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In-situ resistance measurement and Joule heating set-up for Structural transformation

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Abstract

Joule heating set-up, using which in-situ resistance can be measured is developed to evidence the structural transformation occurring within amorphous $Fe_{73.5}Si_{13.5}B_9Cu_1Nb_3$ alloy. It is demonstrated that there exists a minimum value of the current, which induces nanocrystallization within the specimen, below which structural transformation does not takes place irrespective of annealing time. Information about the ordering within the studied specimen can be extracted effectively by monitoring in-situ resistance during Joule heating.

INTRODUCTION & SET-UP DETAILS

Thermal treatments are known to modify the structure and properties of the metastable systems leading to significant improvement of the application oriented physical properties [1]. DC Joule heating technique [2] allows the crystallization of amorphous ribbons to occur in shorter times as compared with conventional thermal annealing, by passing the current through the sample. Nano-crystalline FINEMET-type alloys [3] exhibit extremely good soft magnetic properties. However, a major hindrance in using these materials for various applications arises from their inherent brittleness, induced during the development of the nanocrystalline phase. This feature is detrimental in prospective applications of nanocrystalline ferromagnetic ribbons, as wound transformer cores. Joule heating technique allows one to obtain nanocrystalline materials with improved mechanical properties [4]. The dc Joule heating set-up developed by us allows one to measure in-situ electrical resistance to follow temperature variations within sample and to evidence the structural transformations occurring within a material. The heat flow within a metallic ribbon is given by:

$$\chi \frac{\partial^2 T}{\partial x^2} - \frac{I\sigma_T}{S}\frac{\partial T}{\partial x} + \frac{I^2\rho}{S^2} - f(x,T) = C\frac{\partial T}{\partial t} \quad \textbf{(1)}$$

where x-distance along the sample axis ; χ-thermal conductivity, I - electrical current, S - sample cross section, σ_T – Thomson coefficient and C - specific heat , ρ-the electrical resistivity, T – temperature of the material.

Thermal losses are taken into account by the term *f(x,T)* that can be only temperature dependent e.g. - the present case of ribbons with surface to volume ratio ~ 10^4 m^{-1} suggests that the dominant loss mechanism are the radiative effects which are independent of *x*. Heat repre-sented by first derivative term in eq. (1) is negligible as compared to the Joule heat term (~ $(I/S)^2$). Thermal losses are attributable to the conductive, radiative as well as conv-ective contributions, as sample is kept in air. In the present article we describe a Joule heating set-up, using which in-situ resistance can be measured to evidence the structural transformations occurring within amorphous alloys. Figure. 1 shows the block diagram of the set-up developed and used for the measurement of in-situ resistance and also for Joule heating of amorphous alloys in air. Measurements were done on amorphous $Fe_{73.5}Si_{13.5}B_9Cu_1Nb_3$ alloy (~20 μm thick, 5 mm wide and 10 cm long). A computer controlled dc supply (0 – 5 A) was used to flow the current through the samples. Computer controlled digital multimeters were used to measure current, voltage and temperature during the experiments. Resistance of the sample (during Joule heating) was measured by using four-probe method. Structural characterization of the studied samples was done using Cu-k_α x-ray diffraction (XRD).

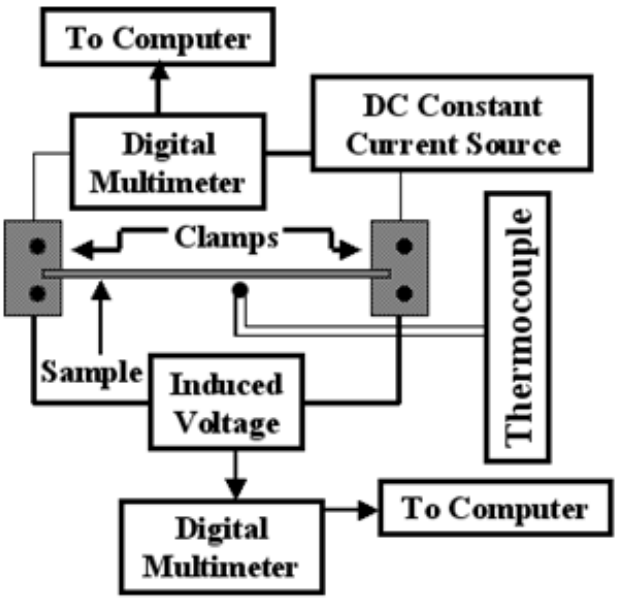

Figure 1: Block diagram of the set-up.

ILLUSTRATIVE MEASUREMENTS & DISCUSSION

In order to carefully evaluate the electrical current value, which induces the amorphous-to-crystalline transformation, one sample has been submitted to a current ramp and the electrical resistance was continuously monitored. The electrical resistance evolution as a function of electrical current passing through the sample and the current ramp (increase of current with time) is shown in figure 2.

