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# Magnetic Nanoparticles of $(Co_{0.94}Fe_{0.06})_{72.5}Si_{12.5}B_{15}$ and $Fe_{78}Si_9B_{13}$ obtained by electric explosion of amorphous wires

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**.Abstract.** Magnetic nanoparticles (MNPs) were produced by the electric explosion of wire method (EEW) using $(Co_{0.94}Fe_{0.06})_{72.5}Si_{12.5}B_{15}$ and $Fe_{78}Si_9B_{13}$ amorphous wires. The wires were exploded in Ar atmosphere at 0.12 MPa. After the explosion the surface of all produced nanoparticles was passivated with oxygen. The produced MNPs are spherical low aggregated particles with average size below 37 nm and rather narrow size distributions with geometric standard deviation less than 1.7. Both materials are multiphase and contain up to 23 wt % of amorphous phase.

## Introduction

Magnetic nanoparticles (MNPs) attract interest because of their technological and biomedical applications [1]. Different techniques are used for the MNPs synthesis. Electrical explosion of wire (EEW) is one of the promising physical methods for the MNP production [2]. Usually wires with polycrystalline structure are used for the production of nanopowders. It's known that the structure parameters of a wire affect the particles size. Thus according to the results obtained in [3] the reduction of the grain size of the wire leads to the reduction of particle sizes. In this connection the explosion of a wire without crystal structure is of interest. In this work we present the results of the synthesis of magnetic nanoparticles (MNPs) by EEW from the amorphous wires made of Co and Fe alloys with Si and B additives. The structure, morphological features and magnetic properties were studied by different techniques.

## Experimental

**Preparation of amorphous wires**. The wires with the elemental compositions $(Co_{0.94}Fe_{0.06})_{72.5}Si_{12.5}B_{15}$ (0.22 mm in diameter) and $Fe_{78}Si_9B_{13}$ (0.14 mm in diameter) were prepared by the "in-water-rotating-spinning" method described elsewhere [4,5]. For that purpose, master alloys of both compositions were prepared using an arc melting furnace. The master alloy was placed into a quartz crucible and melted using the induction method. By means of overpressure the melted material was ejected through the orifice of the quartz crucible and quenched into the water layer at the surface of the rotating wheel.

**Synthesis of MNPs by EEW**. EEW evaporation of the amorphous wires and the synthesis of MNPs were performed in the Institute of Electrophysics, Ekaterinburg, RF. The setup of the apparatus and principle of its functioning is described in [2]. The electrical explosions were made in the closed explosion chamber in argon under the pressure 0.12 MPa. Several portions of amorphous wire (100 mm in length) were simultaneously evaporated by the high-voltage pulse to obtain necessary quantity of MNPs for the attestation and the study of magnetic characteristics. The high-voltage current pulse applied to the parallel wires was supplied by the capacitor C charged to the voltage $U_0$. EEW parameters for the synthesis of both types of MNPs are given in Table 1.

Table 1. Parameters of EEW for the evaporation of amorphous Co and Fe wires.

| Material | $U_0$, kV | C, μF | Number of wires | Number of explosions |
|---|---|---|---|---|
| $Fe_{78}Si_9B_{13}$ | 30 | 3.2 | 6 | 4 |
| $(Co_{0.94}Fe_{0.06})_{72.5}Si_{12.5}B_{15}$ | 34 | 4.8 | 3 | 15 |

The elaborated in the EEW values of the capacitance C, charging voltage and the number of simultaneously evaporated wires provide the overheating coefficient K=2. ($K=W/W_s$ is the ratio of the electrical energy applied to the wire to the energy of sublimation of the evaporated metal). After each explosion the obtained MNPs were passivated by the slow and controlled addition of oxygen to the working gas. As a result on the surface of MNPs an oxide protective layer was formed, which prevented their further oxidation. The thickness of the oxide shell was 1.5 – 3 nm according to TEM.

