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Microstructure of massive iron-carbon alloys obtained by mechanical alloying and sintering

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ABSTRACT

Purpose: The ultimate aim of this work was to investigate structure and properties massive Fe-6.67%mass.C and Fe-0.4%mass.C materials obtained by mechanical alloying and sintering.

Design/methodology/approach: The powders of the iron-carbon alloys obtained by mechanical alloying method and after that the powders were sintering. The sintering process was conducted by using the impulse-plasma method. In this article the usability of mechanical alloying method and sintering to produce the massive Fe-C materials were presented. The morphology of voids of iron-carbon sinters was analyzed using the scanning electron microscopy method. The distribution of powder particles was determined by a laser particle analyzer. The observation of the shape and size of the grains was carried out by means of the LEICA optical microscope. Then one performed the measurements of the hardness with the Vickers method. The density of the sinters was measured using the Multivolume Pycnometer 1305.

Findings: The laboratory tests show that, by using the mechanical alloying method, one can produce powder of Fe-6.67%mass.C and Fe-0.4%mass.C alloys with intentional chemical constitution and desirable structure. The structure of the alloyed materials is homogeneous and fine-grained and inside the materials didn't find some impurities and undesirable phases. The sintering by using the impulse-plasma method makes the sinters with close to theoretical density with non-variable nanocrystaline structure possible. The hardness of the sinters were 1300 HV and 250 HV adequately.

Research limitations/implications: Property of Fe-C alloys correction is possible by refinement of grains and modification of phases composition. Nanocrystaline size of grain is advisable to make it in correct technology of producing bulk materials with nanocrystaline structure. All of the presented experiments in this article are made on a laboratory scale. At the present time, most often, the mechanical alloying and the sintering processes of nanocrystaline materials are only just in the laboratory scientific research. In the nearest future the producing of amorphous and nanocrystaline materials will take place not only in the laboratory scale and move to the industry.

Originality/value: The powders produced by using mechanical alloying techniques can be use to produce bulk materials with desirable mechanical, physical and chemical properties.

Keywords: Nanomaterials; Bulk materials; Mechanical alloying; Sintering; Iron-carbon alloy; Powder materials

MATERIALS MANUFACTURING AND PROCESSING

1. Introduction

The amorphous and nanocrystaline materials are the most widely studied group of materials [21]. Nanomaterials synthesized by mechanical alloying (MA) are in the thermodynamic not equilibrium state. During the MA the change of the chemical composition and material microstructure occurs [2, 8].

Iron-carbon alloys are of great importance in technology of steel and in steel-industry. Hot or cold work together with subsequent treatments perform to control the shape and properties of steels. So essential importance of iron alloys results from the possibility of regulation of their physical and mechanical properties, which in turn, are affected fundamentally by the correct choice of chemical composition of the alloys as well as by their purity [10, 11, 12, 14, 16, 17].

High carbon alloys were studied on a limited scale only. It follows from the sublimation of carbon and from difficulties in preparation of carbides (Fe_3C, Fe_5C_2, Fe_2C, Fe_7C_3) by conventional methods. Mechanical alloying is a method making it possible to obtain high carbon alloys with very fine microstructure content-related approach to the issue worked out and described by authors [1, 3, 6].

High-energy mechanical milling has become a major potential process for processing advanced materials which awaits to be used in industry [13, 18, 19, 20].

The main aim of this work was to determine structure and properties powder and bulk Fe-C materials obtained by mechanical alloying and sintering.

2. Experimental procedure

The investigations on Fe-6.67%mass.C and Fe-0.4%mass.C alloys were conducted. The study material was the mixture of pure iron and graphite powders in suitable weight relation. The mechanical alloying process was conducted in a high-energy SPEX 8000 mill of the shaker type under inert argon atmosphere. The iron and graphite powders were mixed for the 25 and 150 hrs. Ball to powder ratio was equal 2:1. The sintering process was conducted by using the Pulse Plasma Sintering method [4, 5, 7, 9, 15]. Parameters of the sintering of powders are presented in table 1. The final dimensions of sinters measured 8 mm in diameter and 3 mm in thickness.

The investigations were carried out by means of the Philips PW 1140 X-ray diffractometer with digital registration. The microscopic observations of the shape and size of the powdered material particles were carried out by means of the OPTON DS 540 scanning electron microscope. The microscopic observations of the quantity of voids were carried out by means of the ZEISS scanning electron microscope. The observation of the shape and size of the grains was carried out by means of the LEICA optical microscope. Then one performed the measurements of the hardness with the Vickers method. The density of the sinters was measured using the Multivolume Pycnometer 1305.

