

Crystallization Kinetics in Amorphous $\text{Fe}_{68.8}\text{Cu}_{0.5}\text{Nb}_{2.6}\text{Si}_{18.6}\text{B}_{9.5}$ Alloy_B

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Abstract—The crystallization kinetics of amorphous $\text{Fe}_{68.8}\text{Cu}_{0.5}\text{Nb}_{2.6}\text{Si}_{18.6}\text{B}_{9.5}$ alloy has been carried out using Differential Scanning Calorimetry (DSC) technique. The alloy showed two stage crystallization. The activation energy has been calculated using Kissinger, Matusita-Sakka and Augis-Bennet methods and the average value is found to be equal to 206 kJ/mol and 623 kJ/mol for the first and second crystallization peaks, respectively. Calculation of activation energy using other methods is also discussed.

Keywords: crystallization, amorphous alloy, X-ray diffraction, differential scanning calorimetry, rapid quenching, activation energy, frequency factor, kinetic studies.

1. INTRODUCTION

Amorphous alloys of magnetic nature produced by rapid quenching of melt of iron rich alloys, have been of scientific and technological interest in the recent past. Even though these alloys lack long-range crystalline order, they show ferromagnetic properties. They represent a metastable state and tend to show structural relaxation with time which gets accelerated at higher temperature. At high enough temperatures, they crystallize irreversibly in a more stable state. Fundamental interest in multi component alloys has been revived due to nano-structure formation in these alloys [1]. Thus, the Fe-based multi component amorphous alloys are known to exhibit soft magnetic properties and hence, are important for many technological applications. These properties include very high saturation magnetization ($\sim 1.3\text{T}$), low coercivity H_c

($\sim 0.5\text{Am}^{-1}$), low saturation magnetostriction ($\sim 2 \times 10^{-6}$) and high effective permeability μ ($\sim 10^5$). Fe-based Nanocrystalline alloys are used in several commercial applications including power devices, power electronics, telecommunications, information handling and magnetic sensors. They are also indispensable in many applications in magnetic parts and devices such as inductors, low and high energy frequency transformers, motors, generators and sensors. It is found that the substitution of Cu for Fe plays an important role for nanocrystalline structure and excellent soft magnetic properties. Thus, the kinetics of crystallization plays an important role in physics, chemistry, ceramic and

metallurgical sciences. Thermal analysis methods, including DSC, are extensively used for studying kinetics of chemical reactions and crystallization of these alloys. The determination of basic data on crystallization kinetics is important in establishing the mechanism of crystal nucleation and growth [1-6]. The study of crystallization kinetics of these alloys gives useful insight about their thermal stability. In this paper, the crystallization kinetics of amorphous $\text{Fe}_{68.8}\text{Cu}_{0.5}\text{Nb}_{2.6}\text{Si}_{18.6}\text{B}_{9.5}$ alloy using Differential Scanning Calorimetry (DSC) technique is reported.

2. EXPERIMENTAL

Amorphous ribbon of $\text{Fe}_{68.8}\text{Cu}_{0.5}\text{Nb}_{2.6}\text{Si}_{18.6}\text{B}_{9.5}$ alloy having thickness of about $30\mu\text{m}$ and width of 5mm produced by single roller melt spinning was procured from our other group of Researchers. The amorphous nature of the ribbon was confirmed by X-Ray Diffraction (XRD). The thermal behavior of the alloy was investigated by high temperature differential scanning calorimetry (DSC) conducted at different heating rates of $5^\circ\text{C}/\text{min}$, $10^\circ\text{C}/\text{min}$, $15^\circ\text{C}/\text{min}$ and $20^\circ\text{C}/\text{min}$ in the temperature range 300 K – 1000 K.