**Methods of characterization**. TEM and HTEM images were obtained by JEOL JEM2100 microscope operating at 200 KV. The X-ray diffraction (XRD) studies were performed by DISCOVER D8 (Bruker) diffractometer using Cu- K$\alpha$ radiation ($\lambda$ = 1.5418 Å), with a graphite monochromator. Bruker software TOPAS-3 with Rietveld full-profile refinement was employed for the quantitative analysis of all the diffractograms. Magnetic measurements were performed by vibrating sample magnetometer. Microwave absorption as a function of the applied dc field (H) was studied by homodyne detection and a half-wavelength cavity [6] operating at 8.91 GHz. Two kinds of signals were observed: conventional ferromagnetic resonance (FMR) when $H \perp h_{rf}$ ($h_{rf}$ – microwave field) and zero-field absorption which reduces monotonically upon the application of $H \parallel h_{rf}$. Zero field microwave loss in conducting MNPs is caused by a large dynamic permeability [6].

## Results and discussion

Fig. 1 presents TEM images for CoFeSiB and FeSiB MNPs. The particles are spherical and low aggregated. The number particle size distributions for both nanopowders were determined by the image analysis of more than 1000 particles. PSD are lognormal (Fig. 1 insets) with the parameters given in Table 2. Reflexes clearly visible on electronic diffraction patterns (Fig. 1) of both powders indicate the presence of crystalline phases. The fragments of crystalline lattice are seen in the high resolution TEM images (Fig. 2A). The particles are covered by the oxide protective layer Fig. 2B), which was created by the controlled oxidation during the synthesis. Phase compositions of the MNPs are given in Table 3.

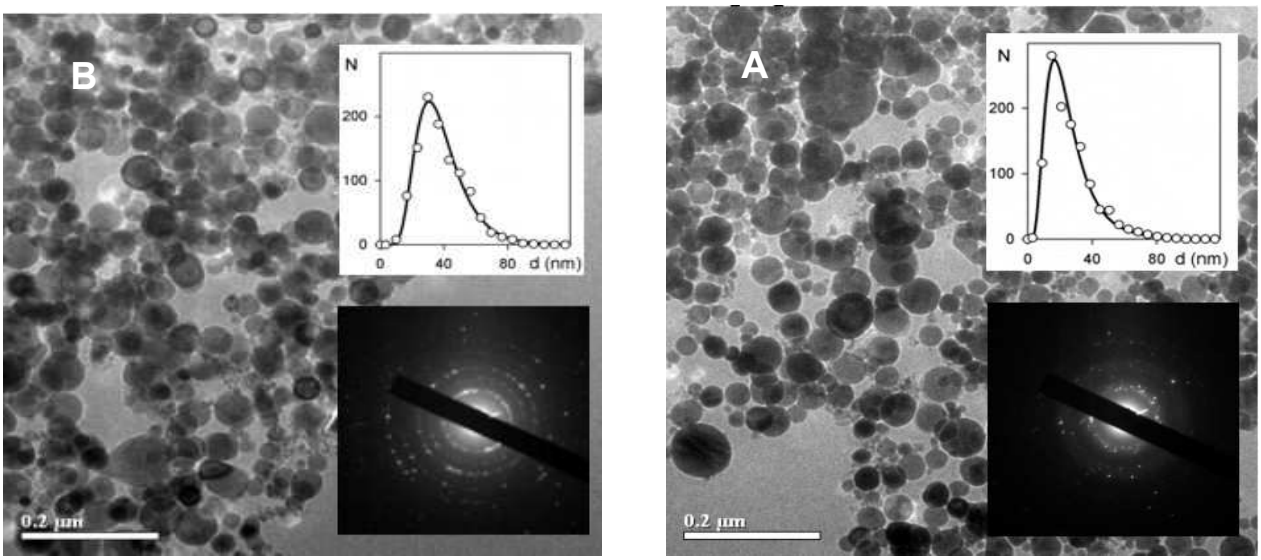


Fig. 1. TEM images of MNPs synthesized by EEW from amorphous wires. (A) – CoFeSiB, (B) – FeSiB. Insets present PSD plots and electronic diffraction patterns.

