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Microstructural characterisation of sintered soft magnetic nanocomposite materials

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Materials

ABSTRACT

Purpose: The main aim of this work is to investigate microstructure of of sintered soft magnetic nanocomposite materials produced by sintering axially on one side in the ambient atmosphere.
Design/methodology/approach: Microstructure observations of 20 mm diameter sintered roller by light microscopy OLYMPUS, scanning electron microscopy OPTON DSM-940 and ZEISS SUPRA 35, and transmission electron microscopy JEOL 3010. The X-ray tests were realized with the use of the XRD 7 SEIFERT-FPM diffractometer equipped with the lamp of the cobalt anode of 35 kV voltage and 30 mA filament current was used. The nanocrystalline ferromagnetic powders were manufactured by high-energy ball milling (8000 SPEX CertiPrep Mixer/Mill) of metallic glasses ribbons in as state. The hot pressing process was made on machine "Degussa".
Findings: The analysis of the results enabled determination of the hot pressing parameters on structure of obtained stampings. This is typical of an dispersion strengthened case.
Research limitations/implications: For the sintered roller obtained from metallic Co-based amorphous ribbons, further mechanical and magnetic examinations are planed.
Practical implications: Conducted research shows that applied technology of sintered roller production allows to obtain good microstructural characteristics. Structure analysis of die stampings of powdered amorphous metallic ribbons is helpful to prepare this material by laboratory methods. Feature an alternative to commercial alloys and composite materials are the amorphous and nanocrystalline metal amorphous ribbons obtained by melt spinning technique and make it possible to obtain the new composite materials with best magnetic properties, which dimensions and shape can be freely formed.
Originality/value: The paper presents influence of hot pressing parameters process of metallic powdered ribbons $Co_{77}Si_{11.5}B_{11.5}$ on structure of obtained die stampings.
Keywords: Amorphous ribbon; High energy ball milling; Microstructure; X-ray spectometry

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1. Introduction

Soft and hard magnetic materials can be prepared by various methods [1,2]. Among the soft magnetic materials that have a high magnetic saturation (Bs), low coercion (H_C) and a very high magnetic permeability (μ) and very low (or zero) magnetization losses in amorphous and nanocrystalline metal strips [3-5].

In the production of metallic glasses are only practical significance three ferromagnetic elements Fe, Co and Ni, mainly due to its high Curie temperature, which means the phase transition between the ferromagnetic and paramagnetic materials. This property prevents the use of for example gadolinium of saturation induction of 2.8 T, which loses its good magnetic properties at temperatures of 20°C [6]. In practice thus prepared alloys of transition metals, namely Fe, Co, Mn, Cr, Ni with a metalloids such as B, P, C, Si, S. The atomic fraction of non-metals in the alloy is approximately 20 to 30% [7-9].

Metallic glass, in which the main alloyed component is cobalt with almost zero magnetostriction ($\lambda_s \approx 10^{-6}$) and the saturation induction $B_R \approx 0.6$ to 1.0 T. For example, in the glasses of the type $(Co, Fe)_{80}B_{20}$ magnetostriction reaches zero for $Co_{95}Fe_5$ chemistry [10-13]. In the cobalt-based amorphous alloys the Curie temperature is higher than the crystallization temperature (Tc>Tx) unless the metalloid concentration is greater than 25 atomic%. Alloying elements such as Mo, V, W reduces the Curie temperature and cause an increase in permeability μ_e. The value of magnetic permeability μ_e after annealing in optimal conditions reaches 200 000 (Fig. 5) [14-17].

Amorphous strips on the base of Co and Fe show the effect of giant magnetic impedance - GMI. In particular, this effect is characteristic for amorphous alloys of cobalt matrix. The effect of magnetic resistance in the amorphous material based of Co, which is annealed shows that magnetic anisotropy is induced in the material and this value increases with stress [18].

Metallic glasses exhibit the characteristics of a metal properties such as electrical and thermal conductivity, shine, strength, formability and structural disorder characteristic of glasses. However, they are thermodynamically unstable materials and aim to transition equilibrium, which is the crystalline state [19,20].