The distribution of size of particles of powder materials after different time of mechanical alloying was carried out. The measurements were carried out by means of the laser analyzer "Analysette 22". The laser analyzer "Analysette 22" made by Fritsch company is the apparatus designed to defining the distribution of size of solid particles, in the range sizes 0.1-1250μm. The device consists of helium-neon laser, optical system, measuring container and steer module. Diameters of powder particles are estimated by computer unit on the ground of geometric parameters.

3. Results

The X-ray diffraction investigations enabled the identification of ferrite and cementite phases (Fig.1 and Fig.2).

The phase X-ray analysis proved the changes occurring in the mechanical alloying process. The diffraction pattern recorded for the powder of Fe-6.67%mass.C ground for 25h and 150h shows the peaks characteristic for α-Fe and α-Fe, Fe_3C (adequately). When the grinding time increases, α-Fe peaks become wider and their intensity decreases.

The diffraction records of powders of Fe-0.4%mass.C alloy vs. time of grinding are shown in Fig. 5 and Fig. 6. The diffraction pattern of Fe-0.4%mass.C alloy recorded for the powder after 25 and 150 hrs. shows the peaks characteristic for α-Fe only.

The sinters containing the Fe_3C and α-Fe were obtained from the mixture with mechanical alloyed iron and graphite. The diffraction pattern recorded for the sinters of Fe-6.67%mass.C alloy shows the peaks characteristic for Fe_3C and α-Fe (Fig. 3. and Fig. 4.). During the mechanical alloying there are change of phase composition in 25 hrs. milled alloy. The diffraction pattern of Fe-0.4%mass.C alloy recorded for the sinters shows the peaks characteristic for α-Fe and Fe_3C (Fig. 7 and Fig. 8). The sintering process cause the further crystallization of cementite.

Statistical means and diameter sizes of examined powder are presented in Table 2-5. Moreover, the distribution of powder particle size is shown in Figure 9-12.

Table 1.
Parameters of the sintering of powders

Parameter	Stage I	Stage II	Sample
Temperature	100ºC	900ºC	Fe-6.67%mass.C-25h
	100ºC	900ºC	Fe-6.67%mass.C-150h
Time	180s	90s	Fe-0.4%mass.C-25h
Pressure	60MPa	60MPa	Fe-0.4%mass.C-150h
Chamber pressure	$5 \cdot 10^{-2}$ Pa	$5 \cdot 10^{-2}$ Pa	

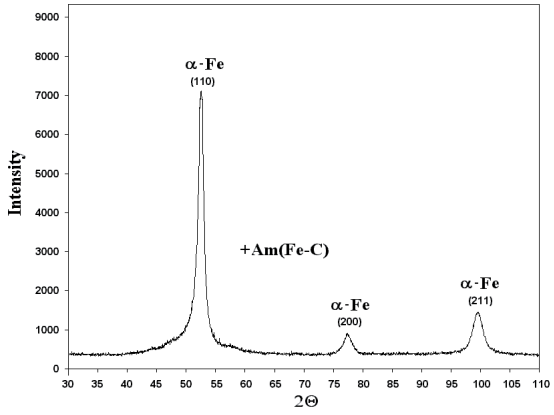

Fig. 1. The X-ray diffraction patterns of Fe-6.67%mass.C powder alloy vs. the grinding time 25 hours

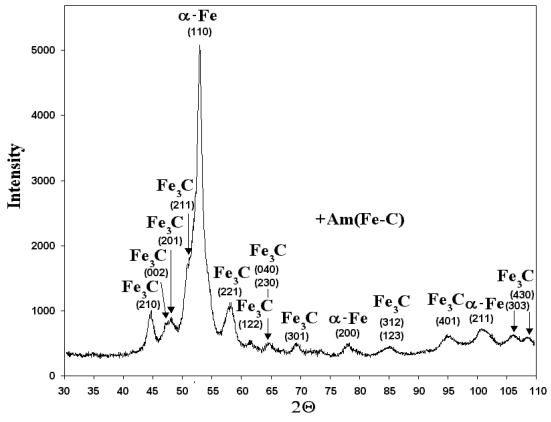

Fig. 2. The X-ray diffraction patterns of Fe-6.67%mass.C powder alloy vs. the grinding time 150 hours

