

# Heat treatment applied to carbon steels sintered powders of Fe and Fe<sub>3</sub>C powder obtained by mechanical alloying

M. CIOBANU, C. NICOLICESCU, I. G. BUCSE, I. STEFAN

\*University of Craiova, Faculty of Mechanical, Department Engineering and Management of the Technological Systems, Drobeta Turnu Severin. Mehedinti, Romania

This paper presents the results of experimental research on thermal treatment of carbon steels sintered and much more, development Fe + Fe<sub>3</sub>C powder using mechanical alloying. It was analyzed the influence of MA time on the morphology of the particles of micrometer/nanometer powders by electron microscopy SEM. The chemical composition of the carbon steel sintered powder was studied using the EDAX analysis. Followed by heat treatment to improve the properties of carbon steels sintered hardness and strength.

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*Keywords:* sintered carbon steels, powder metallurgy, mechanical alloy

## 1. Introduction

The materials elaborations by performance technologies represent one of the priority objectives in scientific research of materials science.

Formation of powdered metal parts by die pressing process is most often used because it has a high productivity and can be adapted easily to the production profile changes. Powder pressing plays an important role in the flow chart of parts made by Powder Metallurgy (PM) technologies.

Cementite or iron carbide is a chemical compound with the formula Fe<sub>3</sub>C (or Fe<sub>2</sub>C:Fe), and an orthorhombic crystal structure. It is a hard, brittle material, normally classified as a ceramic in its pure form, though it is more important in metallurgy.

For research cementite was obtained by direct carburization of Fe powder.

The original elements are obtaining and using the Fe<sub>3</sub>C powder into the homogeny mixture of Fe+Fe<sub>3</sub>C in order to obtain sintered carbon-steels. Using cementite powder mix with Fe powder is to ensure a better compaction by pressing, less time to decompose and C diffusion into the Fe network in remarkable chemical homogeneity conditions.[1]

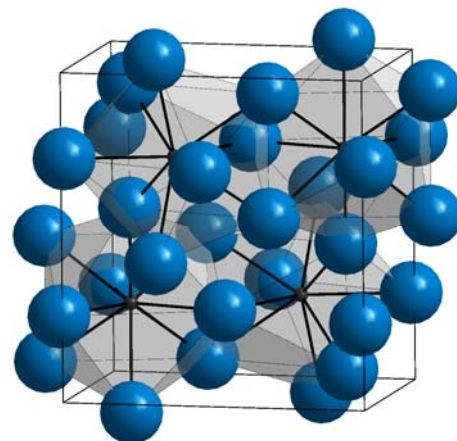


Fig. 1 Orthorhombic Fe<sub>3</sub>C. Iron atoms are blue [8]

## 2. Experimental details

For the research used Fe powder produced by atomization, sorted ANCORSTEEL 1000B, 6086 code produced SC Ductile S.A. Buzau and powder / nanopulbere of Fe<sub>3</sub>C.

Fe powder characteristics are shown in Tables 1, 2, 3 respectively in the graphs in Figures 2 a) and b).

Table1. Technological characteristics of the powder ANCORSTEEL 1000B, code 6086 [46]

Particle size distribution (µm)	-250 / 150	-150 / +45	-45
%	12	67	21
Hall apparent Density, g / cm <sup>3</sup>	2,92		
Hall Flow Rate, s / 50g	26		
Density, pressed, g / cm <sup>3</sup>	6,8		

Table 2 The chemical composition of the powder 1000B [46]

Compoziție	C	O	N	S	P	Si	Mn	Cr	Cu	Ni
[%]	<0.01	0.09	0.001	0.009	0.005	<0.01	0.10	0.03	0.05	0.05

Table 3 Fe distribution obtained by sieving, sorting ANCORSTEEL 1000B.

