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Studies regarding the structural characteristics of sintered alloyed steels with nanometric master alloys

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In this paper are presented the results of the experimental research on the homogeneity of sintered alloyed steels made of Fe–Mn-Mo-Cr-B master alloy powders. Three types of mixtures containing Fe, Mn, Mo, Cr and B were elaborated by mechanical alloying (MA). The particle size of the powders was about 600 nm. After MA the samples were uniaxially compacted at 600, 700, 800 MPa and sintered at tree different temperatures (1050, 1100 and 1150 °C) in argon atmosphere. The experimental results pointed out that the steels with nanometric master alloys have higher density and structural homogeneity comparative with those obtained by micronical powders. The steel obtained from the homogeneous mixture, sintered at 1150 °C presents a ferrite-pearlite structure (corroborated with 243 $HV_{0.2}$ respectively 193 $HV_{0.2}$ hardness). Structural constituent of AM20 steel, sintered at 1150 °C presents a pearlite-bainite structure (corroborated with 290 $HV_{0.2}$ hardness). MA40 steel sintered at 1150 °C/60 min, presents a pearlite-bainite structure (corroborated with 376 $HV_{0.2}$ hardness).

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1. Introduction

In conventional powder metallurgy steels, copper, molybdenum, nickel are common alloying elements due to their low oxygen affinity, and good mechanical properties are obtained in the sintered steels made from partially diffusion alloyed Fe–Cu–Mo–Ni powders [1-4]. But the use of the elements mentioned above as powders has disadvantages because on the one hand they are expensive and on the other hand some of these, such as Ni are dangerous for human health and environment [5].

Using master alloy technique, alloying elements such as vanadium, manganese and silicon, which have higher oxygen affinity, can be easily introduced into sintered steels. Typical alloys produced by master alloy technique are Fe–Mo–Cr–Mn–C and Fe–Mo–V–Mn–C [6-8]. To prevent the master alloy powders from oxidisation, a high content of carbon is needed, and such master alloys give a transient liquid phase sintering, which accelerates diffusion of the alloying elements and densification. The properties of the sintered steels containing master alloy powders depend strongly on sintering conditions, such as the type and sintering atmosphere.

The objective of the current research is represented by the development and elaboration of some new technological processes for sintered ecological alloyed steels, with the elimination of Ni and Cu as alloying elements to assure ecological conditions for steel fabrication along with the fabrication costs reducing for these materials and assuring some remarkable using characteristics.

So have been researches conducted by replacing Cu and Ni with Mn, Mo and Cr, adding B in proportion of 0.5%, in order to facilitate sintering with the presence of the liquid phase. It was shown that transient liquid phase sintering accelerates the sintering process, which leads to improved mechanical properties [9].

The aim of the present work was to study the effect of Fe–Mn–Mo-Cr-B master alloy made by different particle size, sintering temperature and time on the mechanical properties and sintering behaviour of Fe–Mn–Mo-Cr-B master alloy steels and to get improved mechanical properties in Fe–Mn–Mo-Cr-B steels [10].

The use of these alloying elements has been carried out because of the following reasons [9, 11, 12-15]:

- manganese is one of the alloying elements which contribute to the augmentation of the steels hardenability;
- molybdenum is one of the alloying elements which contributes to the augmentation of the hardenability of steels;
- chromium because it contributes to the augmentation of the steel hardenability, is cheap being a good replacement for Ni, which is employed recently in order to improve the mechanical properties of sintered steels.
- boron favors the appearance of the liquid phase during sintering, thus contributing to the densification of the sintered steel.
- graphite powder represents the carbon source in order to obtain steel. Carbon is the most important alloying element of the iron because for each 0,1% C the resistance to breakage of carbon steels increases with about 9 daN/mm^2, and the yield point with about 4 ÷ 5 daN/mm^2.

