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# Structure and properties of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy powders bound with polyethylene*

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The paper presents results of merging the thermal and mechanical nanocrystallisation processes of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy, and further examination of the structure, as well as the magnetic and mechanical properties of powders obtained in this way and also of composites developed by their binding with polyethylene (PEHD). The composites were obtained in the one-sided uniaxial pressing at the temperature of 170 $^oC$ under the pressure of 350 MPa.

## 1. INTRODUCTION

The nanocrystalline soft magnetic materials based on Fe are manufactured most often by mechanical synthesis (mechanical nanocrystallization) and fast cooling of the liquid with the subsequent controlled crystallization (thermal nanocrystallization) [1-3], because of the advantageous connection of the relatively low manufacturing costs and good mechanical and physical properties. The main disadvantages of these methods are their limitations connected with the geometrical form of the produced nanomaterials (powder or thin strip), restricting significantly the range of their applications. To increase the application range of nanomaterials obtained using these methods they are merged with other materials, yielding composite materials in this way. In this way bigger products with complex shapes can be made. Thermosetting or chemosetting polymers are used most often for making composites; their volume fraction does not exceed 20 % [4-8].

The goal of this work is merging the thermal and mechanical nanocrystallization processes of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ metallic glass and investigation of structure and properties of the powders and composites obtained by their binding with the polyethylene (PEHD) in the one-sided uniaxial pressing.

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## 2. EXPERIMENTAL PROCEDURE

Amorphous strips of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ as quenched metallic glass 0.04 mm thick and 7 mm wide were used for investigations. The strips were heat treated by holding at the temperature of 550 $^oC$ for 1 h in the argon atmosphere (thermal nanocrystallization). Phases: crystalline αFe(Si) and the amorphous intergranular one - developed as a result of the primary crystallization, are the products of heat treatment of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ metallic glass strips [9].

Powders of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy after its thermal nanocrystallization were made by the high-energy grinding of the initially crumbled strips in the shaker type 8000 SPEX CertiPrep Mixer/Mill for 15 minutes, 1 hour, 3 hours, and for 5 hours in the air (mechanical nanocrystallization).

The powders obtained in this way were used to make the composite. The high density low-pressure polyethylene (PEHD) powder was used as binder – with mass fractions of 2.5 %, 5.0 % and 7.5 %. The components were mixed in the shaker type 8000 SPEX CertiPrep Mixer/Mill. Mixing time was determined experimentally and was 20 minutes. The composites were one-sided, uniaxially pressed at the temperature of 170 $^oC$ under the pressure of 350 MPa for 15 minutes in the air. Specimens were obtained by pressing: toroidal with the O.D. D = 25 mm, I.D. d = 17 mm, and height h = 5 mm for magnetic properties tests, and cylindrical ones with diameter d = 17 mm and height h = 25 mm for compression strength tests.

Tests of the magnetic properties of the nanocrystalline powders and composites were carried out on cores with the O.D. 25 mm, I.D. 20 mm on FERROMETR-1 unit, consisting of the PC computer with the Advantech (PCL – 812 PG) PC-Lab data acquisition card and a software package controlling the system.

Structure changes accompanying the thermal and mechanical nanocrystallization processes were examined on the DRON-2 X-ray diffractometer with the HZG-3 goniometer and computerized reflected radiation recording system, equipped with the cobalt anode lamp, powered by current with 40 kV voltage and with 20 mA heater current. Recording of the investigated diffraction lines was made by the step method in the angular range of 40÷120 $^o$. Counting time at the measurement point was 5 s.

OPTON DSM 940 scanning electron microscope was used for examination of powders with the 500x magnification.

Compression strength of composites was determined on Instron 1195 universal testing machine.

## 3. EXPERIMENTAL RESULTS AND THEIR DISCUSSION

X-ray examinations show that the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy initial, as quenched structure is amorphous, which is revealed on the diffraction pattern as the wide-angle peak coming from the amorphous phase (Fig. 1A). Heat treatment at the temperature of 550 $^oC$ causes crystallization of the amorphous matrix , which is testified by peaks coming from the αFe(Si) phase (Fig. 1B).

No widening of the diffraction lines or decrease of their intensity were observed after the strip grinding process following their thermal nanocrystallization and pressing to make composites (Fig. 2).

