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Structure and properties of Fe-Co-Ni-B-Si-Nb alloy prepared by mechanical alloying method

W. Pilarczyk *,[a], R. Nowosielski [a], A. Pilarczyk [b]
[a] Division of Nanocrystalline and Functional Materials and Sustainable Pro-ecological Technologies, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland
[b] The Institute of Welding, ul. Bł. Czesława 16/18, 44-100 Gliwice, Poland
* Corresponding author: E-mail address: wirginia.pilarczyk@polsl.pl

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Materials

ABSTRACT

Purpose: The goal of this work is to investigate structure and properties of $Fe_{57.6}Co_{7.2}Ni_{7.2}B_{19.2}Si_{4.8}Nb_4$ powders alloys obtained by mechanical alloying.
Design/methodology/approach: The test material was the mixture of Fe, Co, Ni, B, Si and Nb powders obtained by the mixing in suitable weight relation. The powders were ground for the 10 and 100 hrs in a high energy planetary ball mill. The microscopic observation of the shape and size of the powdered material particles was carried out by the scanning electron microscope with the magnification 500 times. The changes of the powder structure were tested by means of the X-ray diffractometer. Powder samples by energy dispersion spectroscopy were analyzed too. The measurements of particles size by means of the laser analyser were carried out.
Findings: The present paper is the attempt at proposing the mechanical alloying method to obtain multicomponent, Fe-based nanocrystalline alloys.
Research limitations/implications: The experiments in this article are made on a laboratory scale.
Practical implications: The examined alloys belong to a modern group of soft magnetic materials, which can be used as transformers, sensors, power and electronics devices, etc.
Originality/value: In addition a good structural homogeneity and first of all mechanical properties was achieved, also practical application will be possible.
Keywords: Metallic alloys; Powder metallurgy; Mechanical alloying

1. Introduction

Up to now, the interest in fabrication of nanomaterials was focused on receiving and investigation of magnetic materials. The Fe-based multicomponent alloy systems are of great importance in industry. So essential importance of iron and its alloys results from the possibility of regulation of their structure and properties. The correct choice of chemical composition of the alloys as well as their purity have an impact on the desirable structure. The Fe-Co-Ni-Nb-Si-B alloys are materials exhibiting very good soft magnetic properties. It has been found that $[(Fe_{0.8}Co_{0.1}Ni_{0.1})_{0.75}B_{0.2}Si_{0.05}]_{94}Nb_4$ bulk glassy alloy exhibits super-high strength of over 4000 MPa and some ductile strain up to 0.005, combined with good soft magnetic properties [13]. It is well known that Fe-based nanocrystalline alloys are used in several commercial application including power devices, power electronics, telecommunications, information handling, magnetic sensors. They are indispensable in many applications in magnetic

Table 1.
Purity, particle size and chemical composition

Elements	Purity [%]	Granulation [mesh]	Granulation [μm]	mass. [%]	at. [%]
Fe	99	200	74	67	57.6
Co	99.5	325	44	9	7.2
Ni	99.8	325	44	9	7.2
B	99	325	44	4	19.2
Si	99	100	149	3	4.8
Nb	99.8	325	44	8	4

parts and devices such as inductors, low and high energy frequency transformers, alternating current machines, motors, generators and sensors [6, 7, 15].

The substitution of small amounts of Co or Ni for Fe in Fe-based magnetic materials generally results in an increase of saturation magnetization. The application of Ni favour the development of metastabile structures at low milling times. In addition, the increase of Ni content results in the higher permeability, too [1, 3]. It is known that addition of elemental niobium improves the mechanical properties. Unfortunately, poor solid solubility of Nb in Fe lattice and a remarkable difference in the melting points between pure iron (1809K) and niobium (2742K) restrict the fabrication of these alloys in a wide range of concentration [4].