2. Experimental details

In order to obtain through mechanical alloying of nanometric master alloys, alloying of iron powders have been carried out, micronic, with elementary powders of alloying elements (purchased from Goodfellow Cambridge Limited Company), that is graphite powders with the chemical composition presented in the table below.

Table 1. Chemical composition of master alloys / materials symbol.

Master alloys /materials symbol	Complex constitution	Chemical composition [%]					
		Mn	Mo	Cr	B	Graphite (C)	Fe
HM	[Fe-3Mn-1Mo-1,5Cr-0.5B-0,45C] homogenous mixture	3	1	1,5	0,5	0,45	Bal.
MA20	[Fe-3Mn-1Mo-1,5Cr-0.5B-0,45C] mechanical alloyed 20 h	3	1	1,5	0,5	0,45	Bal.
MA40	[Fe-3Mn-1Mo-1,5Cr-0.5B-0,45C] mechanical alloyed 40 h	3	1	1,5	0,5	0,45	Bal.
MA60	[Fe-3Mn-1Mo-1,5Cr-0.5B-0,45C] mechanical alloyed 60 h	3	1	1,5	0,5	0,45	Bal.

The samples were obtained from micronic iron powders and alloying elements (manganese, molybdenum, chromium, boron and graphite) with code **HA** - homogenous mixture. The mixtures were subjects of the MA process for 20 to 60 hours. Each sample has the code function the MA time such as **MA20** for 20 hours of MA, **MA40** for 40 hours of MA respectively **MA60** for 60 hours of MA.

Also, in order to prevent granules from clinging to milling balls and to the walls of working areas, that is in order to prevent powders from agglomerating during the process of mechanical alloying, 1% of zinc stearate has been introduced.

Mechanical alloying has been carried out in ball mill Pulverisette 6 with the following working parameters:

- mill speed: 550 rot/minute;
- changing direction of rotation every 5 minutes;
- loading mill bolus with 100 g of powder and 50 balls of inoxidable steel with ϕ10mm;
- grinding times: 20, 40, 60 h;
- grinding environment: dry, in the presence of argon.

From 20 to 20 hours ware taken samples to study the influence of the MA time on the morphology and homogeneity of the samples.

The distribution of the alloying elements was studied by SEM and EDS using a JEOL 5600 LV microscope. The particle size distribution was studied using BROOKHAVEN 90 PLUS BI-MAS apparatus. Phase composition of mixtures of powders resulted after mechanical alloy was determined by X-ray diffraction using a difractometer Bruker-AXS type D8 ADVANCE with Mo tube, 40kV / 40 mA, Zr filter and wavelength 0.70930Å.

In Fig. 1 below there is a SEM microstructure, and in Fig. 2 the maps of iron distribution maps and the distribution of alloying elements of sintered steels obtained from master alloy powders, resulted through mechanical alloying during 60 hours.