The disadvantage of the materials obtained by the melt spininng their shape (strips), and brittleness (after heat treatment) that hinder their practical application. Powder nanocrystalline and amorphous materials are produced by the mechanical alloying or a high energy ball milling can solidify resulting in the possibility of practical application [21-25].

Grinding the pieces of the strips obtained by melt spinning in Retach type planetary mills or vibratory type 4000 Fritsch Pulverisette (0), with different weight ratio BPMR (Ball Powder-to-Mass Ratio) was performed at different alloys [26-29]. Both in which the ambient temperature and at low temperatures (77 K) so. (cryo-milled) [30].

One of the ways to solidify the powder material is sintered. It depends on the junction of the individual powder particles upon heating to form a composite with a specific physico-chemical and mechanical properties [18]. As a result of the diffusion of the powder particles loosely linked under inert gas (argon) or vacuum formed solid material [31]. There are the ways to perform the sintering process:

- freely sintering,
- sintering under the force, connected with the moulding.

Another method of consolidation of nanopowders is isostatic pressing, which can be both cold and hot. Isostatic powder cold compaction technique overcomes the drawbacks of the product as a result of uniaxial, which is associated with reduced ratio H/D and an inhomogeneous distribution of density. The result of this process the molding is narrow. The second type of isostatic pressing is hot isostatic pressing (HIP - Hot Isostating Pressing). By using the HIP method may be produced with complex shapes. The sintering process under pressure has the additional advantage that it can be carried out at lower temperature and shorter time compared to the free sintering process. It is typically carried out in the matrixes by compression of graphite bilateral or hot isostatic pressing [1].

Explosive pressing also known as explosive merge allows compaction of the material without prolonged exposure to elevated temperatures. The energy of the shockwave may influence the powder material by the impact of the metal plate in a container with powder (unidirectional pressing) either directly by the action of the explosive gas pressure (isostatic pressing). The material thickened by pressing explosive exhibits good magnetic properties. The effect of the detonation wave, the powder is compressed axially and radially, and thus generated heat causes local, superficial melting of the powder [1,2].

In a large group of magnetic nanocomposite materials are very interesting class of magnetic particles suspended in the polymer matrix [25, 32-40]. They can be used as electromagnetic shields, as well as the warp highly reactive particles. Depending on the size of the magnetic particles, polymer matrix can act as a separating providing intermolecular interactions interchangeable. It was also observed the influence of these particles on the structure of the polymer.

The most common method of bonding the magnetic powder material is a bond polymeric materials comprising the mixing of the magnetic powder with a polymer chemo- or thermosetting and cold pressing or at elevated temperature [2,8,41,42].

Selection of appropriate technologies solidification powder material provides the required structure, physical properties and the shape of the composite nanocrystalline soft magnetic material.

The aim of this work is to investigate the microstructure of the sintered powder $Co_{77}Si_{11.5}B_{11.5}$ alloy obtained from the metallic glass in the high energy ball milling process.

2. Material and methods

The investigations were carried out on a $Co_{77}Si_{11.5}B_{11.5}$ metallic glass in form of 0.025 mm thick and 10.2 mm wide ribbons. A 8000 SPEX CertiPrep Mixer/Mill high energy ball mill was applied to mill the ribbons both in „as quenched" state and heat treated. The vibration times were between 5 and 80 hours.

The hot pressing process was made on machine "Degussa". The composite powders were produced by high-energy ball milling in SPEX 8000 mill. The parameters of the sintering process are show in Table 1.

The X-ray tests were realized with the use of the XRD 7 SEIFERT-FPM diffractometer equipped with the lamp of the cobalt anode of 35 kV voltage and 30 mA filament current was

used. Diffraction tests were carried out in the 2θ angle range from 40 to 120° (measurement step 0.1°). Pulse counting time was 5 s.

