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THE ANALYSIS OF THE TECHNOLOGIC PARAMETERS INFLUENCE IN OBTAINING THE ALLOYED SINTERED STEELS BY TWO-STEPS SINTERING

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Abstract. In the paper there are presented the results of the experimental research for elaborating *Mn, Mo, Cr, B and Graphite* alloyed steels. There were elaborated mixings with 3% Mn and Fe, 1.5% Mo, 1% Cr and 0.5% B, which have experienced a 20, 40 and 60 hours mechanical alloy and produced nanocomposite mixings with powder grains of about 300nm. In this sense there were compactized samples at 600, 700, 800 MPa. The samples were heated in argon, at 1250 ºC, then in the same cycle the temperature were reduced at 1100 ºC, and kept 60 minutes. There were analyzed both the samples obtained by two-steps sintering and the samples obtained by classic sintering. The experimental results pointed out that the nanostructured steels obtained by two-steps sintering have an increasing of the density, structural homogeneity and mechanical properties (hardness, friction coefficient), comparing to the classical sintering. There is also presented the optimization of the technological process of elaborating the alloyed steels. In this purpose it is used the factorial experiment and is established that the polynomial model of degree 2 is optimal for estimating the output surface corresponding to the required experimental element. The results of the graphical data processing using the soft STATISTICA 8.0 proved interesting information concerning the variation of the mechanical characteristics of the alloyed steels.

Keywords: mechanical alloy, nanocomposite, alloyed steels, two-steps sintering, polynomial optimization

1. Introduction

Sintered steels alloyed of powders containing Mo, Cr, Ni, Cu are most often used for sintered samples, which have remarkable mechanical features.

According to the works written in the domain of powders metallurgy, the most often used elements are Ni and Cu as alloying elements for creating alloyed sintered steels by means of Powder Metallurgy especially because their lack of affinity towards oxygen which constitutes a major advantage in the process of sintering [1, 2, 3]. 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 [4].

Producing sintered steels out of the homogeneous alloy of iron powders and alloying elements (manganese, molybdenum, chromium, boron and graphite) has disadvantages especially from the point of view of the chemical and structural non-homogeneity of these steels.

In addition, the process of sintering influences the granulation and implicitly the mechanical characteristics of sintered steels.

Therefore, nowadays the research is performed in order to find new methods and techniques of processing sintered steels.

In order to control the granulation of alloyed steels during sintering, a new method is adopted nowadays, called the sintering in two steps [5, 6].

Research has been conducted in 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.

The use of these alloying elements has been carried out because of the following reasons [7]:

- manganese is one of the alloying elements which contribute to the augmentation of the steels hardenability; depending on the concentration it determines the almost linear augmentation of the resistance to traction and hardness;

- molybdenum is one of the alloying elements which contributes to the augmentation of the hardenability of steels; depending on the concentration it determines the almost linear augmentation of the resistance to traction and hardness. Mo forms carbides of the type of penetration phases (MoC, Mo2C, Mo6C), even when molybdenum is in small quantity (0.5%), being twice more carburigen than W. Where quantities of Mo are of 0.5%, the speed of pearlitic transformation reduces and thus hardenability increases. Molybdenum in quantities below 2.5% reduces the temperature of martensitic transformation and decreases the softening tendency to recovery of the hardened steel, even when Mo is in quantities of 0.2%;

- 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. When alloying with B the relation B/C is important for improving the mechanical properties;

- 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 90 N/mm^2, and the yield point with about 40 ÷ 50 N/mm^2.

2. Experimental procedure

In order to obtain through mechanical alloying of composite nanopowders, alloying of iron powders have been carried out (ANCORSTEEL 1000B brand), micronic, with elementary powders of alloying elements, that is graphite powders with the chemical composition presented in the table below.