Sieve size (mm)	Quantity (g/%)
0.4	0.006
0.25	0.003
0.2	0.15
0.315	0.028
0.125	87.654
0.1	5.86
0.8	2.2
0.071	0.604
	3.215

The physico-mechanical properties of samples prepared from Fe powder ANCORSTEEL 1000B are shown in the following figures:

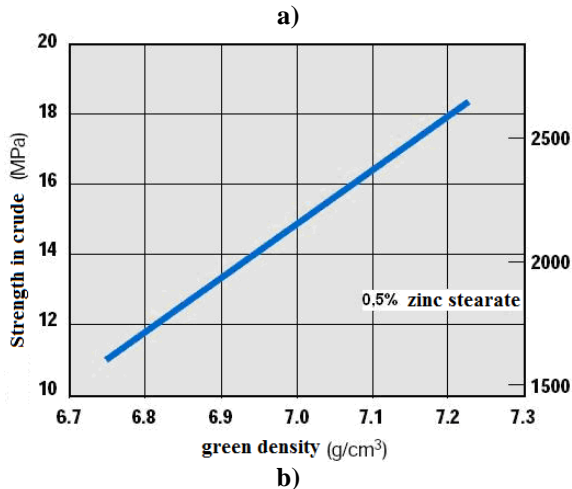
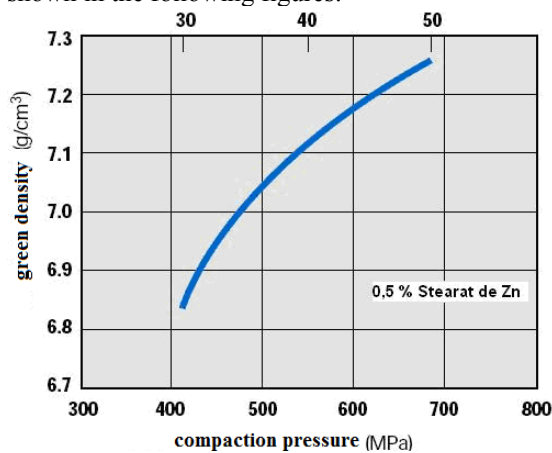


Fig. 2 The physico-mechanical properties of powder ANCORSTEEL 1000B[4]

The composition of powder grains was obtained by diffractometric analysis with a Bruker-AXS diffractometer of D8 ADVANCE type, X-ray tube - Mo anode, 40kV / 40 mA, Zr kb filter, 0.70930Å wave-length.

The diffractogram by powders ANCORSTEEL 1000B in Fig. 3

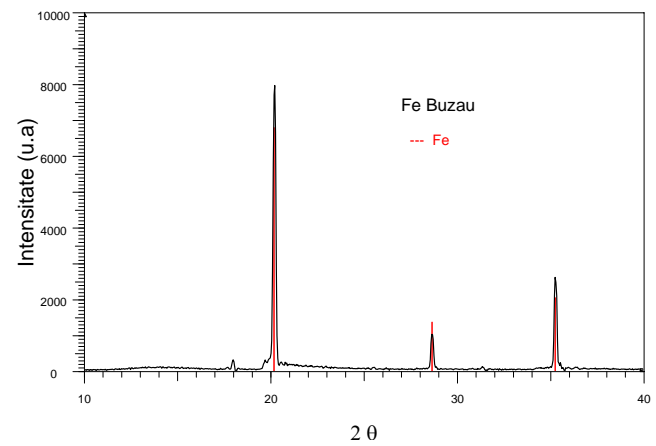


Fig.3. The diffractogram by powders ANCORSTEEL 1000B

The mechanical alloying was realized in a Pulverizette 6 planetary mill type using the next parameters:

- speed 550 rot/min,
- alloy report 3/10,
- milling medium: argon,
- milling time 60 hours.
- filling the milling box: 100 g powder and 50 balls of stainless steel ( $\phi$ 10mm);

During mechanical alloying were prevailed samples for investigations from 20 to 20 hours.

One of the important parameters of the grinding is grinding temperature inside the enclosure. Due to collisions between balls, rolling and grinding chamber walls have a series of reactions and shock that increases the temperature inside the enclosure.