Microstructure observations were made by light microscopy Leica MEF 4A, scanning electron microscope OPTON DSM-940 and ZEISS SUPRA 35, and transmission electron microscope JEOL JEM 3010.

Thin foils of investigated composites were prepared by mechanical pre thinning, followed by low angle ion polishing realized in Gatan PIPS^{TM} high milling rates unit. The microstructure examination and diffraction investigations of phase composition of the thin foils were made on the transmission electron microscope at the accelerating voltage of 300 kV. The diffraction patterns from the transmission electron microscope were solved using a computer aided program.

3. Results and discussion

The starting material for the process are high energy ball milling were sections of the ribbon length l≈10 mm. The powder $Co_{77}Si_{11.5}B_{11.5}$ alloy obtained after 5 hours of high energy milling characterized in grain shape "petals" of the ribbon (Fig. 1). The average size of these flakes is 230.9 μ m (standard deviation s2 = 163.2). After the milling time increased to 10 hours powder average grain diameter is 89.7 μ m (standard deviation s^2=136.6). The lowest average grain diameter of 7.4 m μ characterized by a powder obtained after 60 hours of milling (Table 2). The powder obtained from the amorphous strip $Co_{77}Si_{11.5}B_{11.5}$ after 5 hours of milling grains share a diameter of 100 to 200 μm is 31% (Fig 2), after 25 hours of milling grain participation in diameter 0 to 20 μm is 75% (Fig. 3), and the powder obtained after 60 hours of milling grain participation in diameter 0 to 10 μm was 88%. Effect of milling time of high particle size of the obtained powder is shown in Fig. 4.

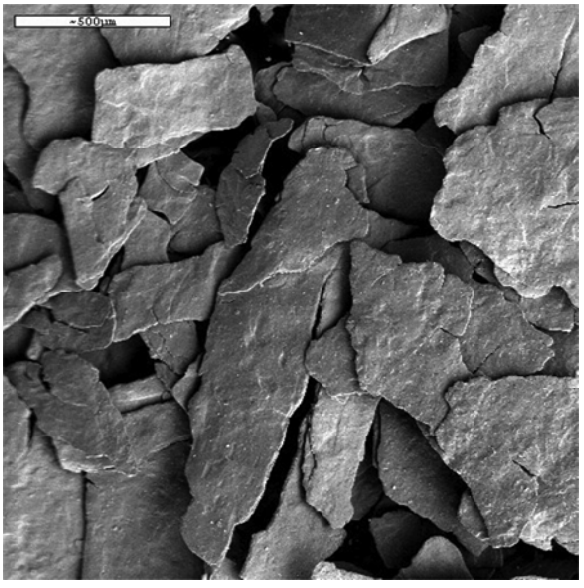

Fig. 1. Powder grains image after 5 hours the high energy milling, SEM

Table 1.
The parameters of the sintering powder

Process parameters	Values
sintering temperature [°C]	800
stamp pressure [kg/cm^2]	150 (15 MPa)
sintering time [min.]	20
atmosphere:	vacuum 2×10^{-2} T

Table 2.
Average particle size of the powder CoSiB after various times of milling

value	The diameter of the powder particles during the milling [h]:						
	5	10	15	20	25	30	60
average value [μm]	230.9	89.7	62.3	38.7	15.6	26.1	7.4
standard deviation	163.2	136.6	42.7	20.5	7.3	13.0	12.0
maximum value [μm]	840.4	603	159.4	109.8	46.7	71.0	91.4
minimum value [μm]	8.4	5.7	4.7	11.4	5.0	7.7	1.3

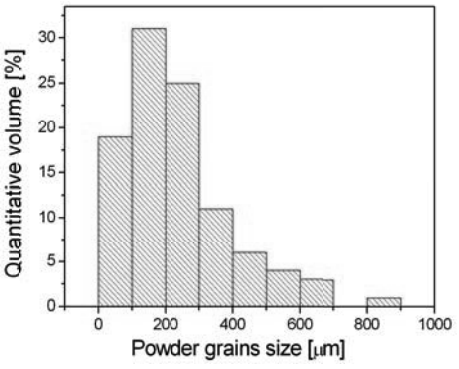

Fig. 2. Histogram particle size measurement results of the amorphous powder obtained from $Co_{77}Si_{11.5}B_{11.5}$ alloy subjected to high energy ball milling for 5 hours