This article shows structural, morphology and metallographic changes during milling process and thermal stability of alloyed powders. The mechanism of alloy formation in mechanical alloying process has been partially understood. Predominantly, its preparation is mainly through the crystallization of amorphous ribbon or rods obtained by rapid solidification method. Although it is difficult to obtain bulk amorphous materials by this method. Mechanical alloying is an alternative solid state technique by which novel materials may be synthesized from elemental or prealloyed powders. Mechanical alloying is a simple method making it possible to receive alloys with very fine microstructure. Therefore it create the possibility of manufacture of an advanced materials. It effective methods of nanomaterial's production were sough, because conventional ways exhaust already the possibility of production of materials about more and more better properties. The interest in fabrication of nanomaterials follows from difficulties in preparation of materials with better magnetic properties by conventional methods. In the mechanical alloying process fundamental issue is that structure and morphology of metallic powders as well as their changes on each stage of the process may be defined with the use of predetermined technological procedure of receiving powders with present phase composition. The MA enables to obtain amorphous and nanocrystalline materials, intermetallic phases, solid solutions, mixtures of component or metastable phases [5, 8-12, 14, 16].

In this study, the structure and thermal stability of iron based alloy produced by mechanical alloying method were presented. The following investigate techniques to study of Fe-Co-Ni-Nb-Si-B alloy were carried out: X-ray analysis, microscopic observations, differential scanning calorimetry and laser distribution of particle size. These investigations permitted to qualify of studied alloy as well as to set a crystallization temperatures. The main aim of this work was to obtain $Fe_{57,6}Co_{7,2}Ni_{7,2}B_{19,2}Si_{4,8}Nb_4$ alloy by mechanical alloying method. Another aim of this study was to determine an influence of time of mechanical alloying process on alloys' structure and crystallization temperatures, too.

2. Material and methodology

The test material was the mixture of iron, cobalt, nickel, niobium, boron and silicon purity powders obtain by the mixing in suitable proportion (Table 1). The powders were ground for the following time: 10, 45 and 100 hrs.

The mechanical alloying process was conducted in a high energy SPEX 8000 Certi/Prep Mixer/Mill of the shaker type under inert argon atmosphere. The ball to powder weight ratio was 9:1. In this process wasn't added process control agent.

The investigations were carried out by means of the Philips PW 1140 X-ray diffractometer with digital registration. The filtered cobalt anode radiation (35kV, 20mA) was applied. The time of counting in the measuring point was ten second. The crystallites' size was measured by the Scherrer's method basing onself on the diffraction records.

The microscopic observation of the cross section, size and shape powdered particles was carried out by means of the OPTON DS 540 scanning electron microscope provided with the ISIS software for computer recording of image, within the magnification of 500 times.

The distribution of powder particle size after different time of mechanical alloying process was carried out. The measurements were made by means of the laser analyzer "Analysette 22". This laser analyzer made by Fritsch company is the apparatus designed to defining the distribution of solid particles size in the range size 0.1-1250μm. The device consists of helium-neon laser, optical system, measuring container and steer module. Diameters of powder particles are estimated by computer unit on the ground of geometric parameters.

Thermal analysis, differential scanning calorimetry (DSC) was carried out in a Mettler Toledo DSC 822e analyzer. The powder sample (about 30 mg) was taken and used for the DSC measurement. It was heated to 700ºC at a constant heating rate of 10 ºC/min.

3. Results and discussion

The X-ray analysis proved the changes occurring in the mechanical alloying. The X-ray diffraction patterns of the Fe-Co-Ni-Nb-Si-B powders showed the dependence of changes in the

phase composition on the milling time. The diffraction records of powders versus the milling time are shown in Fig.1-2. The X-ray diffraction pattern for the powder after 10 hrs of MA shows the peaks characteristic for iron, nickel, niobium, boron and silicon and nickel oxide. When the grinding time increases, all X-ray peaks become wider and their intensity decreases. The diffraction pattern recorded for the powder ground for 100 hrs shows the peaks characteristic for iron, small peak characteristic for nickel and nickel oxide.

