CRYSTALLIZATION KINETICS OF AMORPHOUS Fe\textsubscript{75-x}Cu\textsubscript{x}Si\textsubscript{9}B\textsubscript{16}

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Abstract

In this work the influence of Cu admixtures on the crystallization process of amorphous Fe–Si–B alloys is studied, based on measurements of differential thermal calorimetry of the series Fe\textsubscript{75-x}Cu\textsubscript{x}Si\textsubscript{9}B\textsubscript{16} (x = 0, 1, 2, 2.8 and 3.5) during their heating with different heating rates. The first crystallization stage can not be traced for any of the amounts of Cu content examined, while the second stage is observed only when the Cu content is 1 at%. The activation energy as estimated with Kissinger’s method for the third crystallization stage has a mean value of 326 kJ mol\textsuperscript{-1} and with the isoconversional Flynn, Wall and Ozawa method is almost constant when 0.05 < \alpha < 0.6 and exhibits a small monotical decrease when \alpha > 0.6. The main crystallization peak can not be described by means of a single JMA-type function.

Keywords: activation energy, crystallization, DSC, Fe–Si–B

Introduction

Many studies have been carried out during the last decade, aiming at the amelioration of the soft magnetic properties of the amorphous Fe–Si–B alloys. The tools used towards this aim are mainly the thermal treatment of the said alloys and their enrichment with various admixtures. A typical example is the preparation of nanocrystalline materials with stoichiometry Fe\textsubscript{74}Cu\textsubscript{1}Nb\textsubscript{3}Si\textsubscript{9}B\textsubscript{13} [1], having much better soft magnetic properties than the corresponding ones of the amorphous materials.

This work focuses on the influence of Cu admixtures on the kinematics of the crystallization of the amorphous Fe–Cu–Si–B alloys. In our previous works [2, 3], based on measurements of the saturation magnetization, electrical resistance and electron microscopy (TEM) of the alloy series Fe\textsubscript{75-x}Cu\textsubscript{x}Si\textsubscript{9}B\textsubscript{16} (x = 0, 1, 2, 2.8 and 3.5) and on the work of other researchers as regards the crystallization of Fe–Si–B alloys, the following conclusions were arrived at:

The crystallization of the amorphous alloys Fe\textsubscript{75-x}Cu\textsubscript{x}Si\textsubscript{9}B\textsubscript{16} is completed in three stages, corresponding to the growth of bcc Fe[Si,Cu], then of bct Fe\textsubscript{3}B and finally to the disintegration of the latter into bcc Fe and bct Fe\textsubscript{2}B. The final product of the crystallization consists of 52% Fe\textsubscript{83-x}Cu\textsubscript{x}Si\textsubscript{17} (x = 0/52) and 48% Fe\textsubscript{2}B. The presence of Cu admix-
tures in the amorphous material causes microsegregation, leading to an increase of the crystallization rate. As the ratio of the number of B atoms to the number of Fe atoms increases, the crystallization of bct Fe₃B is favored and that of bcc Fe is hindered.

The aim of the present work is to validate and supplement the above conclusions by measurements of differential scanning calorimetry (DSC), so that a more complete picture of the crystallization procedure of the alloys under investigation can be drawn.

Experimental procedure

Four amorphous ribbons with stoichiometry Fe₇₅₋ₓCuₓSi₉B₁₆ (x = 0, 1, 2, 2.8 and 3.5) were prepared in a melt-spinning machine (single-roll quenching technique), the alloys having been prepared in an arc-melting apparatus, from 3N+ purity elements. The composition of the alloys was verified, as regards the relative ratios of Fe, Cu and Si, by SEM. Regarding B, its atomic percentage was set from the mass of B added to form the alloy.

The thermal behaviour of Fe₇₅₋ₓCuₓSi₉B₁₆ was studied using Setaram DSC-131. Temperature and energy calibrations of the instrument were performed using the well-known melting temperatures and melting enthalpies of high purity zinc and indium supplied with the instrument. Ribbon-shaped specimens weighing about 7 mg, cut into small pieces were crimped in stainless steel crucibles, an empty stainless steel crucible was used as reference. A constant flow of nitrogen was maintained in order to provide a constant thermal blanket within the DSC cell, thus eliminating thermal gradients, and ensuring the validity of the applied calibration standard from sample to sample.

A series of non-isothermal DSC experiments was carried out on the Fe₇₅₋ₓCuₓSi₉B₁₆ ribbons with heating rates in the range 3–12.5 K min⁻¹.

Results and discussion

Figure 1 shows, for example, the DSC curves of the rapidly quenched Fe₇₅₋ₓCuₓSi₉B₁₆ alloys containing 0, 1 and 2 at% Cu obtained with a heating rate of 3 K min⁻¹. None of the observed anomalies was reproduced in the subsequent measuring run on the crystallized samples. In comparing these figures, we notice that there is a distinct difference in the

![Fig. 1 DSC linear heating curves of Fe₇₅₋ₓCuₓSi₉B₁₆ with heating rate 3 K min⁻¹](image)

1 – 0 at% Cu, 2 – 1 at% Cu, 3 – 2 at% Cu