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NUCLEATION EFFECTS DURING CRYSTALLIZATION IN DISORDERED METASTABLE METALLIC SYSTEMS

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The study of some new methods for investigation of amorphous and nanocrystalline aluminium based alloys will be presented. Nanocrystalline alloys obtained by crystallization from amorphous rapidly quenched Al-Rare Earth-Transition Metal systems were investigated. Noise analyses technique was used for investigation of these systems. The main visible activity in this field is low frequency noise, also called 1/*f* noise. Changes in noise manifestation during measurements, which can contribute to characterization of materials, were observed.

Keywords: crystallization, 1/f noise, nucleation rate

1 INTRODUCTION

New results in investigation of amorphous and nanocrystalline alloys are presented. Quantitative thermodynamic analyses (TEM, XRD, *etc*), routinely performed to explain behavior of the system during nanocrystallization, are complemented by a new experimental method aimed at monitoring selected physical quantity – electrical noise in this case - which would directly correspond to the nucleation rate of forming nanocrystalline particles from amorphous material.

In this study two types of measurements of electrical noise are presented. The first is the measurement of low frequency (1/f) noise, which depends on voltage drop of the sample *U*, frequency *f* and a material constant *C*. Its Voltage Spectral Density (VSD) is given by relation [1]:

$$Sv \approx C \cdot \frac{U^2}{f} \qquad (1)$$

In general 1/f noise is observed when system is driven out from equilibrium. This noise can be observed with introducing external force (electrical current, magnetic field, *etc)* into the system.

Different amorphous systems were measured to find characteristics of measured samples in low frequency band (Fig. 1). The correlation between 1/*f* noise and material composition and atomic arrangements was investigated. In this work the $Fe_{60}Co_{20}Si_5B_{15}$ and $(Fe_0Co_1)_{73}Nb_7Si_5B_{15}$ systems are presented.

The second part of research effort is to investigate the possibility of using noise analysis for direct experimental measurement of nucleation rate during annealing (nanocrystallization). When nanometer-size particles (grains) appear in the structure of amorphous material, a change of some physical quantities (*eg* electrical resistivity) of measured material is observed. These changes are reflected also in noise manifestation. Thermodynamic measurements on $Al_{90}Fe_7Nb_3$ system were performed.

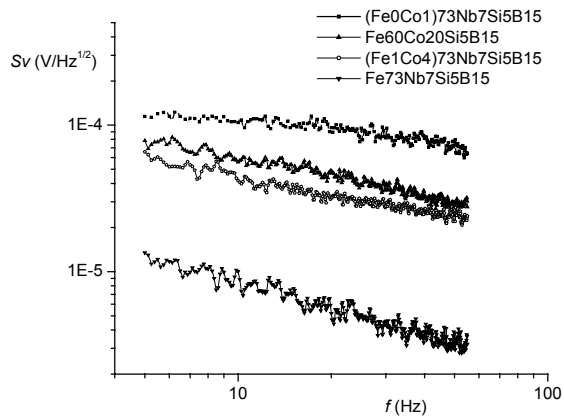

Fig. 1. 1/*f* noise curves for various metallic systems

2 EXPERIMENTAL

Measurement of noise involve both theoretical assumes, which were described in introduction, and experimental setup. Dealing with very small signals, the apparatus part for noise measurements has some special requirements to be fulfilled to gain the required sensitivity. Those involve not only the sensing part to have good noise performance, but also the whole measuring system to be at maximum restricted from external influence, which can affect on the measurements results.

A schematic low frequency noise measurement setup used for noise investigation in amorphous and nannocrystalline metallic systems is shown in Fig. 2.

It consist of three basic parts: sample under test, low noise preamplifier with dc current supply, and external spectrum analyzer.

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Measurements were performed on as-cast amorphous ribbon samples.

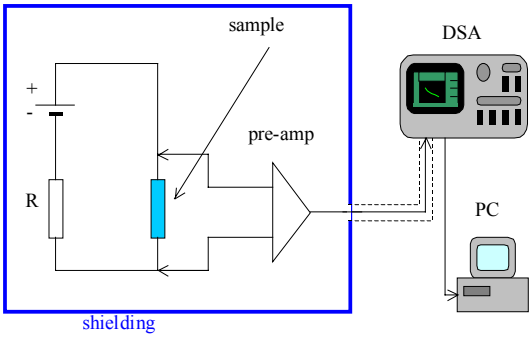

Fig. 2. Measurement setup for 1/*f* noise analyses

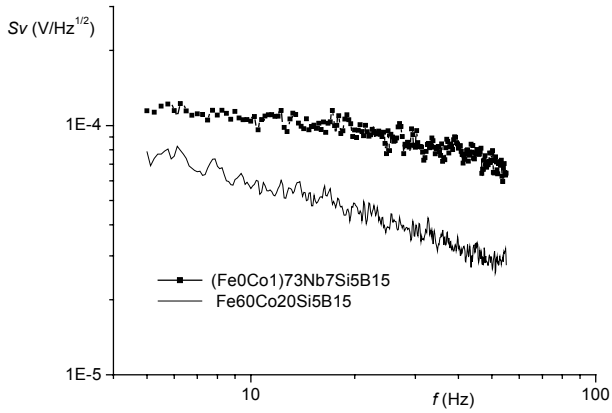

Fig. 3. Low-frequency (1/*f*) noise of $Fe_{60}Co_{20}Si_5B_{15}$ and $(Fe_0Co_1)_{73}Nb_7Si_5B_{15}$ systems

