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NANO-CRYSTALLINE STATE OF FECO50 OBTAINED IN A PLANETARY BALL MILL

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Abstract: *The necessities of high energy milling systems leads to the development of the planetary ball mills. It was considered a planetary mechanism for a planetary ball mill suitable for metallic powder milling. A prototype was tested intensively on a FeCo50 % wt. at several milling time in order to evidence the designed planetary ball mill efficiency. The X-ray diffraction pattern reveals that after 16 hours of milling the nano-crystalline state was achieved. The FeCo50% wt mechanical alloying process is realized according with the ductile- brittle alloying model.*
Key words: *planetary system, ball mill, metallic powders*

1. INTRODUCTION

The metallurgic industry and in the department where new materials are obtained there is a big development in research and accomplishment of materials obtained from sintered metallic powders [1, 2].

The powder crushing and grinding process is characterized by a high complexity of changeable factors, that is: the reciprocal position of the grinding particles, the shape of the material, the physical-mechanical properties of the material, plasticity, humidity, the state of the areas of the body to be ground, the speed, the mass, the acceleration of the breaking parts, which cannot be examined from a quantitative point of view under certain mathematical relations [3 - 5].

The different powders of metallic materials have to be of corresponding dimensions, for technological purposes, so that certain grinding levels be obtained, by the help of the crushing equipment. Several researches reports very good results for the Fe – Co system using high energy ball milling [6].

In order to obtain some final dimensions of the powder particles between 0.5…0.1 mm, balls or cylindrical grinders can be used during the grinding process [1, 3]. This grinding process can be labeled as gross grinding process.

Inclined cylinder, vibrating or planetary grinders are used for a fine grinding, in case of particles reaching up to 0,01 mm.

The equipment to be presented was designed for metallic powder grinding and is destined for research laboratories of powders and material obtaining process from metallic powders. This equipment helps grinding metallic powders, reaching to different dimensions of the particles depending upon the technological requirements.

As the equipment was exclusively destined for laboratory and the powder quantities to be ground are relatively small, it was designed at lower dimensions and grinding capacity.

2. MECHANICS AND CALCULATIONS

The planetary grinder is driven by an electrical engine, which, by means of a trapezoidal belt transmission rotates a disk with four grinding barrels.

The movement transmission from the disk to the four barrels is performed by means of a gear with straight teeth wheel; the barrels and satellite teeth wheels have a common shaft connecting a solar wheel.

The material to be ground is introduced in the four barrels together with the steel balls, which contribute to the grinding of the particles. This particle grinding takes place because of ball collision and particle friction. Fig.1.

The solution adopted confers simplicity to the equipment and easily adopting solutions.

Radial roller bearings were used in all bearings, usual bearings easy to be found which have a low friction coefficient that considerably reduces the power of the engine driving the grinder [7, 8].

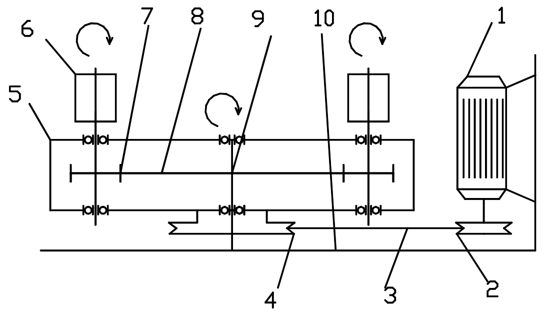

Fig. 1. The mechanical diagram of the proposed planetary ball mill.

The diagram in figure 1 contains: 1. Electric engine; 2. Conducting belt wheel; 3. Trapezoidal belt; 4. Guided belt wheel; 5. Disk (barrel holder); 6. Grinding barrels; 7. Satellite camshaft; 8. Fixed solar teeth wheel; 9. Fixed main axis; 10. Base.