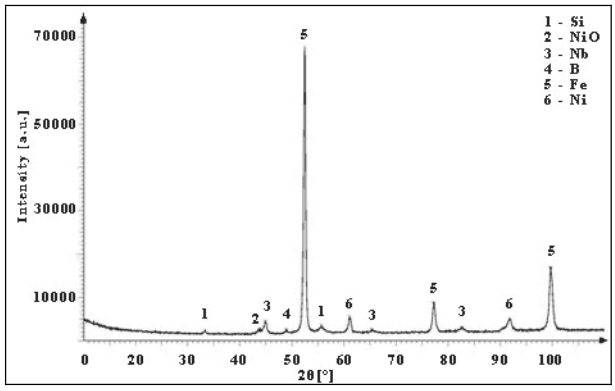

Fig. 1. The X-ray diffraction pattern of $Fe_{57,6}Co_{7,2}Ni_{7,2}B_{19,2}Si_{4,8}Nb_4$ powder alloy after 10 hrs of mechanical alloying process

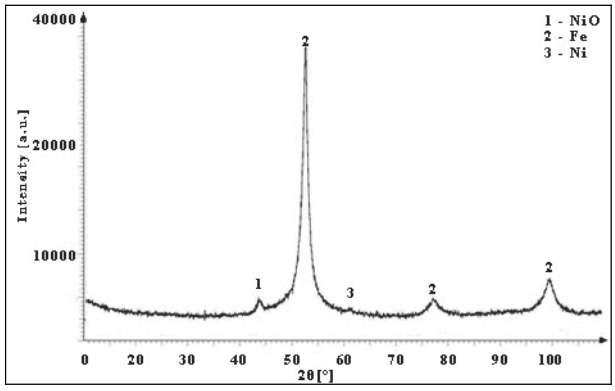

Fig. 2. The X-ray diffraction pattern of $Fe_{57,6}Co_{7,2}Ni_{7,2}B_{19,2}Si_{4,8}Nb_4$ powder alloy after 100 hrs of mechanical alloying process

The widening of peaks is connected with the size reduction in the powder grains and presence of stresses resulting from the intensive plastic strains occurring during the MA process. The amorphous phase become the most visible in the last sample after 100hrs of milling. As the time of MA increases, the crystallites size is reduced and progressively the alloy becomes amorphous. The diffraction patterns obtained on the phase analysis allowed applying the Scherrer's method to determine the crystallite size. After 10hrs of grinding, the crystallite size was estimated as being about 40nm and after 100hrs as being 20nm. From the test carried out on the scanning electron microscope it results that the average grain size of the Fe-Co-Ni-Nb-Si-B alloy powder decreases together with the increased time of milling. Figs.3-6 show the sequent powder structure after 0, 10, 45 and 100hrs of grinding.

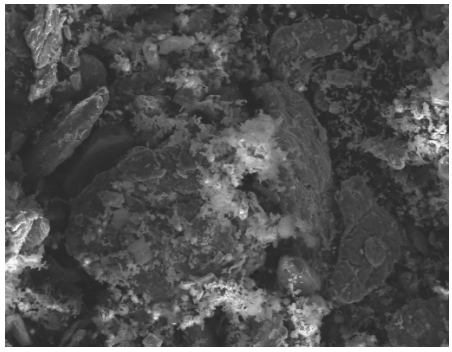

Fig. 3. Structure of initial powder mixture Fe-Co-Ni-Nb-Si-B (SEM, 500x)

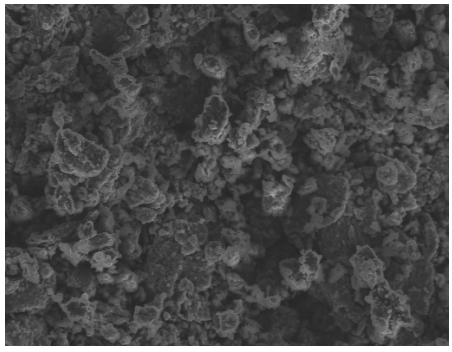

Fig. 4. Structure of powder's particles of Fe-Co-Ni-Nb-Si-B alloy after 10 hrs of mechanical alloying (SEM, 500x)