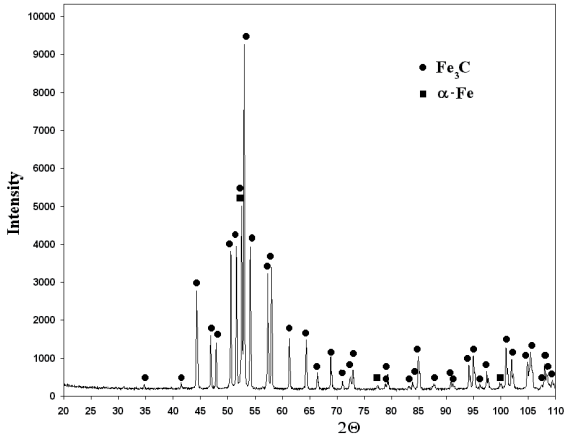

Fig. 3. The X-ray diffraction patterns of Fe-6.67%mass.C powder after mechanical alloying (25 h) and pulse-plasma sintering

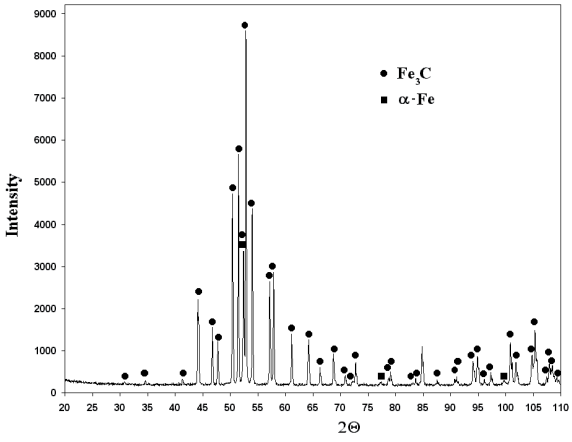

Fig. 4. The X-ray diffraction patterns of Fe-6.67%mass.C powder after mechanical alloying (150 h) and pulse-plasma sintering

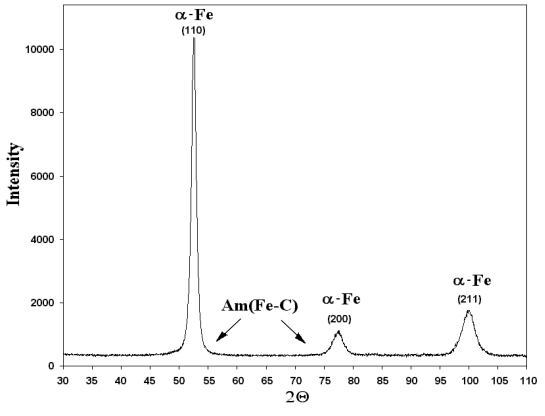

Fig. 5. The X-ray diffraction patterns of Fe-0.4%mass.C powder alloy vs. the grinding time 25 hours

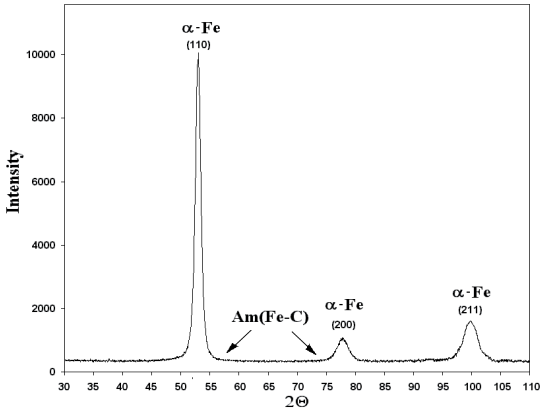

Fig. 6. The X-ray diffraction patterns of Fe-0.4%mass.C powder alloy vs. the grinding time 150 hours

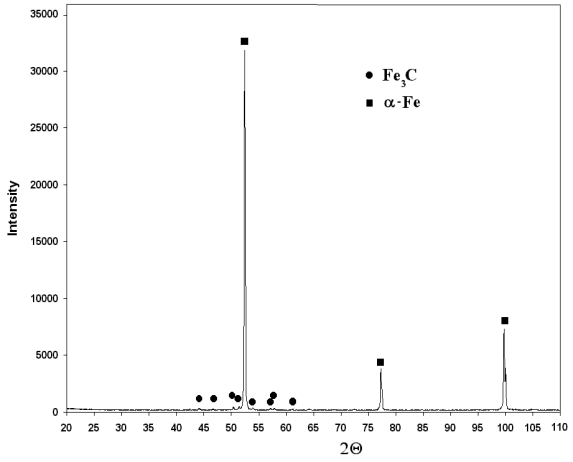

Fig. 7. The X-ray diffraction patterns of Fe-0.4%mass.C powder after mechanical alloying (25 h) and pulse-plasma sintering