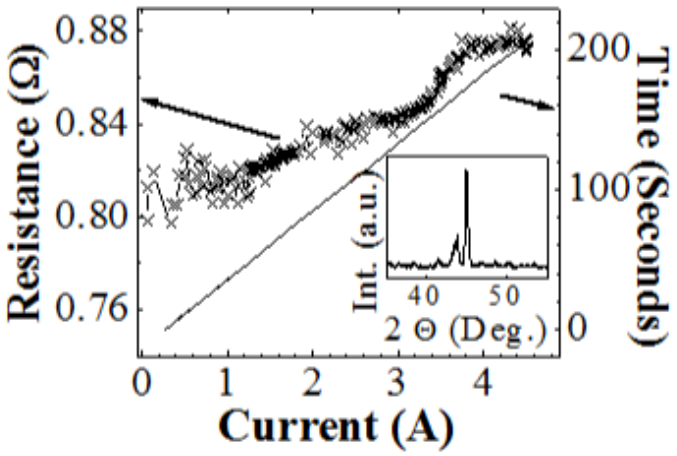

Figure 2: Resistance behavior as a function of Current. Inset: XRD of the studied sample.

Perusal of figure 2 shows that after an increase of resistance (attributable to the increase of the sample temperature) up to ≈ 4.4 A, a decrease of resistance is observed clearly indicating the onset of micro-structural changes within the amorphous matrix (i. e. sample crystallization), as was also observed earlier [5]. The measured temperature corres-ponding to 4.5 A is ≈ 540 ^{o}C, which is above the crysta-llization temperature of the studied specimen [3], so one would expect crystallization within the sample. Inset of figure 2 shows the XRD of corresponding specimen, and as expected the sample exhibits co-existence of the crystalline phase and the residual amorphous matrix. Influence of Joule heating current (I_a) and time (t_a) on evolution of resistance is depicted in figure 3 (a and b). Perusal of fig. 3 (a) shows that the resistance of the sample (Joule heated with 0.8 A for 1 h) almost remains constant throughout the treatment, indicating that no structural change has taken place. Moreover, temperature of the specimen throughout the experiment was below 40 ^{o}C, which is far below the crystallization temperature of the sample, thus no structural change is expected. XRD (inset of figure 3 a) confirming the result obtained by resistance measurements, that the sample is still amorphous. Perusal figure 3 (b) shows that the resistance of the sample (Joule heated with 4.0 A for 12min.) increases (attributable to the increase of the sample temperature), decreases a bit (attributable to the onset of micro-structural changes within the amorphous matrix) remains constant for some time then increases up to a steady state as was also observed earlier [5].

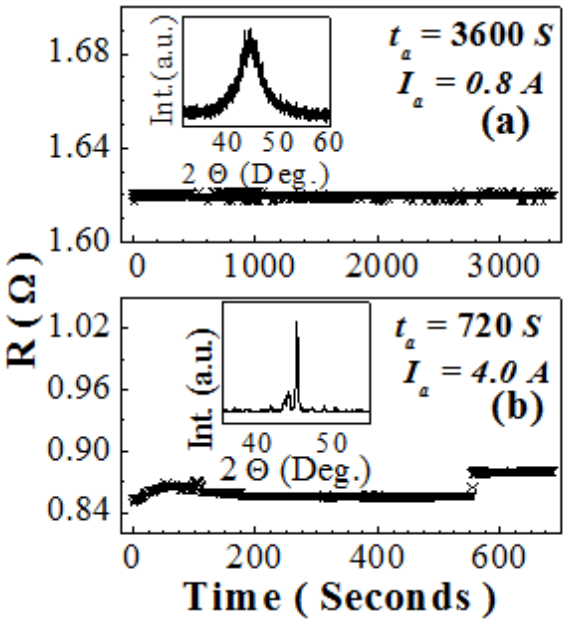

Figure 3: Resistance / time, for (a) I_a = 0.4 A, (b) I_a = 4.0 A. Inset: XRD data.

It should be noted that the temperature of the specimen during the treatment has reached up to 530 ^{0}C, which is above the crystallization temperature of the specimen [3]; hence one expects crystallization of the specimen. XRD (inset of figure 3 b) confirms the result obtained by resistance measurements, exhibiting co-existence of the crystalline phase as well as amorphous phase. Figure 3 clearly demonstrates that there exists a minimum value of the current, below which structural transformation does not take place irrespective of t_a. Observed lower value of resistance for the sample treated with I_a = 4.0 A as compared with that annealed using I_a = 0.8 A can be attributed to the increasing order due to the crystallization.

In conclusion, in-situ resistance measurement and Joule heating set-up is developed and used to study structural transformation in amorphous $Fe_{73.5}Si_{13.5}B_9Cu_1Nb_3$ alloy. Results provide information about the current needed to induce nanocrystallization, ordering within the specimen.

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