Table 1. Powder alloyings composition

Mixture symbol	Complex constitution	Chemical composition [%]					
		Mn	Mo	Cr	B	Graphite (C)	Fe
MA 0	[Fe-3Mn-1Mo-1.5Cr-0.45 C] $_{\text{mechanical alloyed 0h}}$	3	1	1.5	-	0.45	Bal.
MA 20	[Fe-3Mn-1Mo-1.5Cr-0.45C] $_{\text{mechanical alloyed 20h}}$	3	1	1.5	-	0.45	Bal.
MA 40	[Fe-3Mn-1Mo-1.5Cr-0.45C] $_{\text{mechanical alloyed 40h}}$	3	1	1.5	-	0.45	Bal.
MA 60	[Fe-3Mn-1Mo-1.5Cr-0.45C] $_{\text{mechanical alloyed 60h}}$	3	1	1.5	-	0.45	Bal.
MA+B 0	[Fe-3Mn-1Mo-1.5Cr-0.5B-0.45C] $_{\text{mechanical alloyed 20h}}$	3	1	1.5	0,5	0.45	Bal.
MA+B 40	[Fe-3Mn-1Mo-1.5Cr-0.5B-0.45C] $_{\text{mechanical alloyed 20h}}$	3	1	1.5	0,5	0.45	Bal.
MA+B 40	[Fe-3Mn-1Mo-1.5Cr-0.5B-0.45C] $_{\text{mechanical alloyed 40h}}$	3	1	1.5	0,5	0.45	Bal.
MA+B 60	[Fe-3Mn-1Mo-1.5Cr-0.5B-0.45C] $_{\text{mechanical alloyed 60h}}$	3	1	1.5	0,5	0.45	Bal.

MA – mechanical alloyed

In addition, 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.

Compaction of powders has been carried out through unilateral pressing in the matrix with 10.5mm diameter, with compaction pressures of 600, 700, 800 MPa.

During research, it has been taken into consideration the adopting of a special sintering technique in order to create controlled granulations, which favor a series of characteristics depending on the structural grains dimension.

Consequently, it has been chosen a termic cycle of sintering represented in the figure below.

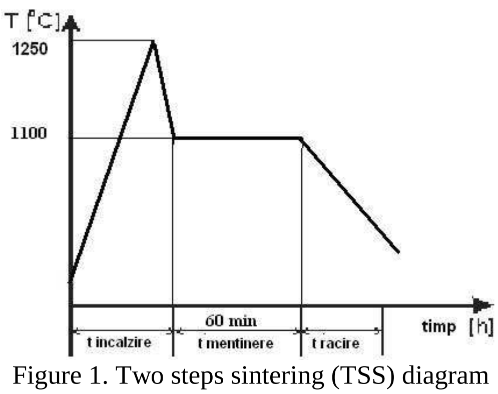

Figure 1. Two steps sintering (TSS) diagram

As it has been shown, the heating of samples has been carried out in argon at 1250 ºC, followed by a fast cooling at 1100 ºC and minting it at this temperature for 60 minutes. The cooling toward the ambient temperature has been carried out with about 30 C/minute.

3. Experimental results

The densities of the sintered samples were determined, their evolution being presented in the graphics from the Figure 2 - 4.

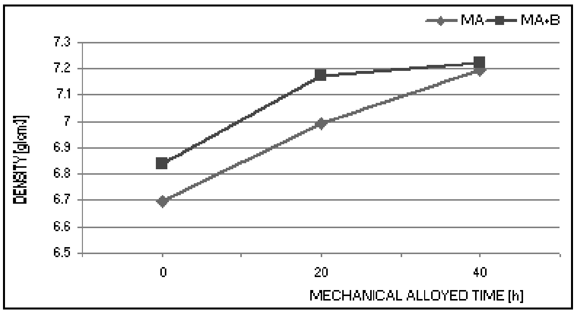

Figure 2. Density vs. particle size for TSS samples at 60 min. dwell time, compacted at 800 MPa

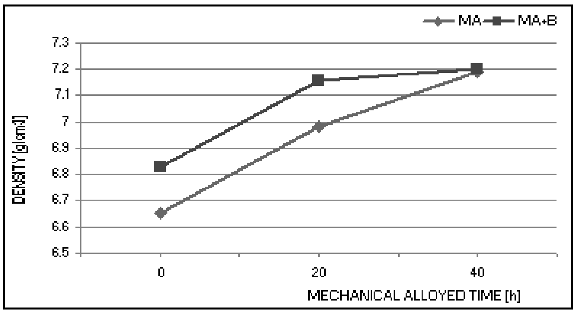

Figure 3. Density vs. particle size for TSS samples at 60 min. dwell time, compacted at 700 MPa

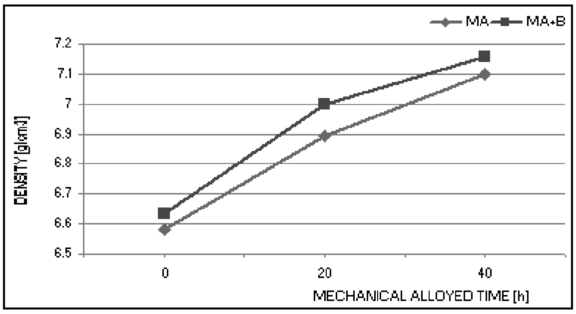

Figure 4. Density vs. particle size for TSS samples at 60 min. dwell time, compacted at 600 MPa

Taking into account the most efficient samples in relation to density, porosity and volumetric contraction, after sintering there were submitted to analysis by marking iron distribution maps and those of alloying elements, sintered alloyed steels samples obtained from elemental powder alloys mechanically alloyed in 60 hours and containing B.