A) B)

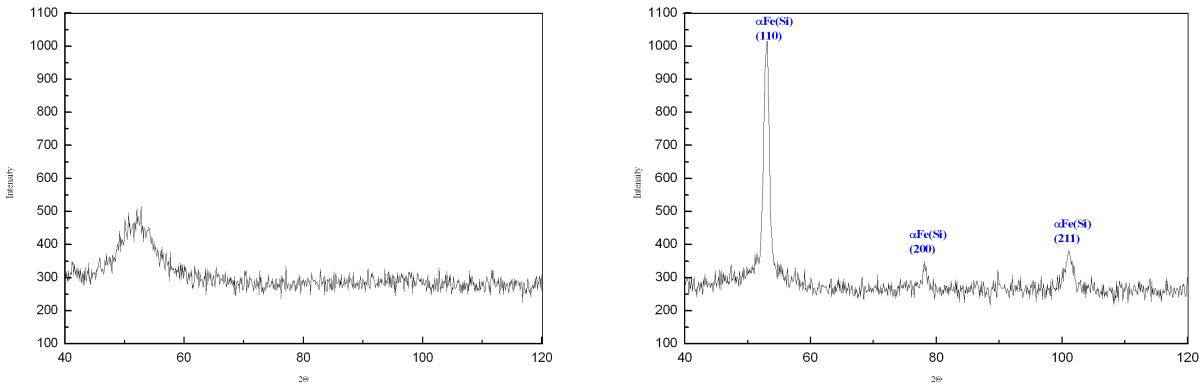


Fig. 1. X-ray diffraction patterns of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy
A) in the initial as quenched state, B) after heat treatment – 550 °C/1 h

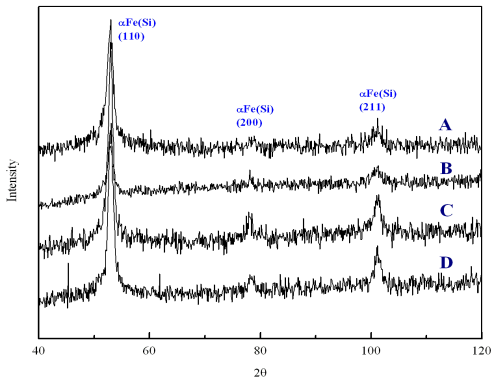


Fig. 2. X-ray diffraction patterns of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy powders:
A) thermal nanocrystallization 550°C/1h + mechanical nanocrystallization, grinding 5h
B) thermal nanocrystallization 550°C/1h + mechanical nanocrystallization, grinding 3h
C) thermal nanocrystallization 550°C/1h + mechanical nanocrystallization, grinding 1h
D) thermal nanocrystallization 550 °C/1 h + mechanical nanocrystallization, grinding 0.25 h

Observation of shapes and dimensions of powder particles, depending on strip grinding time (mechanical nanocrystallization) after its thermal nanocrystallization revealed that along with the grinding time extension powder grain size and its standard deviation decrease. The biggest grains with the average size of 136.10 μm are characteristic for powder obtained by grinding the strips for 0.25 h. The smallest grains with the average size of 35.12 μm are demonstrated by powder obtained by grinding the strips for 5 h. This testifies to the progressing homogenizing of their sizes, albeit their spread remains rather big (Fig. 3, Table 1).

Table 1
Grain size of powders used as a component in the investigated composites

| Grain size | Strip grinding time [h] | | | |
|---|---|---|---|---|
| | 0.25 | 1 | 3 | 5 |
| average [μm] | 136.10 | 85.40 | 43.39 | 35.12 |
| maximum [μm] | 200.75 | 107.33 | 76.75 | 59.05 |
| minimum [μm] | 85.75 | 60.44 | 40.68 | 29.72 |
| standard deviation | 89.07 | 52.05 | 43.39 | 30.25 |