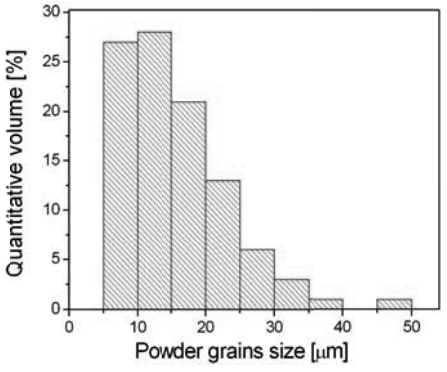

Fig. 3. Histogram particle size measurement results of the amorphous powder obtained from $Co_{77}Si_{11.5}B_{11.\ 5}$ alloy subjected to high energy ball milling for 25 hours

Result of measurement of grain size, depending on the milling time summarized in Figure 4 were approximated by Blotzmanna model. Obtained equation determining the effect of time grinding grain size of the amorphous alloy powder obtained $Co_{77}Si_{11.5}B_{11.5}$. Compatibility of the equation (1) with experimental results is higher than 98%.

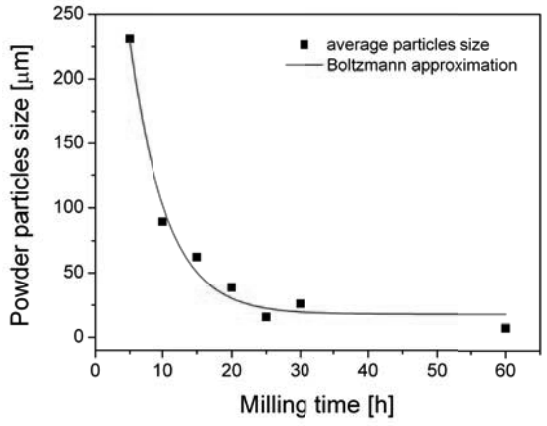

Fig. 4. Effect of the milling time for the amorphous strips made out of $Co_{77}Si_{11,5}B_{11,5}$ alloy on the powder particles size for the obtained powders

$$y = A_2 + \frac{A_1 - A_2}{1 + \exp\left(\frac{x - x_0}{dx}\right)} \quad (1)$$

$R^2 = 0.98431$
$A_1 = 2775,37878$
$A_2 = 17.82147$
$x_0 = -7.8864$
$dx = 5.16802$

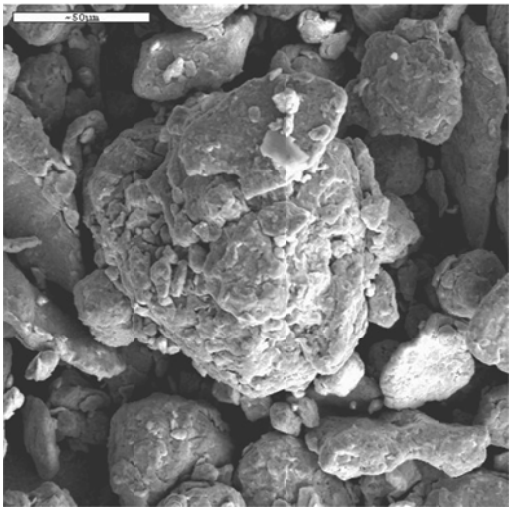

Fig. 5. Powder grains image after 25 hours the high energy milling, SEM

On the basis of observations made on scanning electron microscope, it was found that the microstructure of the sintered (Fig. 6) is very close to the model developed by Suryanarayana (Fig. 7). There are clearly grain boundaries, which are probably the place of contact of the powder particles during sintering. Grains have an elliptical or close to oval shape, without sharp edges. Observed grains are very different in size. Revealed the presence of particles with a size of 1-2 µm as well as the particle size of 20-25 µm.

