{ZE_a}{R^2} p(x); p(x) = \int_0^\alpha \frac{\exp(-x)}{x^2} dx, x = \frac{E_a}{RT}$$

Using Eq. (6) for different conversion functions, Table 2, we tested the applicability of the criterion of the independence of acti-

### Table 1

<table>
<thead>
<tr>
<th>Overall values</th>
<th>Step 1</th>
<th>Step 2</th>
<th>Step 3</th>
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<tbody>
<tr>
<td>$E_a$ [kJ mol⁻¹]</td>
<td>$K$issinger</td>
<td>Ozawa</td>
<td>$K$issinger</td>
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<tr>
<td>$\ln A$</td>
<td>375.1 ± 0.8</td>
<td>388.7 ± 0.8</td>
<td>341.6 ± 0.5</td>
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<tr>
<td>$R$</td>
<td>0.999</td>
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Fig. 2. The resolved DSC peaks for heating rates (a) 5 K min⁻¹ and (b) 10 K min⁻¹.
Dependence of apparent activation energies $E_a$ (a) and intercepts (b) on the conversion degree for different crystallization steps. As a result, we obtained satisfactory linearity only for JMA model in the form $g(\alpha) = -\ln(1-\alpha)^{1/n}$ where $n = 3$ for both steps of crystallization occurring as single-step processes is the most satisfactory.

Further verification of the applicability of this conversion function was conducted using Málek’s [21–23] and master plot [20] methods. In order to apply Malek's method, we tested our experimental data using two special functions, $y(\alpha)$ and $z(\alpha)$. For JMA model, $y(\alpha)$ and $z(\alpha)$ functions have a convex shape, the maximum values being designated as $\alpha^*_y$ and $\alpha^*_z$, respectively. For practical reasons these functions are usually normalized within $[0,1]$ range. In non-isothermal conditions, these functions are

<table>
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<tr>
<td>Conversion function</td>
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<td>Peak 1</td>
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<td>P3</td>
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<td>F1</td>
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Fig. 3. Dependence of apparent activation energies $E_a$ (a) and intercepts (b) on the conversion degree for different crystallization steps.
Fig. 4. Independence of activation parameters on the heating rates for different forms of JMA model, for the first and second crystallization peak.

The “fingerprint” of JMA model is that the value of $\alpha^*_z = 0.632$ and that the value of $\alpha^*_y$ is always lower than the value of $\alpha^*_z$, under the assumption that the crystal growth occurs only after the nucleation has been finished. The convex shape of the obtained normalized functions $y(\alpha)$ and $z(\alpha)$ (Fig. 5), and their independence on the heating rate $\beta$, show that the proposed conversion function is correct for both crystallization peaks. Both functions exhibit well defined maxima located at an exactly defined values of $\alpha$. However, the values of $\alpha^*_z$ are not 0.632, but 0.5, and the values of $\alpha^*_y$ are higher, at 0.6, instead of lower than the values of $\alpha^*_z$. As JMA model [24,25] can be applied to non-isothermal conditions only under the assumption that the nucleation is completed before the further crystalline growth, the deviation from the model exhibited in our system indicates that this condition is not met. However, the correct shape of the curves indicates that JMA model approximates the reaction mechanism to a very good degree.

Having determined the values of apparent activation energies and the conversion functions for crystallization steps 1 and 2, the experimental data were reconstructed numerically by applying the “master plot” method [20]. According to this method, for a single-step process, the following equation is easily derived from Eq. (2), using a reference point at $\alpha = 0.5$:

$$\frac{d\alpha}{dt} \left( \frac{d\alpha}{dt} \right)_{\alpha = 0.5} = \frac{f(\alpha)}{f(0.5)}$$

where $f(0.5)$ is constant for the selected conversion function. Eq. (9) means that, for selected $\alpha$, the experimentally determined value of the reduced-generalized reaction rate in the form $(d\alpha/dt)/(d\alpha/dt_{\alpha=0.5}) \exp(Ea/RT)/(\exp(Ea/RT_{0.5}))$ and theoretically calculated value of $f(\alpha)/f(0.5)$ are equal when the correct conversion function, $f(\alpha)$, is applied. Fig. 6 shows theoretical master plots of $f(\alpha)/f(0.5)$ versus $\alpha$, using selected $f(\alpha)$ functions listed in Table 2, together with experimental plots $(d\alpha/dt)/(d\alpha/dt_{\alpha=0.5})\exp(Ea/RT)/(\exp(Ea/RT_{0.5}))$ for crystallization steps 1 and 2.

The best agreement of the theoretical master curves with the experimental master curves was achieved using $g(\alpha) = [-\ln(1 - \alpha)]^{1/n}$, where $n = 3$. The model is a very good approximation for the parts of the curves corresponding to lower $\alpha$, while parts of the curves corresponding to higher $\alpha$ show a discrepancy...
Fig. 5. The normalized functions $y(\alpha)$ and $z(\alpha)$ for peaks 1 and 2.

3.3. Correlation of established kinetic triplets and structural transformations

Considering the results presented above, we investigated the possible reasons for the deviation of the experimental curves from the theoretical model in the second part of the reaction in detailed correlation of the structural transformations with the conditions inherent in the application of JMA model. The system fits the model well in the first half of the reaction, indicating that primary nucleation is completed before the start of the crystal growth. If nucleation is the cause of this deviation from JMA model, then it must be secondary nucleation, starting at the point in the reaction where the system begins to deviate from the model. This secondary nucleation would proceed in parallel with crystal growth, invalidating the necessary condition for successful application of JMA model. The validity of the JMA equation can be extended to non-isothermal conditions if the entire nucleation process takes place during the early stages of the transformation and becomes negligible afterwards [26,27].