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The skeleton diagram of the planetary grinder is described in Diagram 1; the planetary grinder, by the help of the electric engine (1), by means of the belt transmission (2,3,4) drives and imprints a rotation movement to the central disk (5) which rotates around its main fixed axis (9). The bearing between the central disk and the fixed axis are, by means of two radial roller bearings, disposed as follows: one in the lower part and the other in the upper part. The grinding barrels (6) revolve and perform a satellite movement around the fixed axis (9). This is accomplished by means of the straight teeth wheel gear. On the fixed central axis (9) there is a teeth wheel (8) which drives the camshafts (7) located on the grinding barrel axis (6). The teeth wheel (8) being fixed, and the central disk moving around it together with the grinding barrels, these driving the teeth wheels (7), thus a rotation movement around their own axis is also impressed to them. The movement is in the same direction as the one of the big disk, but with a higher rotation speed.

In studying the constructive options, it was taken into consideration the destination of the equipment, its exploitation conditions and capacity [2, 6].

Thus at the existing options of planetary grinders, six grinding barrels are usually used, this leading to an increased productivity. The fact that there was no need for such a quantity of material for grinding was debated in the research laboratory, in this case the barrels usually staying unoccupied.

A special importance, while using the equipment, is paid to loading the grinding barrels with the same material quantity or at least of two barrels, these being located on opposite sides. If this condition is not fulfilled, a static and also dynamic disequilibrium occurs, leading to equipment destruction and risks of accident [8, 9].

Thus the number of the barrels was cut to four, their capacity being relatively small, but sufficient for the laboratory. The reduction of barrel number implicitly led to a cut in the equipment volume to a quarter of the six-barrel equipment. The main desired technical features are:

1) The rotation speed of the central disk; $n_d = 400$ rot/min
2) The rotation speed of the grinding barrels; $n_t = 493$ rot/min
3) The number of the grinding barrels: 4
4) Acceleration = 20 g
5) The engine rotation speed = 930rot/min
6) Engine power = 1.1 kW
7) Total barrel capacity = 1396 cm^3
8) Useful capacity = 977 cm^3
9) The range of the container center = 112 mm
10) The ratio n_t / n_d = 1.25
11) Overall dimensions of the equipment: 620x410x425 mm

12) Equipment weight = 120 kg.

The grinding mechanism is as follows: the balls are driven by the grinding barrels together with the material to be ground. These, in certain points, detach themselves from the barrels' walls and are projected inside the barrel hitting the material to be ground and even the other existing balls, impressing those boils within the barrels.

Grinding takes place upon the friction and collision process of all elements in the barrel.

The acceleration in the barrel center from Fig.1 was imposed at 20g so that the grinding process is more efficient.

The process is characterized by a detachment point A of the balls during the continuous rotation of the cylinder and a point of return of this to the B point, Fig. 2.

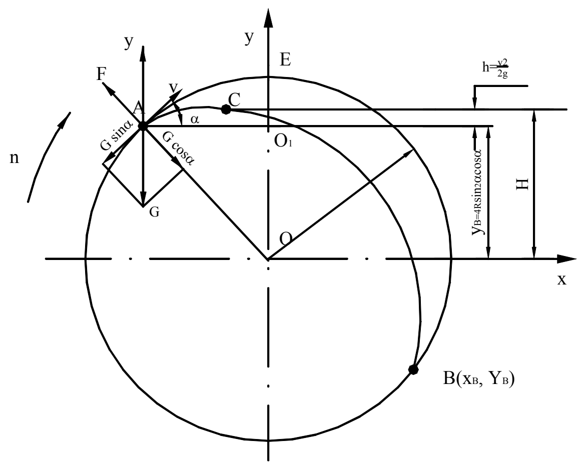


Fig. 2. The trajectory of a particle in the ball grinder

Between A and B the particles have to accumulate the highest energy. The rotational speed of the cylinder is given by:

$$n = \sqrt{\frac{20g \cdot 30^2 \cdot \cos\alpha}{\pi^2 \cdot R}} \tag{1}$$

Where real data are introduced, resulting:

$$n_{critic} = \sqrt{\frac{18000\ g}{\pi^2 \cdot R}} = 704{,}96 \text{ rot/ min}$$