Based on the results of X-ray phase analysis (Fig. 8), using the Scherer equation (2) calculated crystallite size of the Co-β phase [29]. In the calculations made show that with increasing milling time increases the size of crystallites, that after 15 hours is 30 nm, 32 nm after 20 hours and 41 nm after of 80 hours is achieved.

$$B = \frac{k \cdot \lambda}{d \cdot \cos\theta_B} \quad (2)$$

where:
d - diameter of the crystalline particle,
B - width of the diffraction peak measured at half of its height,
k - coefficient assumed as equal to 1 [43]
λ - X-ray radiation wavelength,
$2\theta_B$ - radiation beam diffraction angle corresponding to the Bragg maximum.

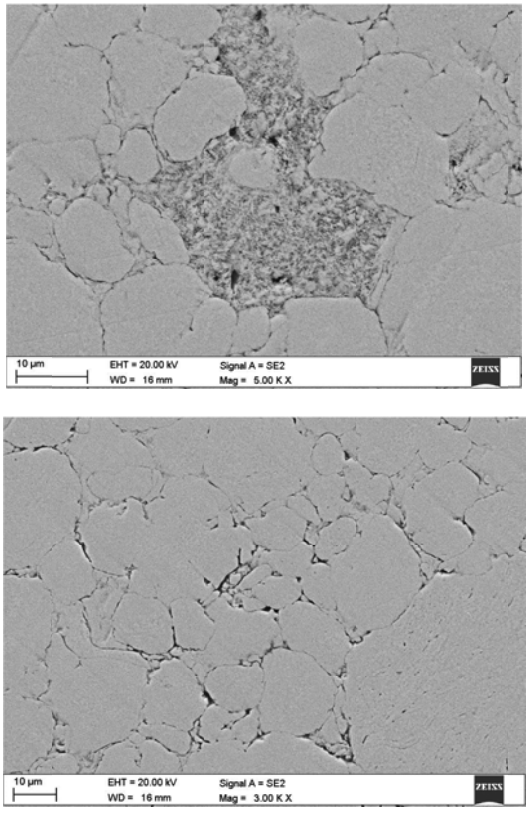

Fig. 6. The microstructure of the sinter produced from the powder $Co_{77}Si_{11.5}B_{11.5}$ after 80 hours of milling, SEM

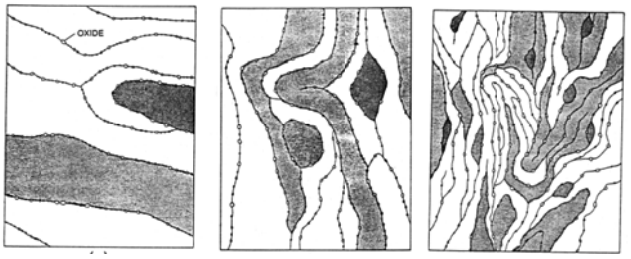

Fig. 7. Schematics of microstructural evolution during milling of a ductile-brittle combination of powders. This is typical of an dispersion strengthened case [44]

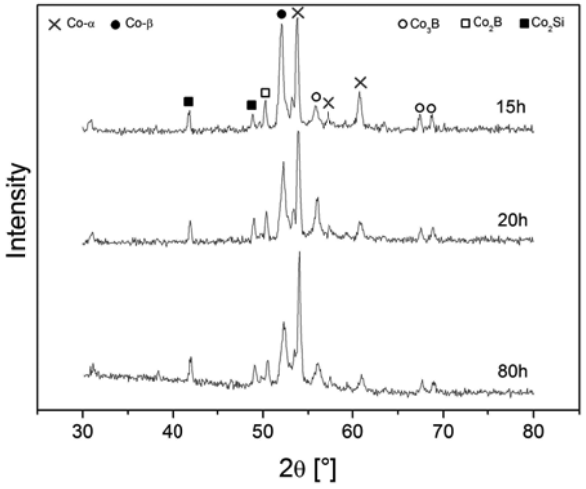

Fig. 8. X-ray diffraction pattern of the powders produced at 15, 20 and 80 hours of milling the sintered under a pressure of 15 MPa at 800°C for 20 minutes