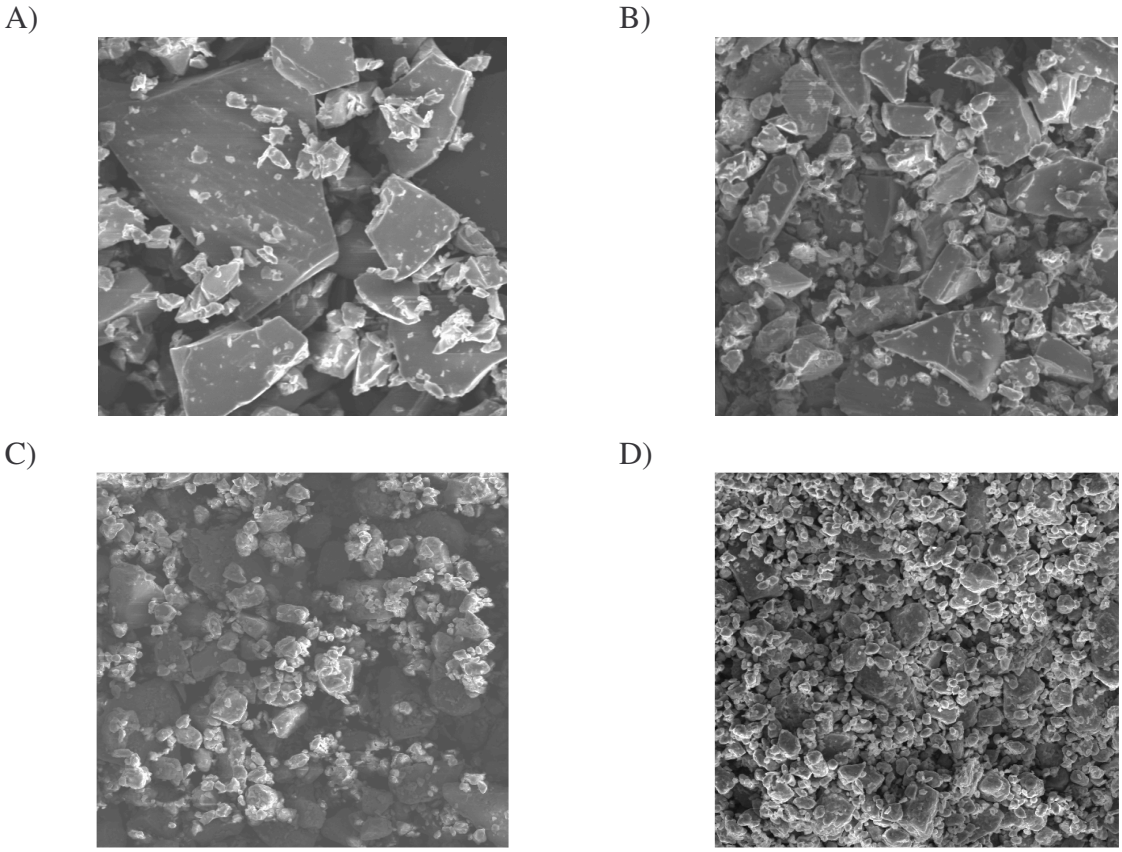


Fig. 3. Forms and sizes of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy powder particles after its thermal nanocrystallization: A) after 15 minutes of strip grinding, B) after 1 hour of strip grinding, C) after 3 hours of strip grinding, D) after 5 hours of strip grinding, magnification 500x

Test results of the magnetic properties of the source strips and composites are presented in Table 2. Coercion $H_c$ for the as quenched strips from the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy is 36.68 A/m, remanence $B_r$ 0.315 T; whereas, its magnetic permeability $\mu$ is 7678 and power loss is 1.55 W/kg. Magnetic properties of strips from the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy improve after its thermal nanocrystallization and are: coercion $H_c$ 8.42 A/m, remanence $B_r$ 0.927 T, magnetic permeability $\mu$ 168568, power loss 0.91 W/kg.

Magnetic properties of the investigated composites deteriorate in comparison with properties of the as quenched strips from the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy after its thermal crystallization at the temperature of 550 $^oC$ for 1 h. Coercion $H_c$ is from 70.44 A/m (PEHD composite 2.5 % + powder obtained from grinding the strips for 5 h) to 293.87 A/m (PEHD composite 5.0 % + powder obtained from grinding the strips for 0.25 h). Remanence $B_r$ values are in the range from 0.002 T for the PEHD composite 5 % + powder obtained from grinding the strips for 5 h to 0.01 T for the PEHD composite 2.5 % + powder obtained from grinding the strips for 0.25 h and the PEHD composite 7.5 % + powder obtained from grinding the strips for 0.25 h. Magnetic permeability $\mu$ changes from13 for the PEHD composite 2.5 % + powder obtained from grinding the strips for 5 h to 37 for the PEHD composite 5 % + powder obtained from grinding the strips for 0.25 h. Power loss $P_{max}$ is from 0.02 W/kg for the PEHD composite 2.5 % + powder from grinding the strips for 5 h to 0.09 for the PEHD composite 5% + powder from grinding the strips for 0.25 h.