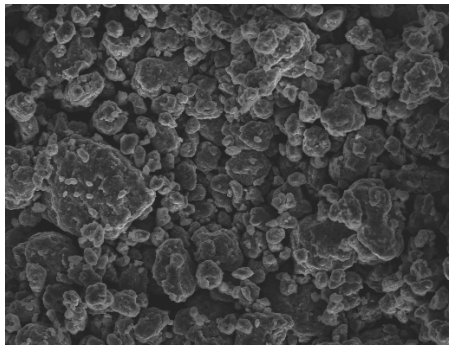

Fig. 5. Structure of powder's particles of Fe-Co-Ni-Nb-Si-B alloy after 45 hrs of mechanical alloying (SEM, 500x)

The cross-sectional microstructure evolution and element distribution of tested powder alloy were investigated using SEM, too. The image of the cross-section of powders' particles after 100hrs of mechanical alloying process are shown in Fig. 7. Bright and dark areas in this figure correspond to Nb and Fe, respectively.

From this images, it can be seen that the elemental distribution of iron, cobalt, nickel, niobium, boron and silicon were not uniform in this stage of process. Investigation of the single powder's particle of the chemical composition made by the X-ray energy dispersive spectrometer (EDS) indicate that niobium

and iron dominate inside of bright (1) and dark (2) area's, respectively. Plots of the X-ray dispersive energy spectrometer measurement are shown on Fig. 8 and Fig. 9. EDS test of the chemical composition of Fe-Co-Ni-Nb-Si-B alloy indicate that the process is not come to an end.

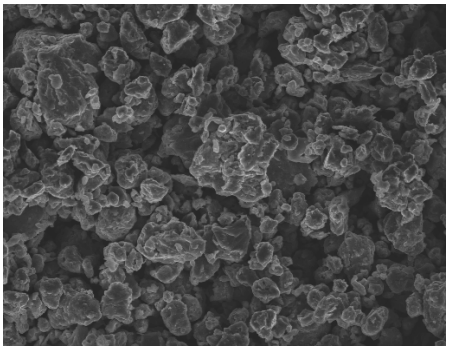

Fig. 6. Structure of powder's particles of Fe-Co-Ni-Nb-Si-B alloy after 100 hrs of mechanical alloying (SEM, 500x)

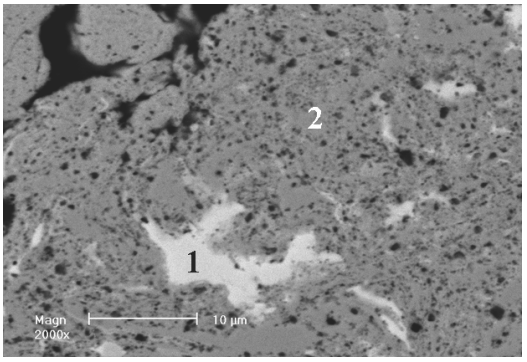

Fig. 7. Cross section of Fe-Co-Ni-Nb-Si-B powder alloy mechanically milled for 100 hrs (SEM)

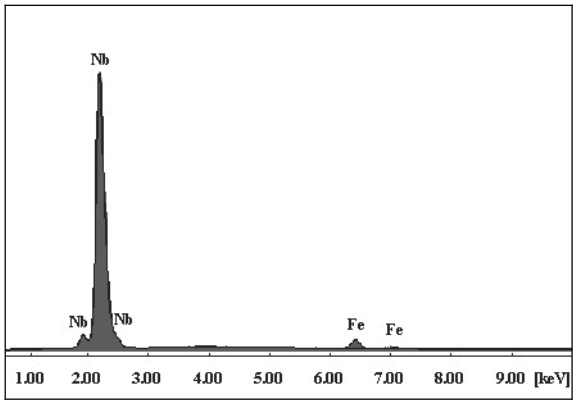

Fig. 8. Plot of the X-ray dispersive energy spectrometer measurement from the Fe-Co-Ni-Nb-Si-B alloy after 100hrs of mechanical alloying process (point 1 in Fig. 7.)