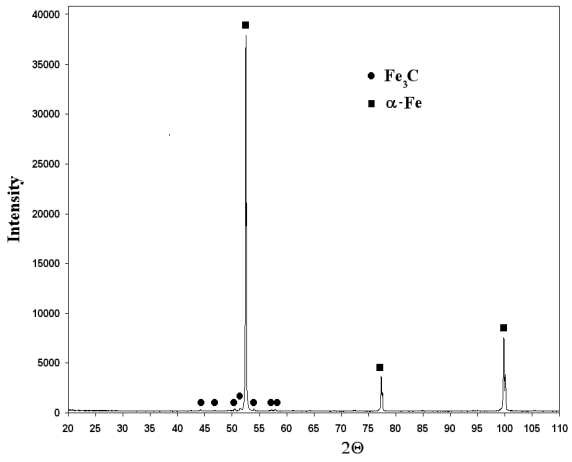

Fig. 8. The X-ray diffraction patterns of Fe-0.4%mass.C powder after mechanical alloying (150 h) and pulse-plasma sintering

The optical microstructure of the Fe-6.67%mass.C sinter produced of powders after 25h mechanical alloying and sintering one is shown in Fig. 13. The grains are fine-grained and homogeneous in this alloy. The structure of the Fe-6.67%mass.C sinter after 150h mechanical alloying and sintering one is shown in Fig. 14. Fig. 14 presents the equiaxial and round grains frequently. The optical microstructure of Fe-0.4%mass.C sinter produced of powders after 25 and 150 hrs are presented in Fig. 15 and Fig.16. The grains of the Fe-6.67%mass.C sinter after 150h mechanical alloying and sintering one are greater than grains of the Fe-6.67%mass.C sinter after 25h mechanical alloying and sintering one. Determination of an effect the conditions of the sintering process on massive materials' property demands of subsequent research.

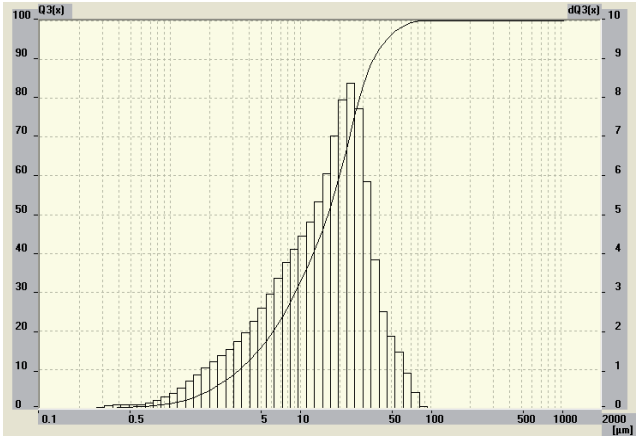

Fig. 9. Distribution and summary curve of powder particle size of studied Fe-6.67% mass. C powder after 25 hrs. of MA

Fig. 17-20 present the microstructure of the sinters. The structure of the sintered materials is characterized with the quantity of voids.

There appear small concentrations of large, approx. 0.5 - 1µm voids. There are small (0.1µm), single voids too. The sinter (Fig. 15, Fig. 16) is characterized with considerably smaller fraction of voids. From the test carried out on the scanning electron microscopy it results that material appear small voids and characteristic cracks beside large voids.

The average hardness of the sinters Fe-6.67%mass.C was 1300 HV and density 96% of the theoretical value. The average hardness of the sinters Fe-0.4%mass.C was 250 HV and density about 99% of the theoretical value (Table 6 and Table 7).