According to our results [10], the first two peaks in DSC correspond to the formation of stable crystal phases $\alpha$-Fe(Si) and Fe$_2$B, while the third could be attributed to recrystallization of these phases and further growth of crystallites. Using this data, we can elucidate the following sequence of crystallization: first to crystallize is $\alpha$-Fe(Si) phase, followed by B$_2$Fe$_{15}$Si$_3$ and Fe$_2$B phase. In addition, metastable B$_2$Fe$_{15}$Si$_3$ phase subsequently decomposes to yield the two stable crystalline phases, $\alpha$-Fe(Si) and Fe$_2$B.

The first peak in the DSC curve would correspond to formation of crystalline $\alpha$-Fe(Si) in the amorphous matrix, since it is the first to be observed in the XRD spectra (after heating at 723 K). The crystal structure of $\alpha$-Fe(Si) can accommodate for some of the boron, which destabilizes its lattice and acts as an inhibitor to the crystallization process, causing this reaction to have the highest activation energy and the lowest reaction rate (Table 1). B$_2$Fe$_{15}$Si$_3$ stoichiometrically corresponds to $2 \times Fe_3B$ plus $3 \times Fe_3Si$, and contains only 2.29 mass% of boron, compared to 13% in the as-prepared alloy, but 8.9 mass% of Si, compared to 8% in the as-prepared alloy. All this indicates that B$_2$Fe$_{15}$Si$_3$ would probably form around the clusters of short-range crystalline ordering, as those regions probably con-
tain the least amount of amorphizing boron. If this is the case, then its enthalpy of formation would be very low, making it very hard to notice in DSC (Fig. 1).

The second peak in DSC would correspond to crystallization of Fe₂B. This phase contains around 8.8 mass% of boron, compared to 1% in the as-prepared alloy. The formation of this crystalline phase would increase the concentration of boron in the amorphous matrix, making the nucleation of Fe₂B in the amorphous matrix less favorable, meaning that it probably initially occurs through decomposition of the metastable B₂Fe₁₅Si₃ phase. The stoichiometry of B₂Fe₁₅Si₃ phase indicates that it would probably decompose to Fe₂Si and Fe₂B, where Fe₂B could then easily be converted to Fe₂B by the excess boron in the amorphous matrix. This reaction would lower activation energy (Table 1), as it would decrease the amount of boron in the surrounding amorphous matrix, and it would create two types of nuclei: one for Fe₂B phase and the other, through Fe₂Si, for α-Fe(Si) phase, providing a source of secondary nucleation for α-Fe(Si).

The third peak in the DSC, which could not be described as a single step process, would correspond to the processes of recrystallization and growth which are observed after the samples have been heated at higher temperatures, as α-Fe(Si) phase gradually gives way to Fe₂B. In this process, Fe₂B nucleates in α-Fe(Si) crystalline matrix, which is probably caused by segregation of boron and Si in α-Fe(Si), where Si-poor regions would allow for nucleation of Fe₂B. This process, as shown by a step-like growth of Fe₂B phase content in XRD spectra, is probably highly dependent on the local composition and lattice structure and the nucleation would happen gradually as favorable conditions are met in any particular area of the sample. This would explain the lowest activation energy of the process (Table 2) and its unusual dynamics (Fig. 5), as nucleation and crystal growth would occur continuously and in parallel, rather than separately and consecutively. In addition, this process would provide secondary nucleation sites for further crystallization of Fe₂B phase. The secondary nucleation for both phases would proceed in parallel with the crystal growth that occurs after the primary nucleation and would invalidate the condition for validity of JMA equation in non-isothermal systems. This would explain the good agreement of the experimental curves with theoretical JMA curves in the first part of the reaction and their subsequent divergence, as the latter part would correspond to the region of secondary nucleation (Fig. 6).

4. Conclusions

Amorphous Fe₇₅Ni₂Si₈B₁₃C₂ alloy undergoes multi-step structural transformations, after annealing in 790–860 K temperature range, exhibiting two asymmetric overlapping exo-maxima in DSC, which were resolved. With the increase in the heating rate, the positions of these peaks shifted to higher temperatures. The process of phase transformation involves formation of stable iron-silicon (α-Fe(Si)) and iron–boron (Fe₂B) phases, and metastable iron–silicon–boron (B₂Fe₁₅Si₃). The primary crystallization starts with formation of α-Fe(Si) phase in the amorphous matrix, followed by metastable B₂Fe₁₅Si₃ and the iron–boron (Fe₂B) phase at temperatures above 780 K. The asymmetric peaks were resolved giving three well-formed peaks corresponding to different steps of the phase transformation. The kinetic parameters corresponding to each step were evaluated and kinetic triplets for the first two crystallization steps were determined, yielding the conversion function in the form

\[ g(x) = -\ln(1 - x)^{1/3} \]

\[ E₁ = 375.1 \pm 0.8 \text{ kJ/mol and } lnA = 56.2 \pm 1.0 \text{ for step 1} \]

\[ E₂ = 341.6 \pm 0.5 \text{ kJ/mol and } lnA = 49.3 \pm 0.5 \text{ for step 2} \]

Structural transformation of the alloy. This established kinetic model was confirmed by application of independence of activation parameters on the heating rates criterion and master plot method, using the established kinetic triplets. We have established that the divergence between experimental and theoretical JMA curves is the result of secondary nucleation processes for both stable crystalline phases, which occurs in parallel with crystal growth. In addition, the complexity of continuous process of crystal growth and nucleation of Fe₂B phase out of α-Fe(Si) matrix, which corresponds to step 3 of the structural transformation of the alloy, makes it impossible to describe as a single step process and, in consequence, to determine its kinetic triplet.

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