The determination of the optimal rotational speed:

$$n_{optim} = 0{,}758 \cdot n_{cr} \text{ rot/min} \tag{2}$$

$$n_{optim} = 534{,}35 \text{ rot/min}$$

Practically the regime rotation speed is chosen:

$$n_{reg} = (0{,}68\ldots0{,}73)n_{cr} \tag{3}$$

The 0,7 coefficient was chosen and it results:

$$n_{reg} = 493{,}472 \text{ rot/min} \tag{4}$$

The n_{reg} = 493, 472 rot/min was chosen. The rotational speed of the disk is considered at 400 rot/min Provided that 20g be obtained, the range of the container center results to be:

$$R = 0{,}112 \text{ m} \tag{5}$$

The satellite teeth wheel has 33 teeth, and the solar one 44 ones.

The barrels are centrally rounded inside, according to Fig.3, because it could be seen that for a small non-satisfactory connection of the inner walls, a part of the loading is no longer ground, but pressed because of the beats in the non-connected area as could be seen in Figure 3.

Fig. 3. Prototype of designed planetary ball mill.

The external dimensions of the barrel are performed at a free quota, because there is no high precision. The barrel is fixed on the plate by the help of a three-armed stretching element clutching the catch pins on the axis plate, and by means of a screw the barrel is concomitantly fixed and closed. (there is a centering housing in the barrel's cover). For sealing purposes there is a thin rubber ring between the barrel and the cover.

3. EXPERIMENTAL RESULTS

The planetary ball mill prototype was intensive tested for mechanical alloying applications. Therefore, a Fe50Co50 wt. % mixture of elementary powders was milled for several times ranging from 1 to 16 hours. For this very purpose we collect powder samples after 1 and 16 hours of milling which were tested by several scientific methods.

The most important parameters monitored are the particle shape and size respectively the crystal grains shape and size. Those parameters are related with the alloy crystal structure. The phase evolution during mechanical alloying process was investigated by X-ray diffraction using a DRON 3 diffractometer equipped with Matmec VI.0 data acquisition module and processing soft. The microscopic changes were investigated by SEM microscopy on a Tesla SEM microscope and the nanostructure was visualized using a JEOL JSPM 4210 Atomic Force Microscope.

The X-ray diffraction patterns resulted for the mechanically alloyed samples are presented in Figure 4.

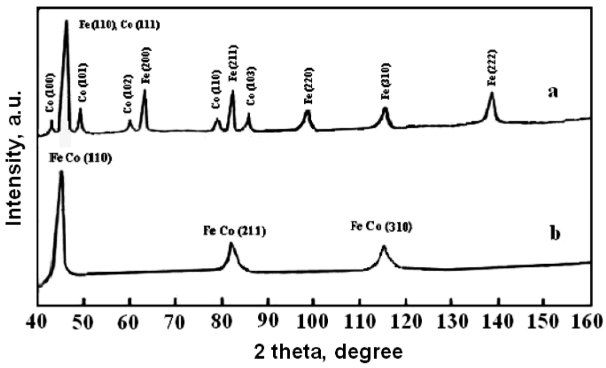


Fig. 4. X-ray diffraction patterns for Fe50Co50 % wt. composition subjected to mechanical alloying for: a) 1hour, and b) 16 hours.

The powder mixture milled for 1 hour presents very well developed diffraction peaks corresponding to the Fe and Co elementary powder. The peaks shape reveals a typical poly – crystal state of the both components.