Table 2.
Main statistical parameters and diameter sizes of studied Fe-6.67% mass. C powder particles after 25 hrs. of MA

No.	Powder size	Value [µm]
1.	Arithmetic mean diameter	18.5
2.	Geometric mean diameter	13.2
3.	Standard deviation	4.3
4.	Mean square deviation	10.7
5.	Mode	23.9
6.	Median	16.2

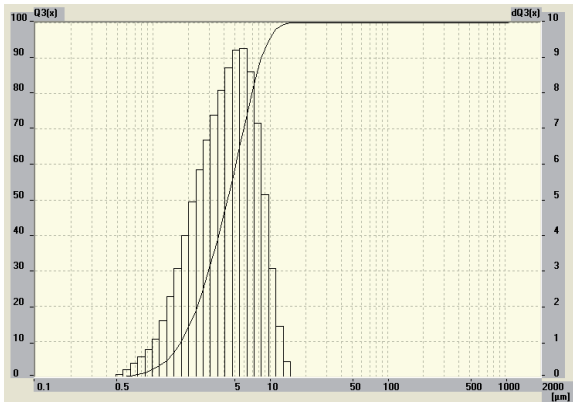

Fig. 10. Distribution and summary curve of powder particle size of studied Fe-6.67% mass.C powder particles after 150 hrs. of MA

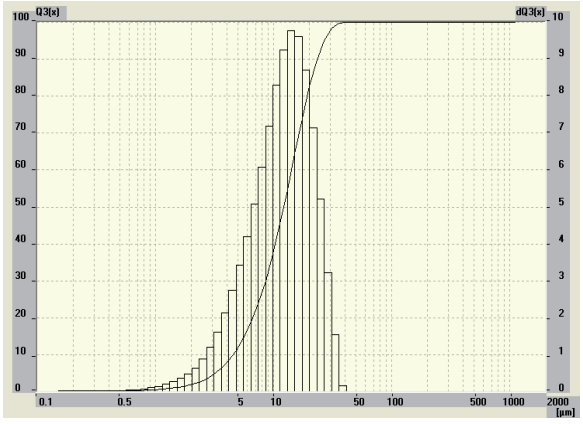

Fig. 11. Distribution and summary curve of powder particle size of studied Fe-0.4% mass. C powder after 25 hrs. of MA

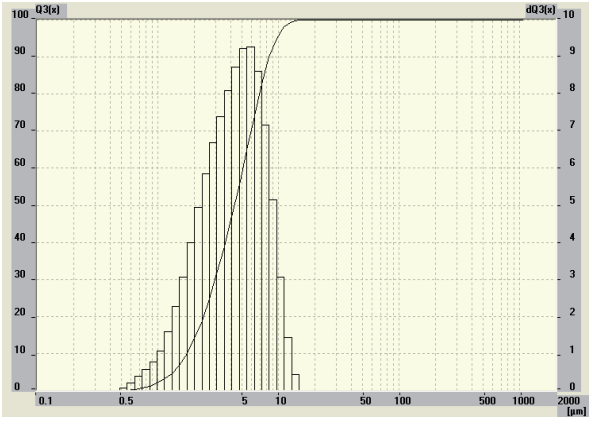

Fig. 12. Distribution and summary curve of powder particle size of studied Fe-0.4% mass. C powder after 150 hrs. of MA

Table 3.
Main statistical parameters and diameter sizes of studied Fe-6.67% mass. C powder particles after 150 hrs. of MA

No.	Powder size	Value [µm]
1.	Arithmetic mean diameter	4.7
2.	Geometric mean diameter	4.0
3.	Standard deviation	2.2
4.	Mean square deviation	2.1
5.	Mode	5.7
6.	Median	4.3

Table 4.
Main statistical parameters and diameter sizes of studied Fe-0.4% mass. C powder particles after 25 hrs. of MA

No.	Powder size	Value [µm]
1.	Arithmetic mean diameter	13.2
2.	Geometric mean diameter	10.9
3.	Standard deviation	3.6
4.	Mean square deviation	5.9
5.	Mode	14.1
6.	Median	12.2

Table 5.
Main statistical parameters and diameter sizes of studied Fe-0.4% mass. C powder particles after 150 hrs. of MA

No.	Powder size	Value [µm]
1.	Arithmetic mean diameter	5.6
2.	Geometric mean diameter	4.9
3.	Standard deviation	2.4
4.	Mean square deviation	2.0
5.	Mode	5.9
6.	Median	5.4