Thus, the choice of time and breaks the grinding, the temperature and pressure inside the bowl were measured using a FRITSCH brand GTM system, fig.4.



Fig.4. The evolution of temperature and pressure inside the bowl for 3 cycles of 5 minutes of alloying and 5 minutes break between them.

Nanopowders morphology and distribution of composite granules of powder Fe + Fe<sub>3</sub>C was determined by SEM microscopy.

To determine the grain shape it has been used the SEM at JEOL 5600 LV microscope, equipped with analyzer EDX RF 600 with wavelength  $\pm 0,000014$  mm

– Oxford Instruments Equipped with a system of analysis INCA 200.

Figs. 5-7 presents the representative micrographs of the Fe+Fe<sub>3</sub>C powder mixes, corresponding to different mechanical alloying times.

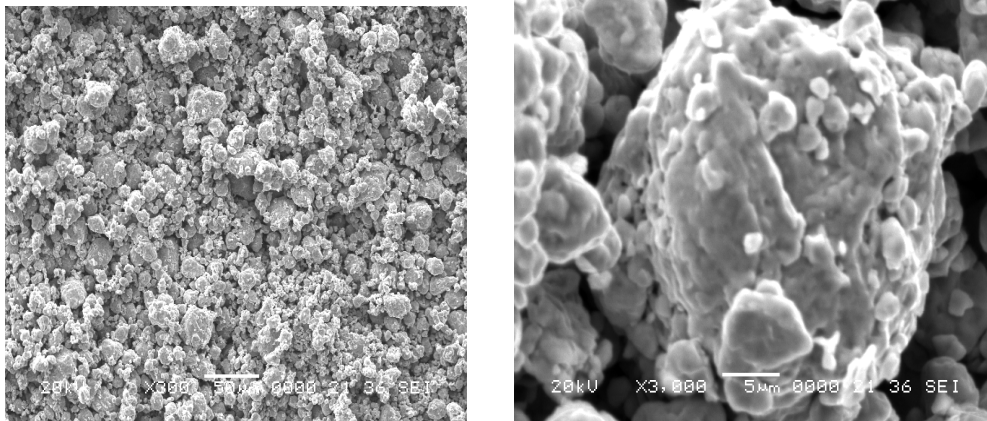


Fig.5 Microstructure SEM at Fe<sub>3</sub>C + μFe after 20h of milling

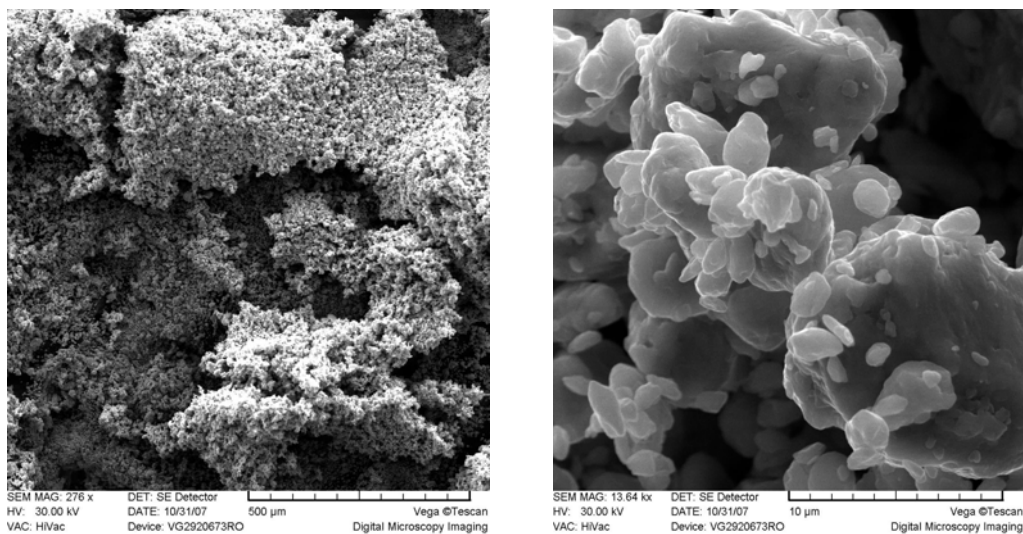


Fig. 6 Microstructure SEM at Fe<sub>3</sub>C + μFe after 40h of milling

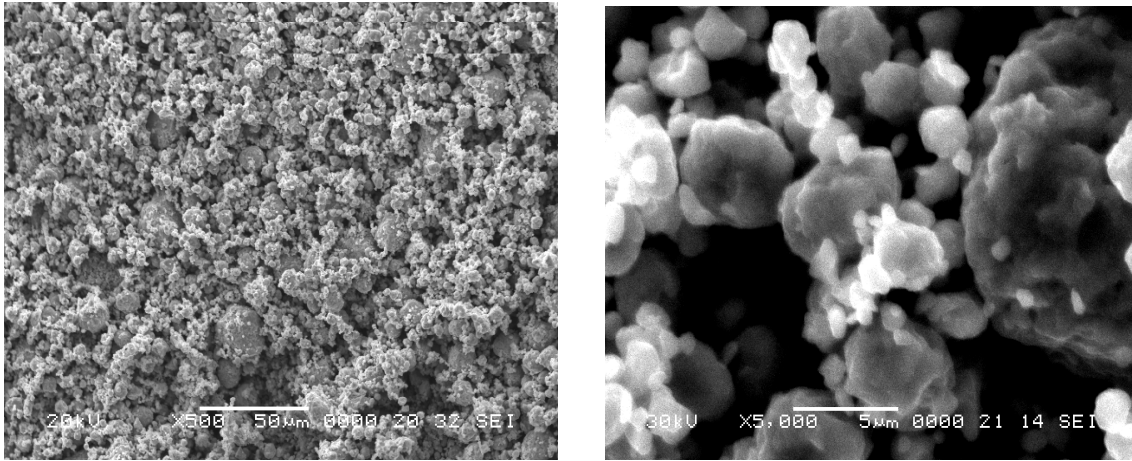


Fig. 7. Microstructure SEM at  $Fe_3C + \mu Fe$  after 60h of milling.

Looking the elements distribution in mechanical alloyed powder after 60 hours, that is showed in figure 8, and from point of view of the quantitative the determinations made it with the same program are showed in Fig. 9 and Table 4.