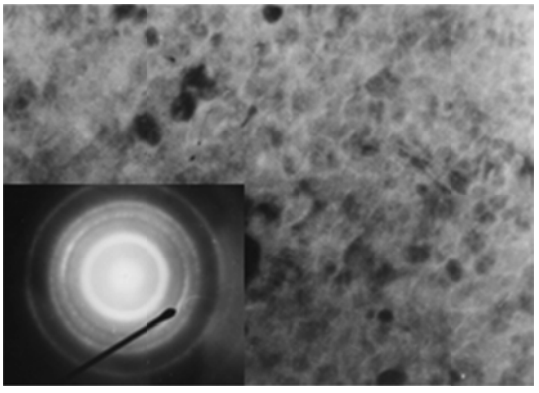

Fig. 9. The structure of the powder obtained after 25 hours of high energy milling of the amorphous ribbon $Co_{77}Si_{11,\ 5}B_{11,\ 5}$ alloy; TEM, magnification 100000x

By observation of the TEM, it was found that the high energy milling process performed even in a short time (10 hours, 25 hours) results in formation of a nanocrystalline structure (Fig. 9). This structure differs from the nanostructure obtained by the isothermal annealing of amorphous ribbons (Fig. 10), it is more irregular, non-homogenous and contained in the grains are very diverse in terms of shape and size. Based on the analysis of diffraction by transmission microscopy found in the structure of the powder obtained after 25 hours of milling the presence of Co-β phase, boride phase Co_2B cobalt, cobalt silicide Co_2Si and $CoSi_2$ phase.

a)

b)

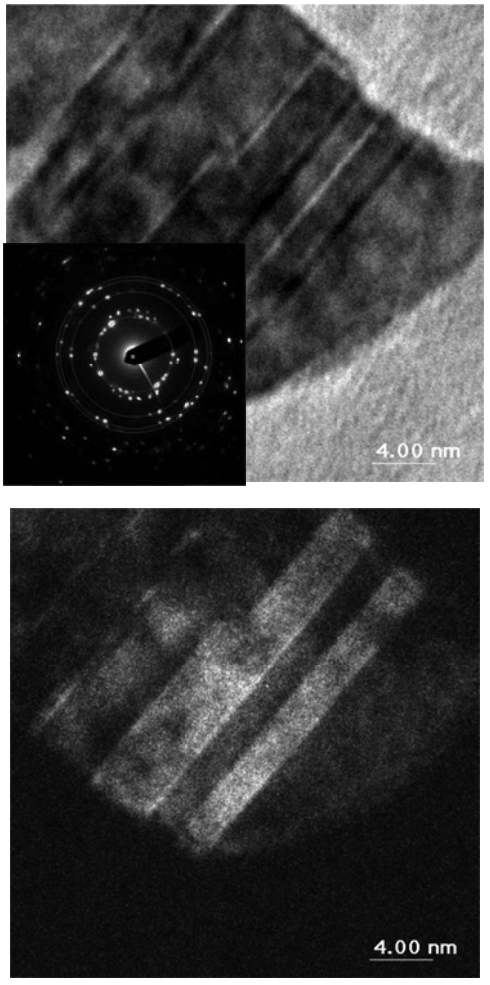

Fig. 10. The twins precipitation $Co_{90}Si_{10}$ phase with structured P63mmc a) dark field view b) in an amorphous ribbon $Co_{77}Si_{11,5}B_{11,5}$ alloy isothermally annealed at 500°C/1h under argon

The microstructure of the sintered powder obtained after 80 hours of milling $Co_{77}Si_{11,5}B_{11,5}$ alloy ribbon revealed the presence of particles with a diameter of about 200 nm in the amorphous matrix (Fig. 11). Larger grain shape similar to the oval characterized by a mean diameter around 235 nm but smaller elliptical in shape with a longer diameter of about 150 nm and less than about 100 nm. The larger grain are structure characteristic of the materials with micrometer structure and in a smaller grain size observed nanometer subgrains. Additionally, it was found that the grains are arranged irregularly in the amorphous matrix.