Table 2. PSD parameters of MNPs

| Sample | Average size, nm | Max diameter, nm | Min diameter, nm | Median diameter, nm | Geometric standard deviation (GSD) | Number of particles |
|---|---|---|---|---|---|---|
| CoFeSiB | 26.0 | 115.9 | 2.9 | 22.6 | 1.7 | 1190 |
| FeSiB | 37.9 | 132.7 | 3.3 | 35.1 | 1.48 | 1092 |

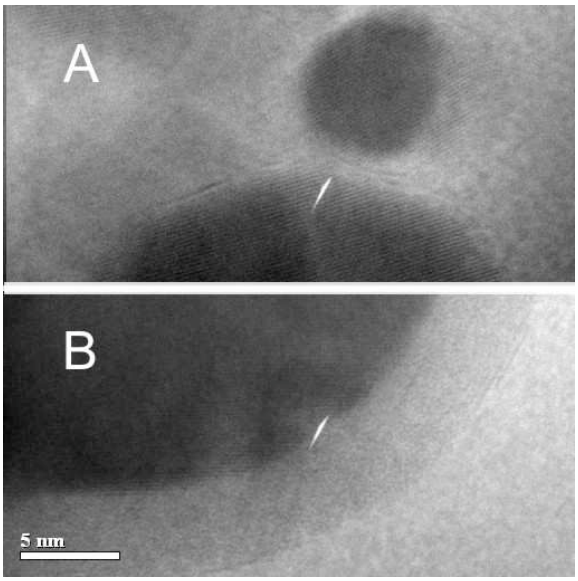


Fig. 2. HTEM images of MNPs. (A) – fragments of crystalline structure in CoFeSiB MNPs. (B) Protective oxide layer on the surface of FeSiB MNPs

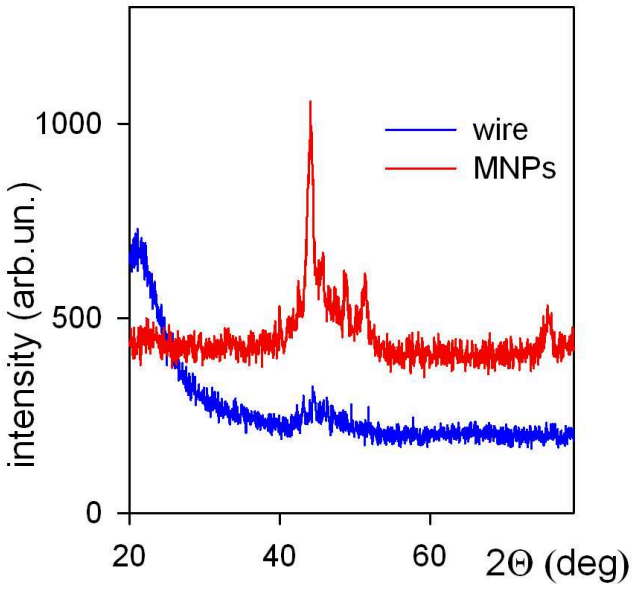


Fig. 3. XRD patterns for CoFeSiB wire and MNPs

Table 3. Crystalline phases in MNPs.

| CoFeSiB | | | | FeSiB | | | |
|---|---|---|---|---|---|---|---|
| Phase | Content, wt.% | Lattice period, Å | Grain size, nm | Phase | Content, wt.% | Lattice period, Å | Grain size, nm |
| Co (cubic) | 29.2 | 3.55(3) | 12(2) | αFe | 38.5 | 2,864(3) | 28(3) |
| $Co_5Si_2B$ | 26.1 | 8.50(2) | 9 | γFe | 10.8 | 3,904(8) | 100 |
| $Co_3B$ | 17.4 | 5,09(2) | 14 | $Fe_3Si$ | 10.0 | 5,665(8) | 23(3) |
| $Co_2Si$ | 4.7 | 4,94(3) | 48 | $Fe_2B$ | 9.2 | 5,127(8) | 28(3) |
| γFe | 1.6 | 3,89(1) | 120 | $Fe_{23}B_6$ | 8.5 | 10,80(8) | 13 |
| Amorphous | 21 | - | - | Amorphous | 23 | - | - |

CoFeSiB and FeSiB MNPs have several crystalline phases in composition. All of them have rather small grain size except γ-Fe. After the evaporation of the wire in the result of EEW and subsequent condensation the crystallization of multiple phases takes place. At the same time the substantial fraction of the material (20%) remained amorphous (Table 3). Calculation of the composition of the amorphous phase for both MNPs revealed that it includes all the elements of the exploded amorphous wires with the enlarged content of B and Si.