In figure 5, there is a SEM microstructure, and in figure 6, the maps of iron distribution maps and the distribution of alloying elements of sintered steels obtained from composite powders containing B, resulted through mechanical alloying of elemental powder alloys mechanically alloyed during 60 hours.

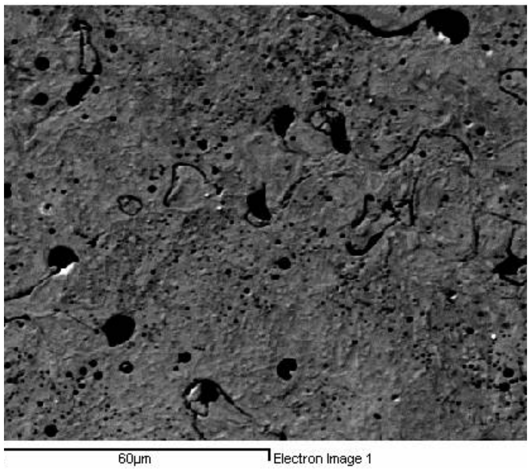

Figure 5. AM60+B (SEM) microstructure aspect

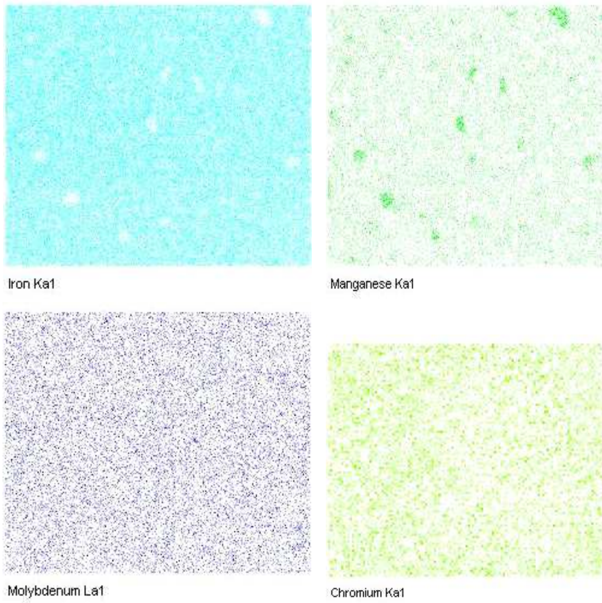

Figure 6. Iron and alloying elements distribution maps on the same surface of AM60+B steel

It has been studied also the influence of nanometric granulation of powders on steel hardenability evolution, and in this case there have been carried out two procedures of classical sintering (CS), in two steps (TSS).

In order to achieve that, there has been measured Vickers hardness of sintered steels obtained from nanostructural composite powders Fe-Mn-Mo-Cr-graphite with B and without B obtained through mechanical alloying during 60 hours. Results are presented in table 2.

Table 2. Evolution of hardness depending on the pressure compactizare respective temperatures steels developed for sintering

Temp. [^oC]	Complex constitution	Hardness HV0.2		
		Compaction 600 MPa	Compaction 700 MPa	Compaction 800 MPa
CS 1050	[Fe-3Mn-1Mo-1.5Cr-0.45C] $_{\text{mechanical alloyed 60h}}$	227	248	258
	[Fe-3Mn-1Mo-1.5Cr-0.5B-0.45C] $_{\text{mechanical alloyed 60h}}$	221	243	274
CS 1100	[Fe-3Mn-1Mo-1.5Cr-0.45C] $_{\text{mechanical alloyed 60h}}$	249	264	286
	[Fe-3Mn-1Mo-1.5Cr-0.5B-0.45C] $_{\text{mechanical alloyed 60h}}$	268	311	331
CS 1150	[Fe-3Mn-1Mo-1.5Cr-0.45C] $_{\text{mechanical alloyed 60h}}$	271	284	298
	[Fe-3Mn-1Mo-1.5Cr-0.5B-0.45C] $_{\text{mechanical alloyed 60h}}$	307	331	342
TSS 1250→1100	[Fe-3Mn-1Mo-1.5Cr-0.45C] $_{\text{mechanical alloyed 60h}}$	**304**	**338**	**383**
	[Fe-3Mn-1Mo-1.5Cr-0.5B-0.45C] $_{\text{mechanical alloyed 60h}}$	**385**	**398**	**417**