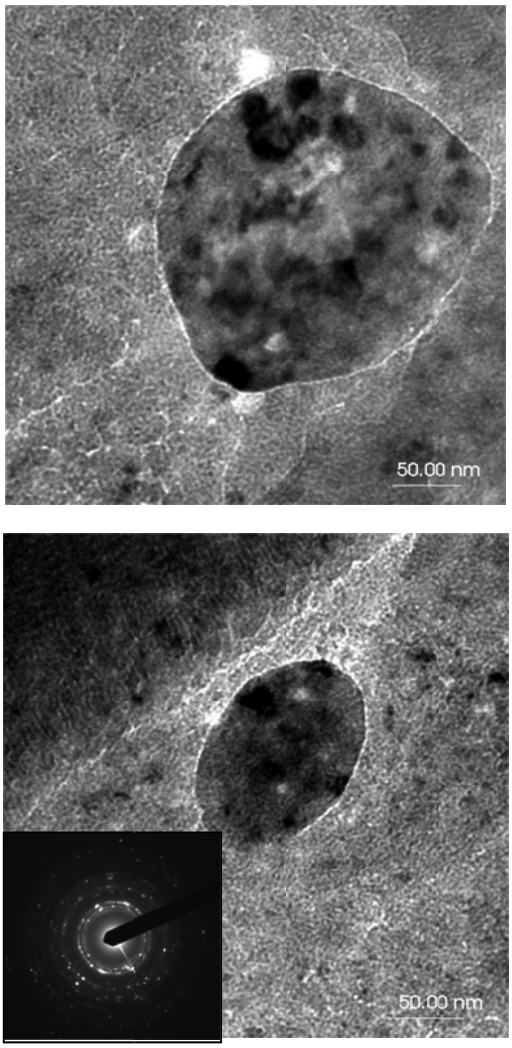

Fig. 11. The structure of the sinter obtained after 80 hours of high energy milling of the amorphous ribbon $Co_{77}Si_{11,5}B_{11,5}$ alloy; TEM

4. Conclusions

On the basis on results of investigations of structure of the sintered material, it was found out that compared to the structure of the amorphous ribbon and powder obtained from ribbons in high energy ball milling as their both precursor, the hot pressing process shapes completely different structure. It is a hybrid, a combination of nano-and micrometer structures.

On the basis on results of investigations of microstructure of sintered soft magnetic nanocomposite materials it was found out that compared to the amorphous ribbon one can we obtained that:

1. It was found out in observations on the scanning electron microscope that along with the milling time increase the powder particles size decreases, and that their shape changes also during the process. The powder grains were flake-sized at the first stage of the process, and actually they were parts of the strips. However, as the milling time grows the grains become spherical with a clear tendency to get smaller. Decreasing of the powder particles size is very intensive during the first hours of milling; whereas, at the later stage of the process changes of the average particle size are of the order of several percent per milling hour. On the basis of the results was determined using the equation of time grinding ribbon impact on the grain size of the obtained powder (1). The acquired results are comparable to results obtained by other authors.
2. On the basis on analysis of diffraction patterns and using Scherrer's relationship (2) the Co-β grains size was calculated, whose dimensions grow in this case along with increasing milling time increases the size of sinter crystallites, but probably the calculated peaks include the background form amorphous phase and the results are not binding
3. The hot pressing of metallic powder obtained in high energy ball milling of amorphous ribbons introduces a lot of slots (Fig. 6) into the structure of material, with negative influence on the properties.
4. Studies by TEM showed that the sinter structure are grain size 235-100 nm irregularly arranged in amorphous matrix.

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