Fig.4 shows results of magnetic and microwave studies. FeSiB EEW products show quite high saturation magnetization of about 130 emu/g of which about 85 emu/g corresponds to a pure αFe (Table 3). CoFeSiB EEW products show lower saturation magnetization of about 65 emu/g of which 48 emu/g corresponds to Co MNPs. If we recalculate the saturation magnetization for the weight of the magnetic products only, it looks quite consistent with the structural data (Table 3).

FMR peaks are very wide (of the order of 2 kOe in both cases) as to expect for multicomponent spherical MNPs. Due to a field-dependent non-resonant absorption which overlap the FMR, precise measurements of resonance fields and linewidths were not possible. At the same time approximate line position is consistent with the presence of Co MNPs in the case of CoFeSiB. Zero field absorption is judge and observed microwave behaviour is not surprising for the phase composition of fabricated MNPs. Unfortunately obtained small amounts of the material did not allowed to make phase separation and study such an interesting phases as $Fe_{23}B_6$ or amorphous part of the vapours. Application of an external magnetic field parallel to the microwave magnetic field causes zero field absorption to become negligible at the fields above 5 kOe. This behavior may be invoked to propose a new type of the composite as microwave absorbers with improved properties.

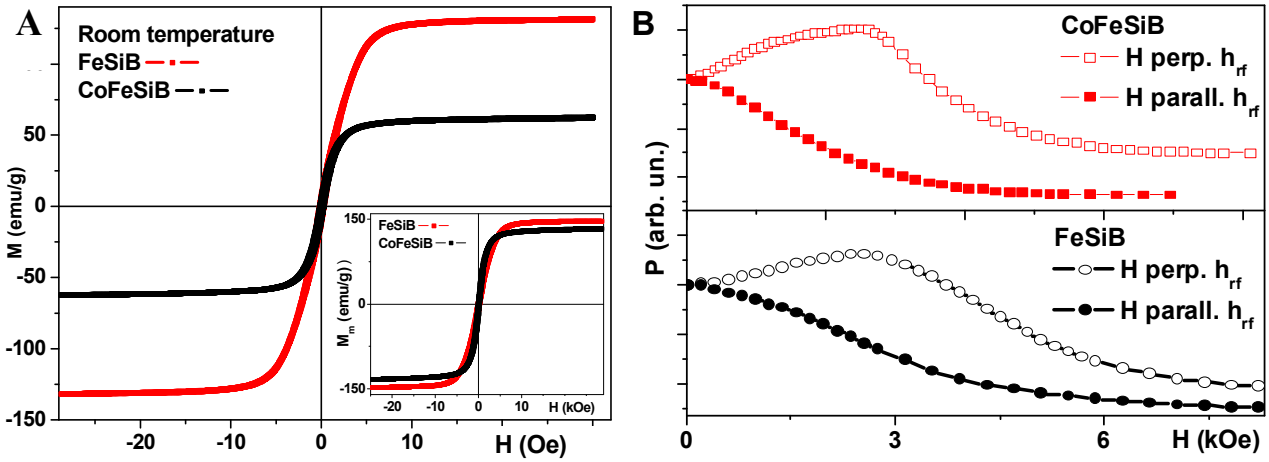


Fig. 4. Hysteresis loops for MNPs obtained from the amorphous wires (compositions are indicated for the wires); magnetization is calculated either per unit mass or per unit of magnetic component (inset) (Table 3 data) (A). Microwave losses of FeSiB and CoFeSiB EEW products measured in FMR and non-resonant absorption configurations.

## Summary

The possibility of production of nanoparticles by EEW using wire in the amorphous state has been shown. The MNPs synthesized by EEW are multiphased and contain about 20 % of amorphous phase which includes all the elements of the exploded amorphous wires with the enlarged content of B and Si. The magnetic measurements of FeSiB and CoFeSiB EEW products were consistent with the results of the structural studies. Microwave behavior of the obtained materials may be invoked to propose a new type of the composite as microwave absorbers with improved properties. Additional studies of such interesting phases as $Fe_{23}B_6$ and separation of the amorphous components are the directions of future research.

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