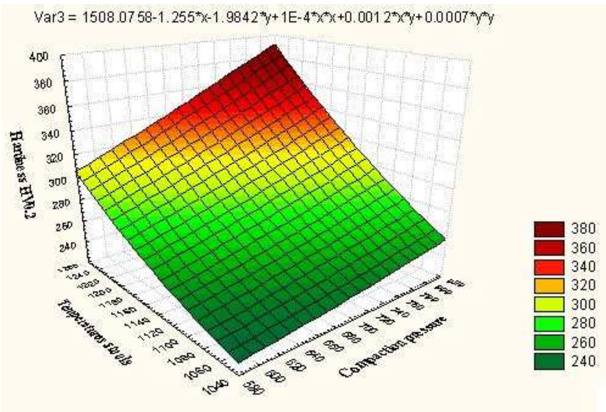

Figure 7. Evolution of hardness depending on the compaction pressure respective temperatures steels with B developed for sintering

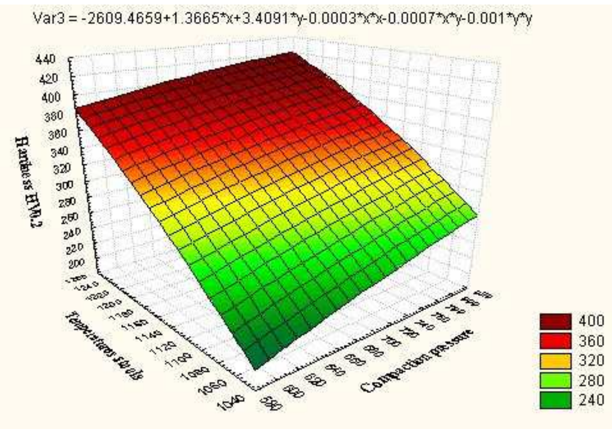

Figure 8. Evolution of hardness depending on the compaction pressure respective temperatures steels with B developed for sintering

After analyzing these data, it can be observed that the steels hardnesses with B are superior to those without B.

Regarding to the influence of the two sintering procedures, it has been observed an important augmentation of the hardness of steels processed through two-steps sintering in comparison to those processed through classical sintering.

This aspect confirms the fact that, as it has been shown at precedent paragraph some of the mechanical characteristics, such as hardness of steels, are preferred by the refined granulations and implicitely by the structural homogeneity of steels.

The samples elaborated according to the proceeding described above underwent tribologic tests using a tribometer type TRN 01 – 02541 from CSM Instruments – Switzerland. The working parameters were:

- Working method: linear testing;
- Normal force: 2 N;
- Amplitude: 5 mm;
- Linear speed: 1 cm/s; testing type: dry;
- The action on the sample is made with a ball with the diameter of 6 mm from 100CR6 steel.

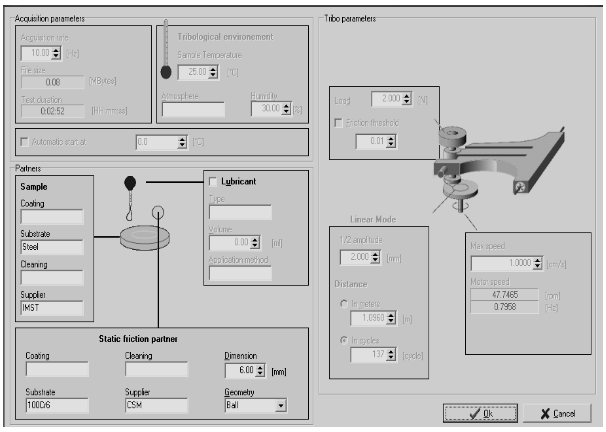

Figure 9. Tribometer parameters

The variation of the friction coefficient of steels obtained through pressing and classical sintering / two-steps sintering can be observed in table 3, figures 10 and 11.