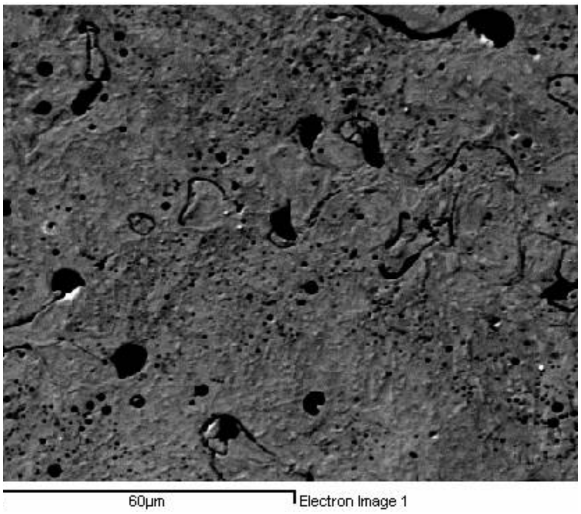
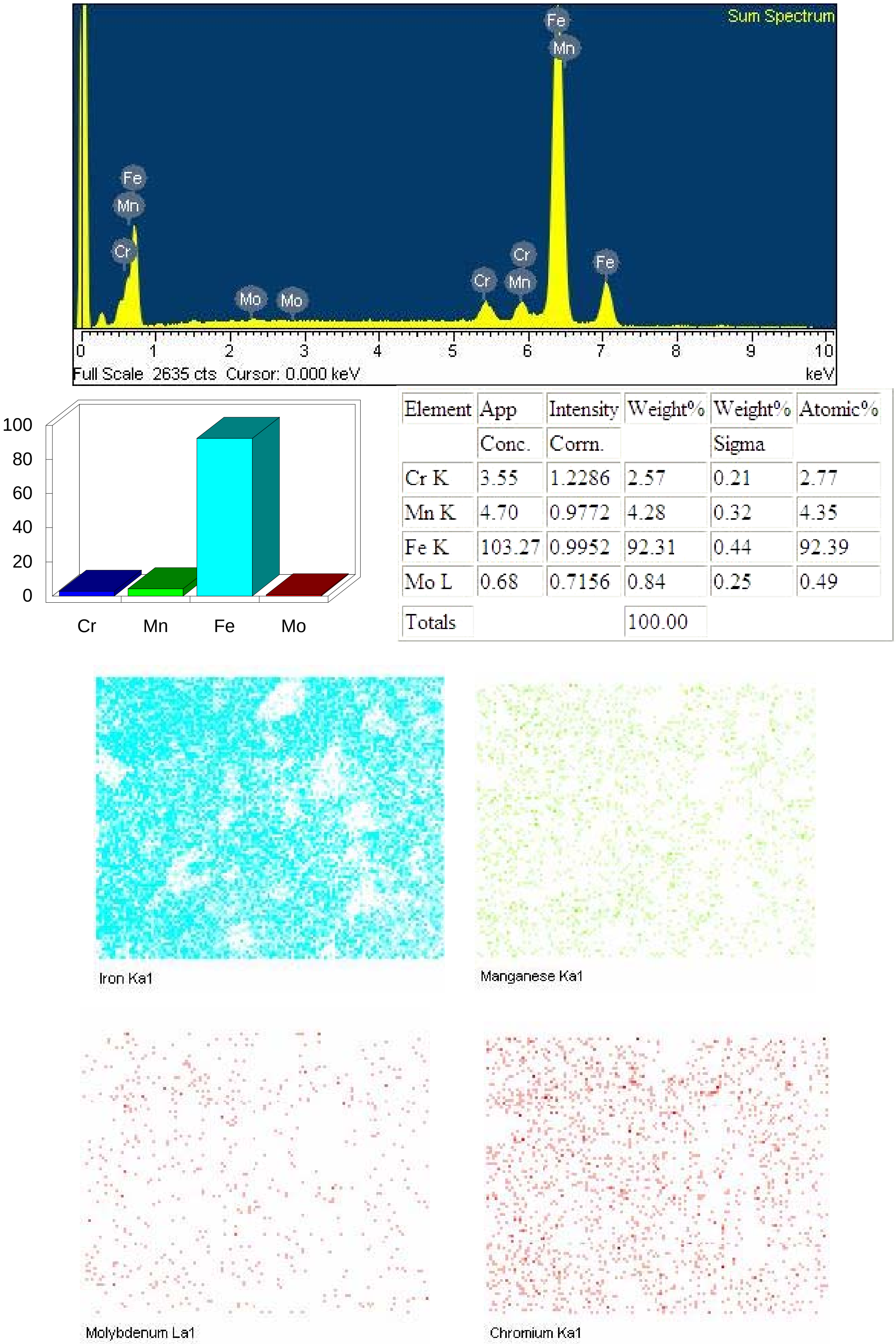


Fig. 1. MA60 (SEM) microstructure aspect.



Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
Cr K	3.55	1.2286	2.57	0.21	2.77
Mn K	4.70	0.9772	4.28	0.32	4.35
Fe K	103.27	0.9952	92.31	0.44	92.39
Mo L	0.68	0.7156	0.84	0.25	0.49
Totals			100.00		

Fig. 2. Iron and alloying elements distribution maps on the same surface of MA6 steel.

Compaction of powders has been carried out through unilateral pressing in the matrix with 10 mm diameter, with compaction pressures of 600, 700, 800 MPa.

After MA process the samples were sintered at different temperature (1050, 1100 and 1150 ºC) in argon atmosphere. Dimensional change and sintering density were measured. Mechanical properties were evaluated in as-sintered condition: hardness and friction coefficient. Finally, a micro structural study was carried out.

3. Results and discussions

In this paper are presented the results of the experimental research on the structural characteristics of sintered alloyed steels.

In terms of microstructure prepared metallographic unchallenged evidence was found that lack porosity (especially samples mechanically alloyed mixtures obtained from 40 to 60 hours) is a consequence of the conditions of preparation (mechanical alloying, annealing, pressing and sintering) (Fig. 3).

For the microscopic analysis, the samples were encapsulated in resin and then were metalographical prepared and etched with 1% nital. In the following pictures are presented the microstructures of the samples function the milling time (Fig. 4).

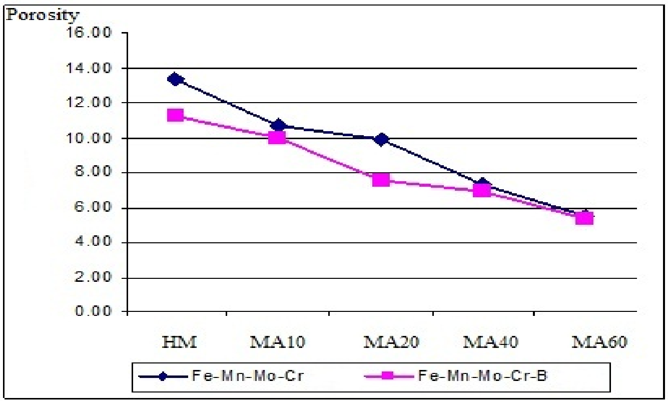
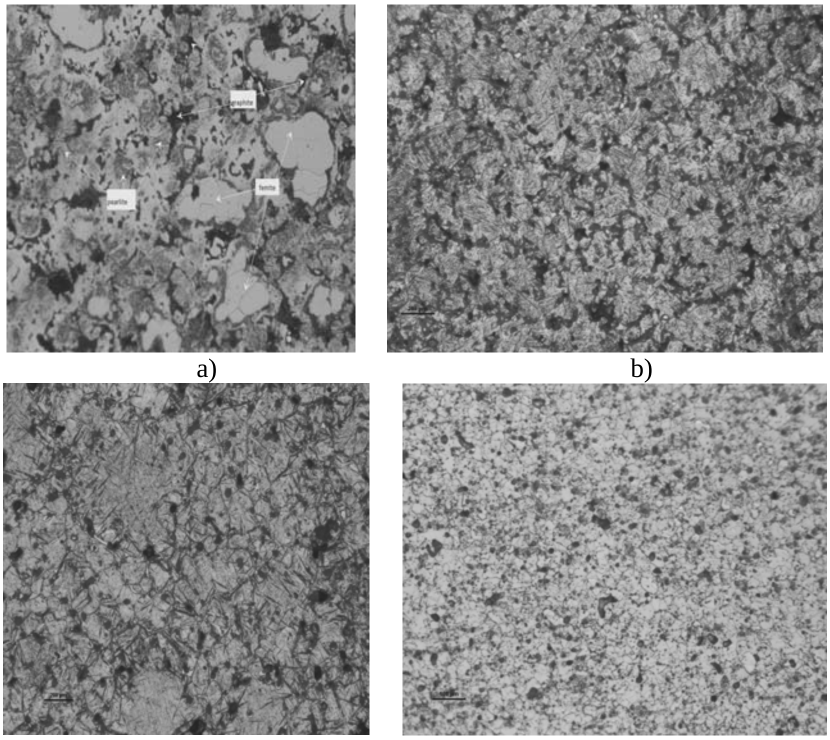