3. RESULTS AND DISCUSSION

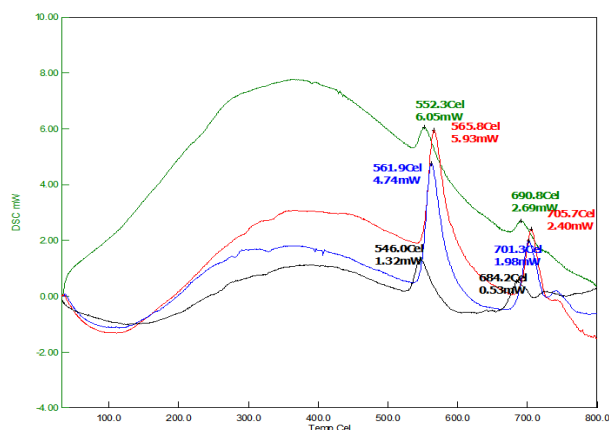


Fig. 1: DSC thermograms of amorphous $\text{Fe}_{68.8}\text{Cu}_{0.5}\text{Nb}_{2.6}\text{Si}_{18.6}\text{B}_{9.5}$ alloy at different heating rates of $5^\circ\text{C}/\text{min}$ (Black), $10^\circ\text{C}/\text{min}$ (Green), $15^\circ\text{C}/\text{min}$ (Blue) and $20^\circ\text{C}/\text{min}$ (Red).

The DSC curves of fresh (as-quenched) samples at different heating rates of 5^oC/min(Black), 10^oC/min (Green), 15^oC/min (Blue) and 20^oC/min (Red) show two-step crystallization as shown in Fig. 1.

The activation energy (E_c) for crystallization of an amorphous alloy under a linear heating rate (non-isothermal) is calculated using Masutika-Sakka, Augis-Bennet and Kissinger methods [2, 3, 4], which relates the peak temperature (T_p) with heating rate (α) using the relations.

$$\ln \alpha = -[E_c/(RT_p)] + \text{Constant} \quad (1)$$

$$\ln (\alpha/T_p) = -[E_c/(RT_p)] + \text{Constant} \quad \dots(2)$$

$$\ln (\alpha/T_p^2) = \text{Constant} - [E_c/(RT_p)] \quad \dots\dots(3)$$

where 'R' is Gas constant.

Table 1: Heating rate, First peak and Second peak temperatures of amorphous Fe_{68.8} Cu_{0.5}Nb_{2.6}Si_{18.6}B_{9.5} Alloy

S. No.	Heating Rate (oC/min)	First peak temperature (oC/min)	Second peak temperature (oC/min)
1	5	546.0	684.2
2	10	552.3	690.8
3.	15	561.9	701.3
4.	20	565.8	705.7

Table.1 shows the heating rate, first peak and second peak temperatures of amorphous Fe_{68.8} Cu_{0.5}Nb_{2.6}Si_{18.6}B_{9.5} Alloy.

Table 2: Activation energy of crystallization (E_c), in kJ/mol, of amorphous Fe_{68.8} Cu_{0.5}Nb_{2.6}Si_{18.6}B_{9.5} Alloy calculated from non-isothermal methods.

	Matusita-Sakka's Method	Augis-Bennet's Method	Kissinger's Method	Average Value
Peak 1	194	207	218	206
Peak 2	591	618	660	623

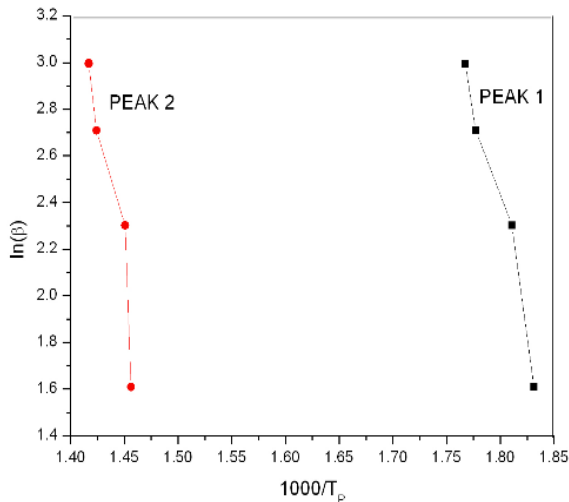


Fig. 2: Matusita sakka plot for amorphous Fe_{68.8}Cu_{0.5}Nb_{2.6}Si_{18.6}B_{9.5} alloy

The values of E_c obtained for the present sample using the above three methods are given in Table 2. Comparison of the E_c values obtained for different non-isothermal methods shows that the values are in good agreement with each other. This means that one can use any of the three methods to calculate the activation energy of crystallization.