Table 3 The evolution of the friction coefficient of the processed steels in function of the compaction pressure and sintering temperature

T ºC	Steels used for tribologic testing	μ (compaction samples at 600 MPa)	μ (compaction samples at 700 MPa)	μ (compaction samples at 800 MPa)
Classical sintering at 1150 ºC	MA 0	0.674	0.655	0.608
	MA+B 0	0.597	0.579	0.515
	MA 20	0.372	0.3.45	0.333
	MA+B 20	0.319	0.282	O.263
	MA 40	0.272	0.255	0.219
	MA+B 40	0.283	0.246	0.200
	MA 60	0.268	0.242	0.179
	MA+B 60	**0.200**	**0.237**	**0.158**
Two-steps sintering 1250→1100 ºC	MA 60	0.230	0.224	0.176
	MA+B 60	**0.189**	**0.145**	**0.116**

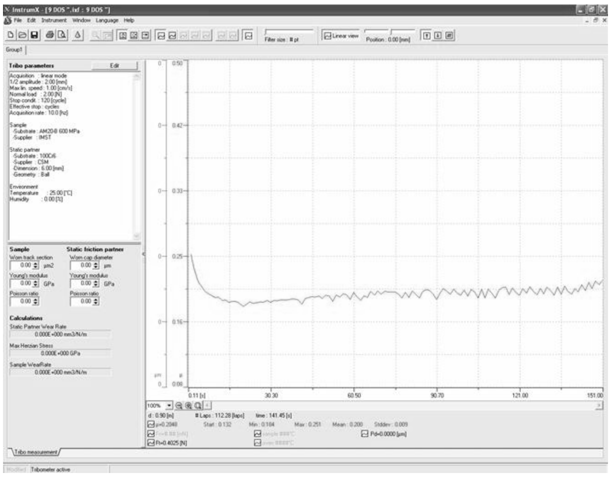

Figure 10 Friction coefficient of the MA+B 60 steel sintering at 1150 ºC

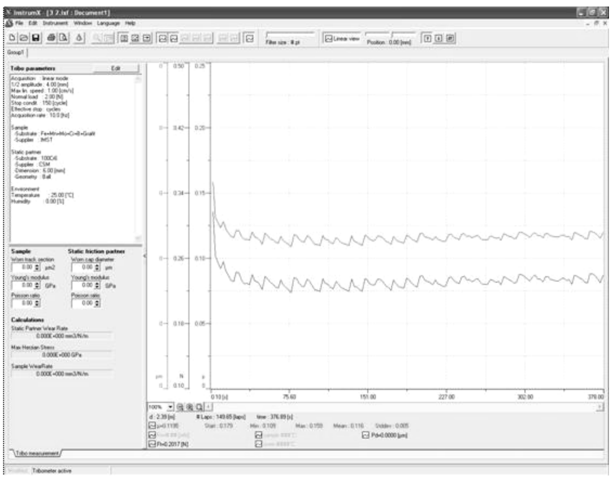

Figure 11 Friction coefficient of the MA+B 60 steel two steps sintering at 1250→1100 ºC

Since sintered steels at 1150 ºC exhibited densities and hardnesses with values superior to those sintered at 1050 and 1100 ºC, the tribologic testing has been carried out on steel samples obtained from alloys of homogeneous powders but also from mechanical alloying during 10, 20, 40 hours, classically sintered at 1150 ºC, and for AM60 steels with or without B addition – processed from alloys of nanometric powders. It has been chosen the two-steps sintering technique.

4. Conclusions

The results of experimental research presented in this work allow drawing the following conclusions:

According to the measurements carried out on samples sintered in two-steps, one can observe that important differences appear in comparison to the same samples sintered by means of classical procedure, as follows [8]:

- sintered samples density increases along with the times of mechanical alloying, thus the highest values about 7.4%/$cm^3$are obtained in case of powders processed through mechanical alloying for 60 hours, while classically sintered samples reach 7.2 g/cm^3 for powders resulted from mechanical alloying for 20 hours;

- the use of mechanically alloyed powders for a long time in order to process alloyed sintered steels is favourable from the point of view of chemical and structural homogeneity, and consequently from this point of view two-steps sintering is a good option;

- in comparison to classical sintering the density of sintered samples records values that are continually increasing while increasing mechanical alloying time in the case of two-steps sintering;

- from the point of view of technological processing parameters, according to the data contained in the table, one can notice that the values of the friction coefficient decrease along with the increase of the compactisation pressure, irrespective of nature and granulation of powders used for their manufacturing;

- From the point of view of adopted sintering procedures, one can determine that during two-steps sintering the friction coefficient is much smaller 0.116 for the steel processed from composite nanopowder with B (AM60+B) in comparison to the value of the friction coefficient of 0.158 of the same steel, but obtained through classical sintering.

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