Fig. 3. The evolution of volume contraction of materials was compacting at 800 MPa, sintered at 1150 °C/60 min.



a) b)

c) d)

Fig. 4. Microstructure of materials was uniaxial compacting at 800 MPa, sintered at 1150 °C/60 min a) HM, b) MA20, c) MA40, d) MA60.

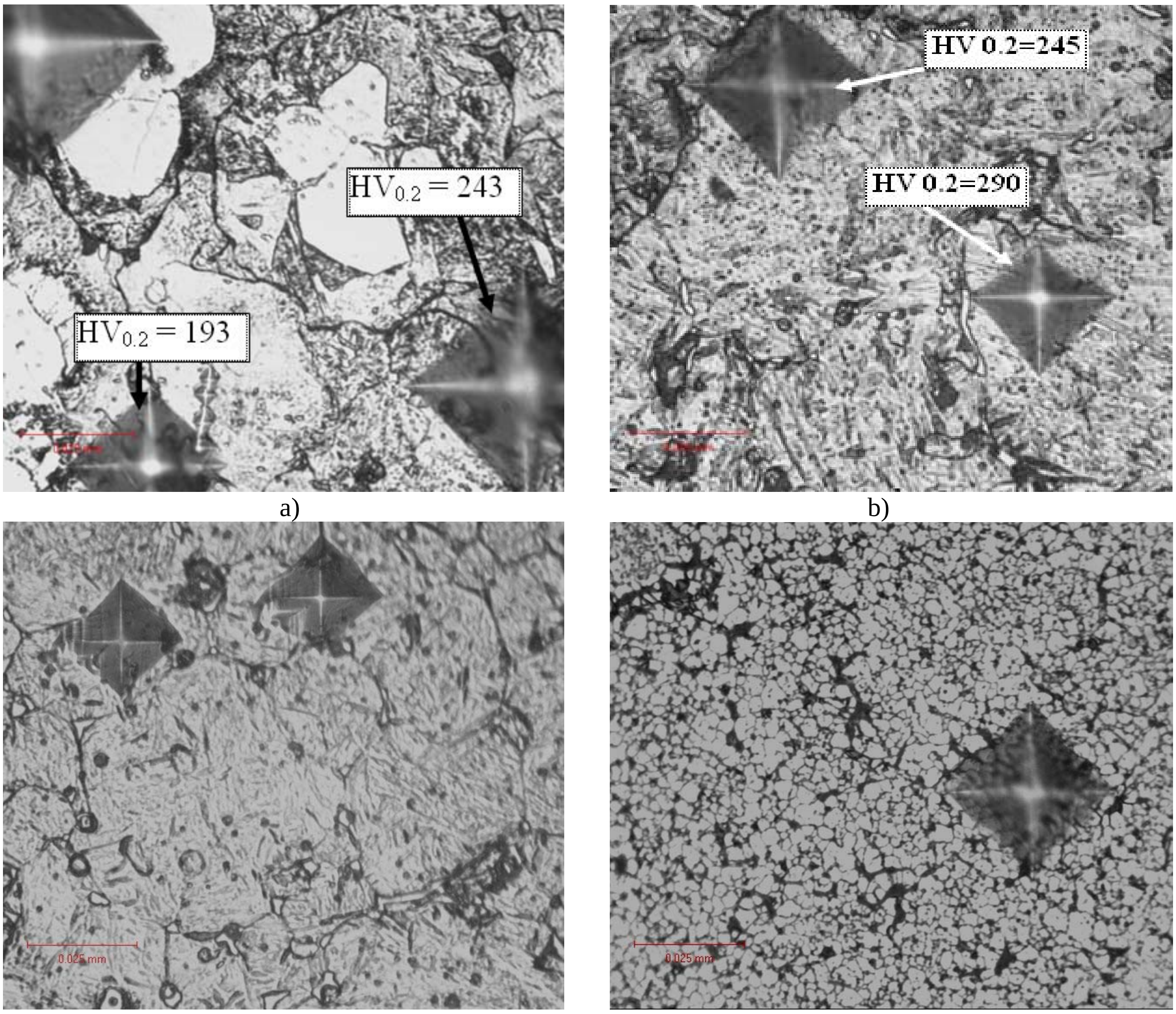
Further has been studied also the influence of nanometric granulation of powders on steel microhardness and friction coefficient evolution. Results have presented in Fig. 5, Table 2 and Table 3.