Table 2
Magnetic properties of strips in the as quenched state, after thermal nanocrystallization, and of the obtained composites

| Composite type | | Size | | | | | |
|---|---|---|---|---|---|---|---|
| PEHD fraction in composite [% mas.] | Strip grinding time [h] | $H_c$ [A/m] | $B_r$ [T] | $B_{max}$ [T] | $H_{max}$ [A/m] | $\mu_{max}$ | $P_{max}$ [W/kg] |
| 2.5 | 0.25 | 161.37 | 0.01 | 0.11 | 2003.5 | 35 | 0.08 |
| | 1 | 106.02 | 0.004 | 0.06 | 1302.6 | 23 | 0.03 |
| | 3 | 186.94 | 0.006 | 0.08 | 1977.7 | 21 | 0.06 |
| | 5 | 70.44 | 0.003 | 0.05 | 1303.3 | 13 | 0.02 |
| 5.0 | 0.25 | 293.87 | 0.006 | 0.11 | 1974.7 | 37 | 0.09 |
| | 1 | 178.56 | 0.007 | 0.10 | 1974.6 | 33 | 0.03 |
| | 3 | 210.20 | 0.007 | 0.09 | 2042.7 | 25 | 0.07 |
| | 5 | 105.18 | 0.002 | 0.09 | 2044.4 | 26 | 0.08 |
| 7.5 | 0.25 | 254.27 | 0.01 | 0.09 | 2000.9 | 30 | 0.06 |
| | 1 | 137.68 | 0.004 | 0.09 | 1974.6 | 23 | 0.05 |
| | 3 | 153.28 | 0.006 | 0.07 | 2044.3 | 20 | 0.05 |
| | 5 | 167.06 | 0.008 | 0.09 | 1997.7 | 21 | 0.05 |
| Strip in the as quenched state | | 36.68 | 0.315 | 0.812 | 926.9 | 7678 | 1.55 |
| Strip after thermal nanocrystallization | | 8.42 | 0.927 | 1.279 | 712.9 | 168587 | 0.91 |

Results of the mechanical properties tests determined in the static compression test are shown in Fig. 4. The highest compression strength of 57.27 MPa is characteristic for the composite with powder obtained from grinding the strips for 3 h with the 7.5 % mass fraction of PEHD. The lowest compression strength of 26.65 MPa is characteristic for composite with powder obtained from grinding the strips for 0.25 h with the 2.5 % mass fraction of PEHD.

For composites with powder obtained from grinding the strips for 0.25 h compression strength is from 25.65 MPa for the PEHD mass fraction of 2.5 % to 50.25 MPa for the PEHD mass fraction of 5.0 %. For composites with powder obtained from grinding the strips for 1 h compression strength is from 53,41 MPa for the PEHD mass fraction of 2.5 % to 57.27 MPa for the PEHD mass fraction of 7.5 %. Values of compression strength for composites with powder obtained from grinding the strips for 3 h range from 43.57 MPa for the PEHD mass fraction of 2.5 % to 56.57 MPa for the PEHD mass fraction of 7.5 %. Composites with powder from grinding the strips for 5 h have compression strength in the range from 28.81 MPa for the PEHD mass fraction of 2.5 % to 54.47 MPa for the PEHD mass fraction of 5 %.

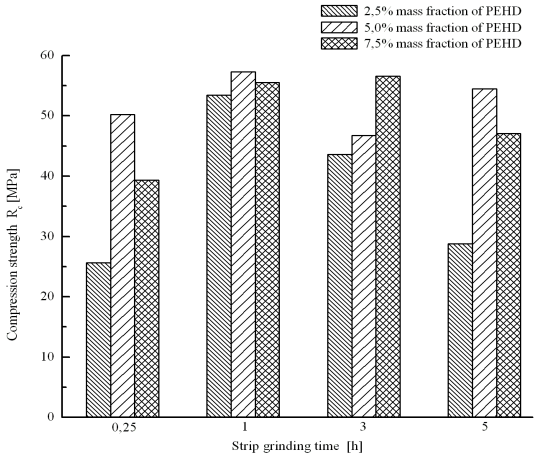


Fig. 4. Comparison of compression strength values of the investigated composites versus the PEHD mass fraction and strip grinding time

## 4. SUMMARY

It is possible to obtain powder with the nanocrystalline structure using a combination of the thermal nanocrystallization (holding strips from the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy in the as quenched state at the temperature of 550$^o$C for 1 h in argon atmosphere) and the mechanical one (high-energy grinding of strips from the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy after its thermal nanocrystallization).

Size of the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy powder particles depends on grinding time of strips after their thermal nanocrystallization. Homogenizing of their sizes improves with grinding time.

Magnetic properties of composites obtained from mixing the $Fe_{73,5}Cu_1Nb_3Si_{13,5}B_9$ alloy powders with PEHD deteriorate compared to the magnetic properties of strips after thermal nanocrystallization.

Mechanical properties determined in the static compression strength test depend on the PEHD mass fraction in the composite.

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