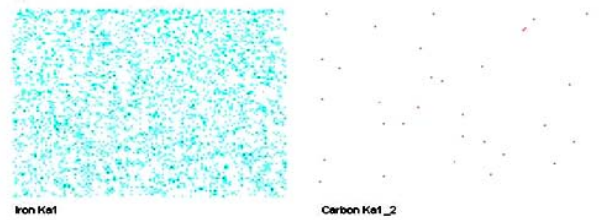


Fig. 8 The elements distribution(Fe and C) after 60 hours

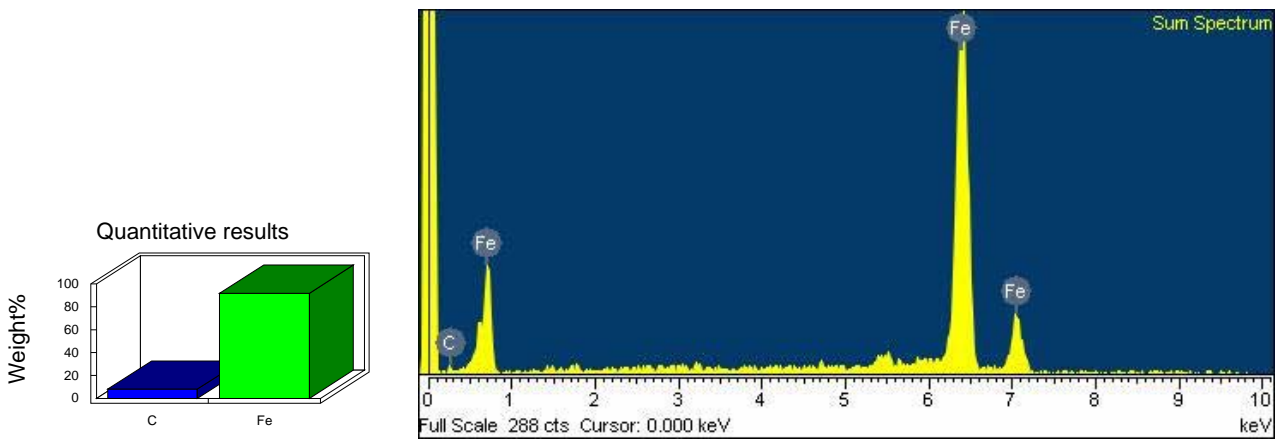


Fig. 9. The elements distribution (Fe and C) from quantitative point of view in mechanical alloyed powder after 60 hours

Table 4 The elements distribution.

Element	App Conc.	Intensity Corn.	Weight%	Weight% Sigma	Atomic%
C K	4.34	0.4193	8.06	2.73	28.94
Fe K	115.61	0.9780	91.94	2.73	71.06
Totals			100.00		

Powder compaction was done by unilateral pressing into a mould of  $\Phi 10$  mm, at pressing force of 500, 600 and 700 MPa, on LBG electromechanical-computerized 100kN

testing machine, equipped with TCSOft2004Plus software . The schematic example of unilateral die pressing with single action is presented in figure 10.

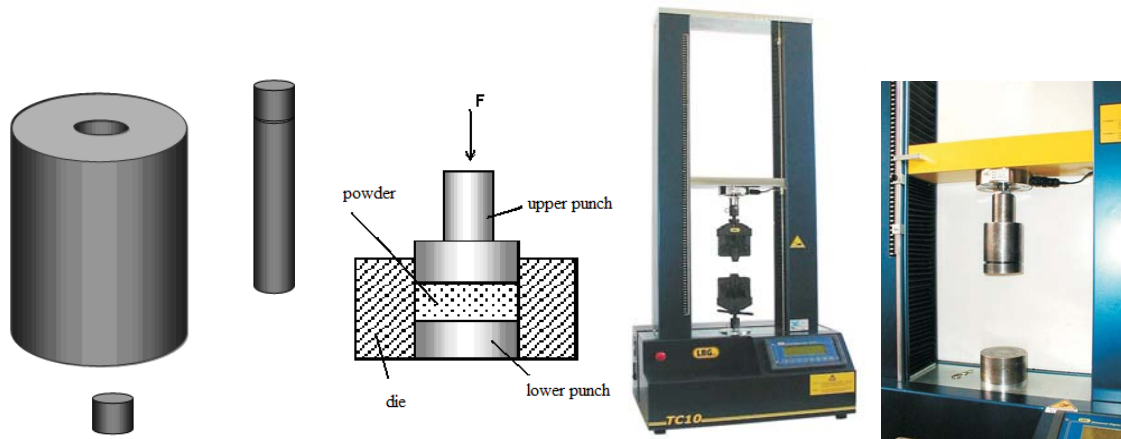


Fig. 10 The cylindrical die used for pressing and testing machine.

The sintering of the samples was done in an electric furnace using a box for sintering in argon atmosphere, with a constant heating rate to ensure a uniform distribution of thermal flux into the entire mass of the part, reducing thus the possibility to appear the cracks or internal defects.

After sintering, the box was cooled in furnace into the protective argon atmosphere.