The diffraction peaks are significant changed after 16 hours of milling, there appear broadened and less intense peaks corresponding to the new formed FeCo50 solid solution. The Co peaks disappear due to the assimilation of Co atoms into the Fe crystal lattice. The Co atom is slightly small than Fe atom. The Co atoms assimilation in Fe crystal lattice will shrink it proportionally with the quantity of assimilated Co atoms. The fact is proved by the slow decreasing of crystal parameter from 285.72 pm after initial stage to 285.38 to the final stage at 16 hours of milling.

The grain size for bulk alloys is calculated using Scherrer formula from the X-ray diffraction pattern using the peak width at half height [11]. The mechanically alloyed powders are stressed at the crystal lattice level. A proper grain calculation for mechanically alloyed powder must to count the residual stress. There are some mathematical methods to eliminate residual stress influence on the grain size resulted by X-ray calculation. In this paper we used the Williamson – Hall method [12], see equation

$$\beta \cos\theta = (k\, \lambda) / d + \eta\, \mathrm{sint}\theta, \qquad (6)$$

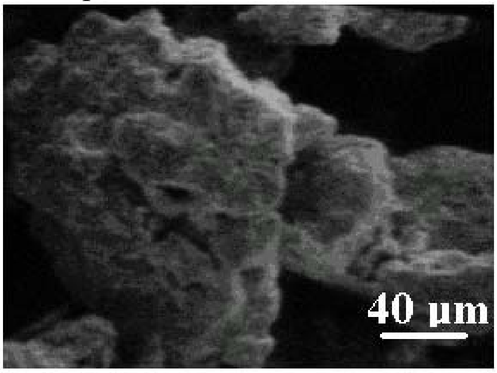
where: d – grain size; k – coefficient (between 0,9 – 1); λ – wave length of used X-ray; β – full width at half height FWHM; θ – Bragg diffraction angle. η – slope of $\sin\theta$ versus $\beta\cos\theta$ graph.

We calculate the grain size according to the Williamson Hall method. It results after 1 hour of milling 91.58 nm and after 16 hours of milling 51.73 nm. The final value corresponds to the middle of nano-crystalline range of mechanically alloyed powders.

The microscopic investigation feature larger particles after 1 hour of milling, Figure 5a. This is due to the intensive plastic deformation of Fe particles which embeds Co particles into a sandwich structure. The particle size is situated from 80 μm to over 200 μm diameters.

The corresponding nanostructure is observed in figure 5b. There appears deformation traces due to the severe impact between balls and powder particles.

The grains are flattened and their border presents flow marks. The flow marks prove the ductile state of the involved particles after first hour of milling. The grain size observed in Figure 5b is situated around of 120 nm, value in good agreement with the one obtained by Williamson Hall method from the X ray diffraction pattern.



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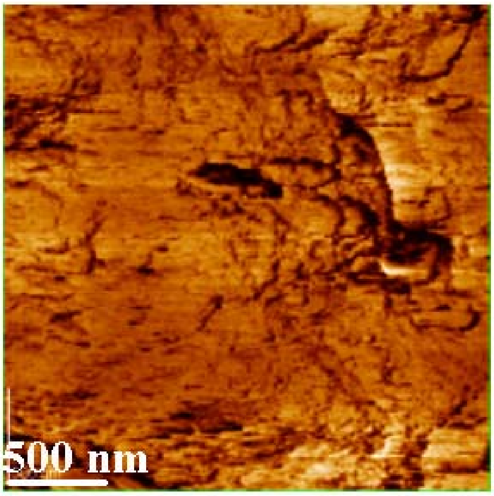
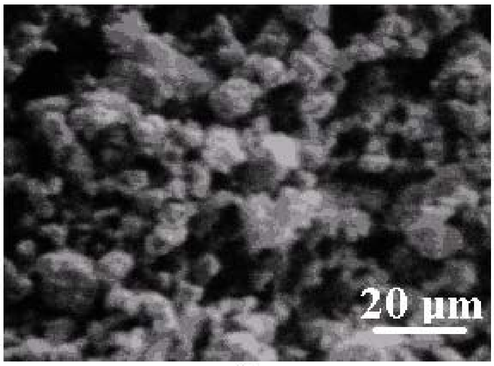


b

Fig. 5. Microscopic analysis for Fe50Co50% wt. composition milled 1 hour: a) SEM image and b) AFM phase image.