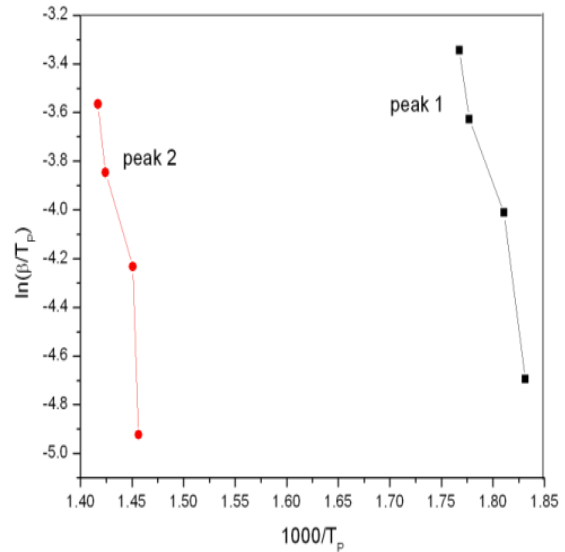


Fig. 3: Augis bennett plot for amorphous Fe_{68.8}Cu_{0.5}Nb_{2.6}Si_{18.6}B_{9.5} alloy

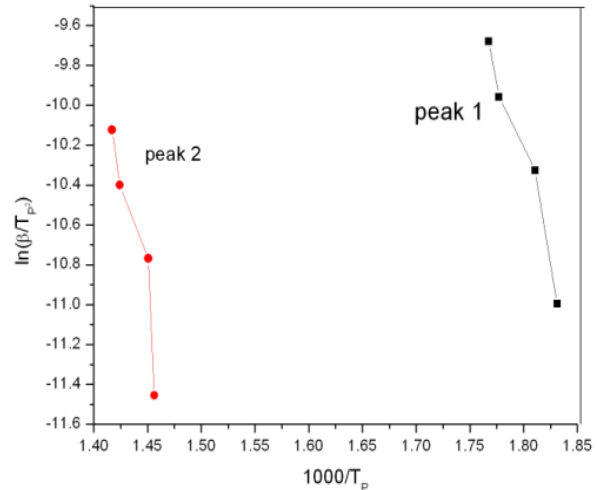


Fig. 4: Kissinger plot for amorphous Fe_{68.8}Cu_{0.5}Nb_{2.6}Si_{18.6}B_{9.5} alloy

Figs. 2, 3 and 4 show the plots of Masutika-Sakka, Augis-Bennet and Kissinger methods for amorphous Fe_{68.8}Cu_{0.5}Nb_{2.6}Si_{18.6}B_{9.5} alloy. Also, Fig. 1 reveals that the alloy undergoes two stage crystallization reactions with wide temperature interval between two crystallization stages. Observation of two step crystallization is more common in metallic glasses contained more than three elements than in

those containing less components. The first broad peak in the present case arises due to (1) Structural relaxation occurring with a broad range of relaxation times due to a variety of atomic rearrangement and (2) Formation of intermediate metastable phases. The second peak arises due to crystallization as in glass which is a defined nucleation and growth reaction. Broadening also occurs due to kinetic reasons as the kinetics involving structural processes slows down due to the disappearance of free volume during relaxation processes.

4. CONCLUSIONS

Amorphous $\text{Fe}_{68.8}\text{Cu}_{0.5}\text{Nb}_{2.6}\text{Si}_{18.6}\text{B}_{9.5}$ alloy showed two –stage crystallization. Thus, the study of the crystallization kinetics in the present sample has been studied under non-isothermal conditions. The DSC technique has been used in the present study to calculate the activation energy of crystallization (E_c). The ' E_c ' values evaluated for the two stages are in agreement with other reported values of the similar compositions.

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