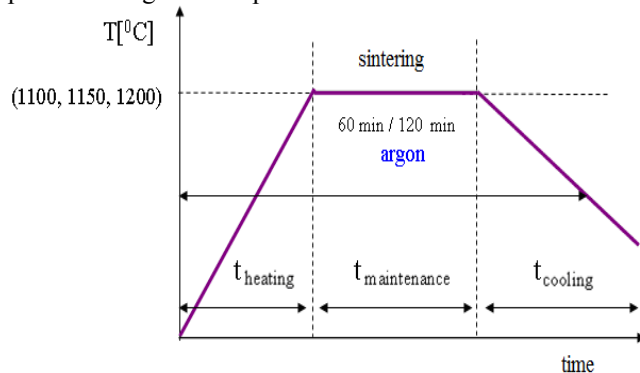


Fig. 11. The thermal chemical treatment cyclogram

### 3. Results and discussion

The research determined HB hardness according SR EN 24498-1, "Sintered metal materials, excluding hardmetals", by applying an impact energy of 11 Nmm.

Brinell attempt was made with a number of 3 prints on the same sample.

In table 5 are shown the values of hardness for steels powders [6,75Fe<sub>3</sub>C + μFe].

The evolution of hardness depending on compaction pressure or sintering temperature.

Table 5 The evolution of the hardness depending on the compaction pressure and the sintering temperatures.

T. [°C]	Materials	Hardness HB		
		Compacted pressure of 500 MPa	Compacted pressure of 600 MPa	Compacted pressure of 700 MPa
1100	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 20h	225	236	294
	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 40h	265	289	312
	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 60h	269	293	319
1150	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 20h	241	256	297
	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 40h	297	309	328
	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 60h	303	314	332
1200	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 20h	302	326	334
	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 40h	346	369	381
	[6,75Fe <sub>3</sub> C + μFe] mechanical alloying 60h	351	372	387

The thermal chemical treatment cyclogram is showed in Fig. 12; the heating to carburizing made it at 930<sup>0</sup> C, the variable being the maintenance time: 90,120,180 min.

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

The thermal chemical treatment is applied to carbon steel sintered parts based on  $[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ .

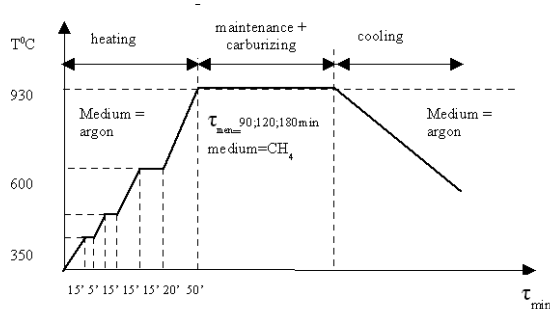


Fig.12. Carburization - thermal chemical treatment cyclogram .

Table 6. The evolution of the hardness depending on the compaction pressure and the sintering temperatures (thermal chemical treatment)

T. [°C]	Materials	Hardness HB		
		Compacted pressure of 500 MPa	Compacted pressure of 600 MPa	Compacted pressure of 700 MPa
1100	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 20h	234	245	294
	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 40h	275	297	321
	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 60h	281	299	328
1150	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 20h	249	265	304
	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 40h	305	319	336
	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 60h	323	334	341
1200	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 20h	312	333	343
	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 40h	353	378	388
	$[6,75\text{Fe}_3\text{C} + \mu\text{Fe}]$ mechanical alloying 60h	365	382	398

#### 4. Conclusions

Hardness increases with increasing compaction pressure and sintering temperature.

It can be concluded that  $\text{Fe}_3\text{C}$  help increase hardness steel sintered, thereby achieving the highest value of hardness at sintering temperature of 1200°C and compaction pressure of 700 MPa, respectively HB 387.

After thermochemical treatment applied carbon steel sintered parts can notice a slight increase in hardness.

Specific transformation during the process of mechanical alloying of Fe with  $\text{Fe}_3\text{C}$  is completed during 40-60 hours of milling, obtaining powders of nano structure.

For the future come into focus the research development in the direction of elaboration of the sintered steels from Fe+ $\text{Fe}_3\text{C}$  composites nanopowders and the study of the influence of the morphology and the different carbon concentrations on the structure and chemical composition of the steels and of the statically and dynamically characteristics of them.

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\*Corresponding author: maryana\_ciobanu@yahoo.com