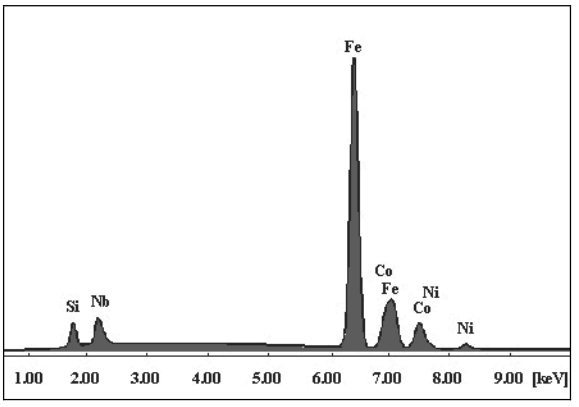

Fig. 9. Plot of the X-ray dispersive energy spectrometer measurement from the Fe-Co-Ni-Nb-Si-B alloy after 100hrs of mechanical alloying process (point 2 in Fig. 7.)

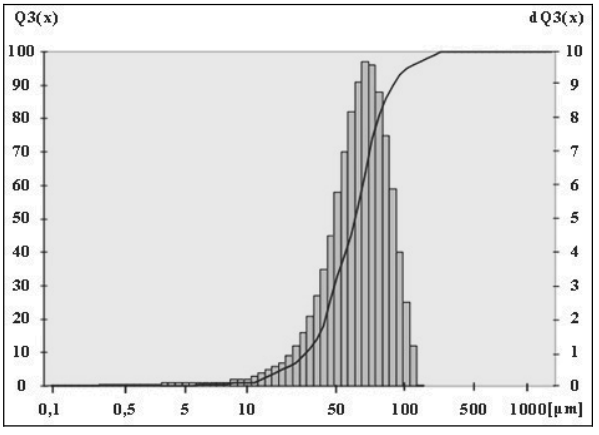

Fig. 10. Particle size distribution of studied Fe-Co-Ni-Nb-Si-B initial powder

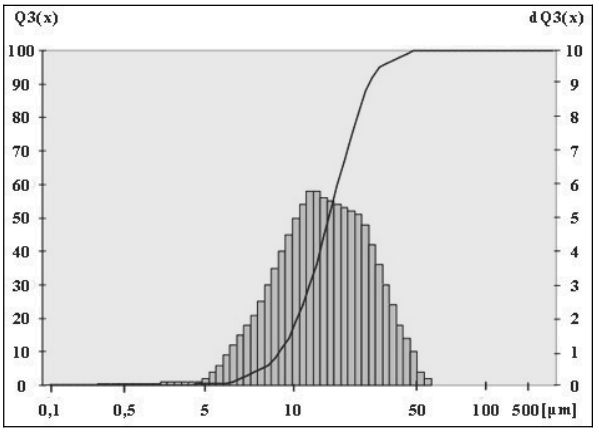

Fig. 11. Particle size distribution of studied Fe-Co-Ni-Nb-Si-B powder after 100 hrs of mechanical alloying

Table 2.
The parameters versus time of mechanical alloying process

	Arithmetic mean diameter [µm]	Mode [µm]
initial powder	67.2	74.1
powder after 10 hrs of MA	55.1	59.8
powder after 45 hrs of MA	32.1	35.6
powder after 100 hrs of MA	21.7	23.9