The steel obtained from the homogeneous mixture, sintered at 1150 °C/60 min (Fig. 5a) presents a ferrite-pearlite structure (corroborated with 243 $HV_{0.2}$ respectively 193 $HV_{0.2}$ microhardness).

Structural constituent of AM20 steel, sintered at 1150 °C presents a pearlite-bainite structure (corroborated with 290 $HV_{0.2}$ microhardness - Fig 5b).

MA40 steel sintered at 1150 °C/60 min, presents a pearlite-bainite structure (corroborated with 376 $HV_{0.2}$ microharness – Fig. 5c).

Vickers microhardness was measured using a CV-400 AAT microhardness tester, with a load of 25 gf and 10 s for holding time (Fig. 5).



a) b)

c) d)

Fig. 5. Hardness /Microstructure of materials was compacting at 800 MPa, sintered at 1150 °C/60 min a) HM, b) MA20, c) MA40, d) MA60.

Table 2. The evolution of the hardness depending on the compaction pressure and the sintering temperatures.

Sintering temperature [°C]	Sample	Vickers microhardness HV [kgf/mm²]		
		compacted pressure of 600 MPa	compacted pressure of 700 MPa	compacted pressure of 800 MPa
1050	MA60	221	243	274
1100		268	311	331
1150		385	398	417

Table 3. The evolution of the friction coefficient of elaborated steels (800 MPa compaction pressure and 1150° C sintering temperature).

T ºC	Steels used for tribologic testing	µ (samples compacted at 600 MPa)	µ (samples compacted at 700 MPa)	µ (samples compacted at 800 MPa)
1150	HM	0.597	0.579	0.515
	MA 20	0.319	0.282	0.263
	MA 40	0.283	0.246	0.200
	MA 60	0.200	0.237	0.158

4. Conclusions

The granulation evolution of composite powders with the Fe-3Mn-1Mo-1.5Cr-0,5B-0.45Graphite, is different, meaning that granulation decreases together with the increase in mechanical alloying time. Is observed that at 60 hours mechanical alloying time, the powders reach around 600 nm [10].

The microstructures lead to the following conclusions:

- the main parameter of the sintering process is temperature;
- microstructures of the steels obtained from mixtures containing 3%Mn; 1%Mo; 1,5%Cr; 0,5%B and 0,45 graphite have the homogeneity dependent to the MA time;
- from the point of view of the nature and the granulation of the powder mixtures, the best values of the friction coefficient are held by the steels elaborated from mixtures of powders resulted from MA for 60 hours.

Depending on the sintering temperature, respectively on the compaction pressure, it can be seen that in all cases, regardless of the nature of the powder mixtures used, the friction coefficient decreases with the growth in compaction pressure.

Elaboration of steels using nanometric master alloys is useful to fabricate some diminutive parts such as gears and axes by µMIM technique. These parts are used to fabricate some MEMS (actuators) and at the macroscopically level in the automobile industry at the fabrication of the gears and the synchrony hubs of the gear boxes [16-19].

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