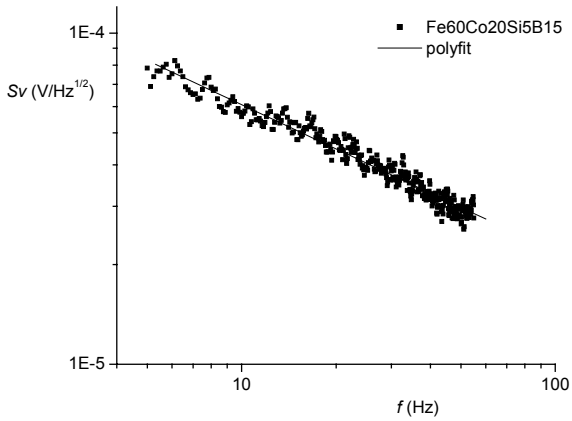

Fig. 4. Polynomial fitting of VSD of $Fe_{60}Co_{20}Si_5B_{15}$ system

Voltage drop on the sample generated by direct electrical current passing through the sample was measured. This current generates 1/*f* noise, VSD of which was measured with dynamic signal analyzer (SR785) after amplification by low-noise preamplifier (SR560).

Sample, electrical current source and preamplifier were located in a shielding box.

Noise analysis of $Fe_{60}Co_{20}Si_5B_{15}$ and $(Fe_0Co_1)_{73}Nb_7Si_5B_{15}$ systems were performed with this measuring setup. Fig. 3 shows VSD dependence of 1/*f* noise on the samples measured under the same conditions (voltage drop, amplification).

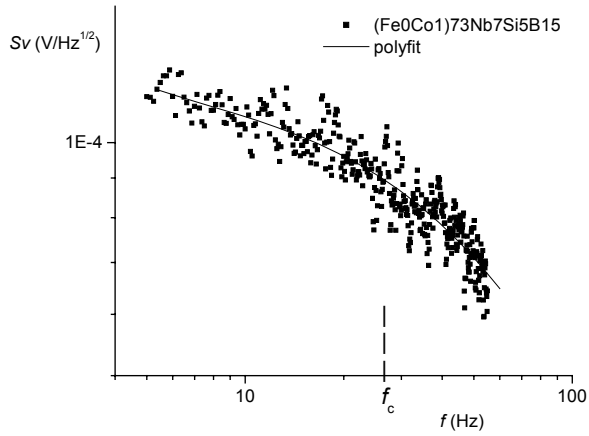

Fig. 5. Polynomial fitting of VSD of $(Fe_0Co_1)_{73}Nb_7Si_5B_{15}$ system

It is clear from the VSD curves that the lower value of noise voltage corresponds to the $Fe_{60}Co_{20}Si_5B_{15}$ system (Fig. 4). More interesting is the fact that on the VSD of $(Fe_0Co_1)_{73}Nb_7Si_5B_{15}$ system (Fig. 5) a "corner frequency" f_c can be observed, where the frequency dependence of VSD changes. Differences in VDSs can be explained by the corresponding increase of the electron flux, whereas the decrease of the current in the Fe-Co-Si-B sample can be a consequence of the lower number of states available for the conduction [2]. This depends on the material composition.

Thermodynamic measurements were performed on $Al_{90}Fe_7Nb_3$ ribbon samples, where nucleation-and-growth process starts at about 480 K [3, 4].

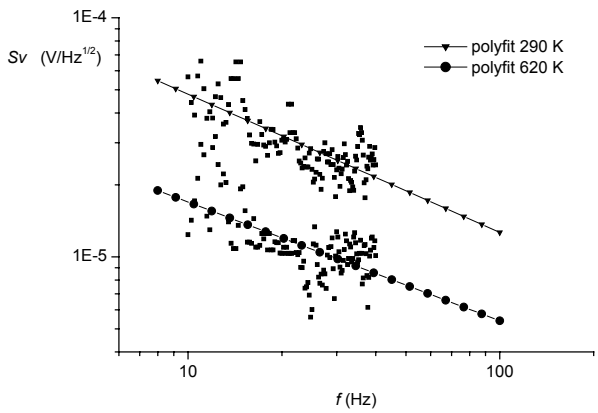

Fig. 6. VSD of $Al_{90}Fe_7Nb_3$ system during annealing

Voltage drop on the sample generated by electrical current passing through the sample isothermally annealed at 620 K in the furnace was measured. The VSD of $Al_{90}Fe_7Nb_3$ system during annealing of the sample decreased systematically with proceeding crystallization,

as indicated in Fig. 6. More detailed analysis of the data obtained is under development.

3 CONCLUSIONS

Noise analyses of the amorphous and nanocrystalline metallic systems by the low noise measurements has some contributions to the complex analyses of these materials. This technique, new in our materials research, provides a new possibility of material characterization.

When a system is driven out from thermodynamic equilibrium, some quantities, *eg* electrical current, should exhibit a $1/f$ behaviour in frequency domain. Differences between VSD of various materials depend on material composition and structure. Measurement setup with sufficient external influence resistance offers $1/f$ noise measurement, which can be used for detection of changes in material structure.

Noise and thermodynamic measurements on $Fe_{60}Co_{20}Si_5B_{15}$, $(Fe_0Co_1)_{73}Nb_7Si_5B_{15}$ and $Al_{90}Fe_7Nb_3$ systems were preformed. Changes in VSD of $Fe_{60}Co_{20}Si_5B_{15}$ and $(Fe_0Co_1)_{73}Nb_7Si_5B_{15}$ systems which can contribute to characterization of materials were observed.

Changes in noise manifestations were detected during crystallization of $Al_{90}Fe_7Nb_3$ system, which can correspond to nucleation process in the material.

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