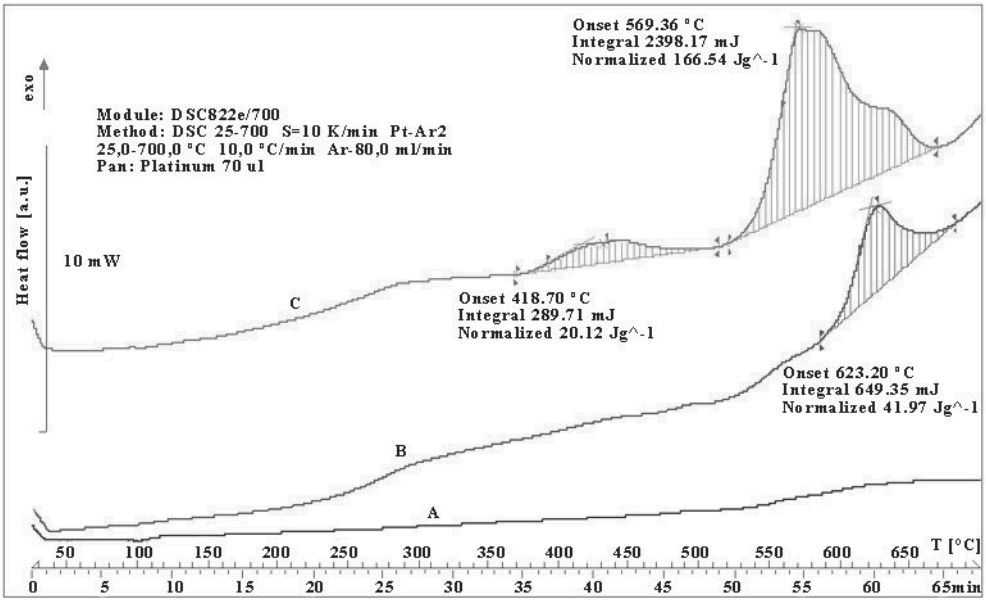

Fig. 12. DSC scans at a heating rate of 10K/min. corresponding to $Fe_{57,6}Co_{7,2}Ni_{7,2}B_{19,2}Si_{4,8}Nb_4$ alloy: A-initial powder, B-powder after 10hrs, C-powder after 100hrs

The investigation of particle size and their distributions were executed. The influence of milling process time on the parameters of mode and arithmetical mean diameter were presented in Table 2.

The distribution of powder particle size is shown in Fig. 10 and Fig. 11. From the tests carried out on the laser analyzer it results that the mode and arithmetical mean diameter decreases together with the increased time of grinding. The application of mechanical alloying process results in relatively constant size of powder particles after very long milling time. The optimum time depends on the mill type, ball to powder weight ratio, chemical composition et al.

The calorimetric measurement enables to detect energetic changes during heating to a temperature of 700ºC. From the test carried out on the differential scanning calorimeter it results that amorphous structure obtained in MA process arranges under heating. During the heating of studied alloys the exothermic peaks form. These peaks testify for phase transformations and chemical reactions. Fig. 12 shows DSC curves over the whole temperature range and the enlarge crystalline transition regions of amorphous alloy powders. The initial powders are characterized with the crystal structure. The DSC analysis proved no changes occurring in the heating to a temperature of 700ºC.

The DSC curve (Fig. 12, curve A) is rather straight. The curves B, C recorded for the powder ground for 10 and 100hrs show the exothermic peaks, whereas none endothermic peak was observed. The amorphous phase become the most visible in the last sample after 100hrs of mechanical alloying. The powdering of the crystalline structure is increased together with the increase in the grinding time, what brings about the rendering the alloy to be partially amorphous again. The volume fraction of the amorphous phase increases progressively.

4. Conclusions

The result of the investigation show that the application of the mechanical alloying process for production Fe-Co-Ni-Nb-Si-B powder alloy enables to obtain nanocrystalline and amorphous structure.

The crystallite size of the obtained alloy decreases when the time of mechanical alloying increases. It is expected that solid solution of boron and silicon in iron appears.

The extension of milling process reduce powder particle' size. It was observed, the powder structure become more homogeneous. The particles after 100hrs of MA are contain within the range 0.4–53.3µm.

The exothermic reaction were conducted by differential scanning method. The crystalline structure at 623ºC and 569ºC was achieved.

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