The situation is dramatically changed after 16 hours of milling. Refined powder particles are observed at SEM microscopically inspection, Figure 6a. The particle shape is equiaxially rounded with irregular surface caused by several cold welding and cold hardening cracks. Their average diameter is situated around of 12 µm. The particle size and shape is close related to the observed X-ay diffraction pattern in Figure 4b. It proves the homogeneous state of mechanically alloyed FeCo50 solid solution.

The nanostructure related to the FeCo50 solid solution is observed in Figure 6b. There appear FeCo50 nano grains with a equiaxial shape cold welded into a compact structure of the alloy particle. The microscopically observed average grain diameter is 56 nm, value in great concordance with the one resulted from the X-ray diffraction pattern.



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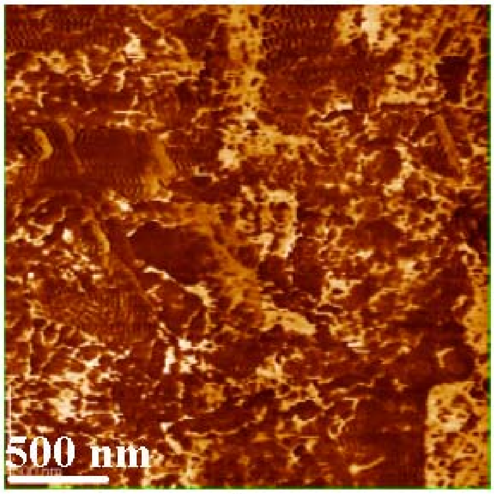

b

Fig. 6. Microscopic analysis for Fe50Co50% wt. composition milled 16 hours: a) SEM image and b) AFM phase image.

The ductile aspect of Fe powder after 1 hour of milling and the effective particle size reducing after 16 hours of milling are according to the ductile brittle mechanical alloying model [1, 3]. Similar behavior was observed for the other Fe – Co compositions milled on the other planetary ball mill with similar characteristics with our prototype [13, 14].

The observed results for FeCo50 wt. % mechanical alloying are similar with other reported in literature [6] but ten times faster. Finally, it results that our designed prototype is the times effective than other used planetary ball mills.

5. CONCLUSIONS

When designing this equipment we have taken into consideration, apart from the functional dimension, also the esthetic aspect. Thus the equipment has straight lines or rounded outline in order to avoid disequilibrium of esthetic nature. We also took care that the moving parts be inside the casing in order to protect the personnel using it against accidents. In the center of the containers appears an acceleration field of 20 g m/s^2 leading to the increased grinding efficiency and reduced grinding duration.

The prototype was tested intensively on a FeCo50 % wt. at several milling time in order to evidence the designed planetary ball mill efficiency. The X-ray diffraction pattern reveals that after 16 hours of milling the nano-crystalline state was achieved. The FeCo50% wt mechanical alloying process is realized according with the ductile- brittle alloying model.

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Stare nano-cristalină a FeCo50 obținută într-o moară planetară

Rezumat: *Necesitatea sistemelor de măcinare de inaltă energie conduce la dezvoltarea morilor cu bile planetare. Luând în considerare mecanismul planetar s-a proiectat şi realizat o moară planetară care se pretează măcinării pulberilor metalice. Acesta a fost testată intensiv pe un amestec pulverulent de FeCo50 % masă la diferiți timpi de măcinari. Analizele de difracție cu raze X şi investigațiile microscopice arată că după 16 ore de măcinare s-a atins stadiul nanocristalin. Observațiile făcute indică faptul că alierea mecanică a amestecului FeCo50 % masă decurge după modelul ductil – fragil.*

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