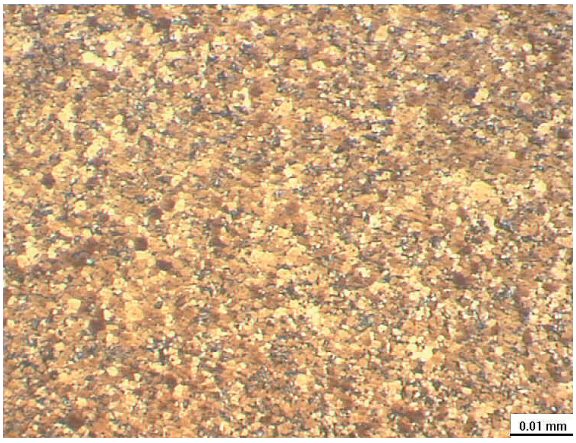

Fig. 13. Image of the microstructure of the sintered Fe-6.67%mass.C powder obtained by 25h of MA and sintering

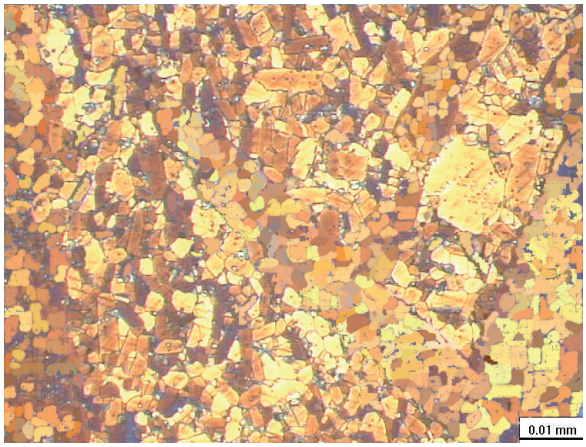

Fig. 14. Image of the microstructure of the sintered Fe-6.67%mass.C powder obtained by 150h of MA and sintering

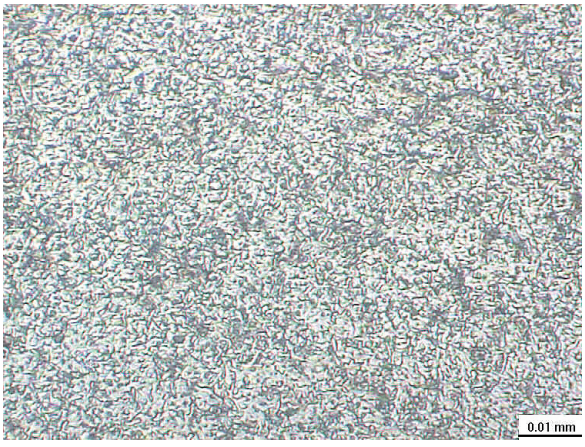

Fig. 15. Image of the microstructure of the sintered Fe-0.4%mass.C powder obtained by 25h of MA and sintering

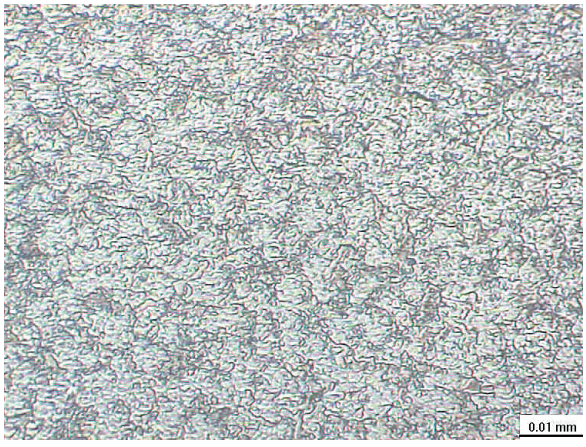

Fig. 16. Image of the microstructure of the sintered Fe-0.4%mass.C powder obtained by 150h of MA and sintering

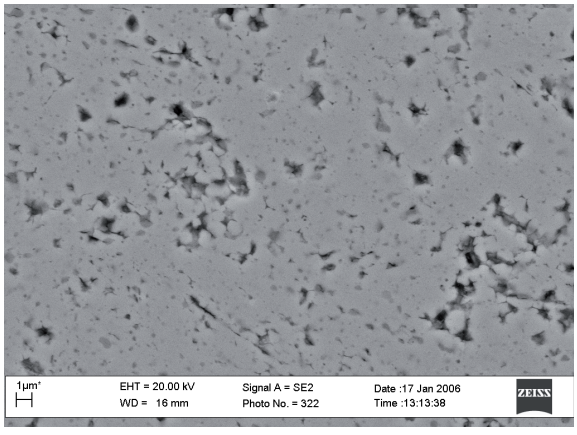

Fig. 17. Image of the voids of the sintered Fe-6.67%mass.C powder obtained by 25h of MA and sintering

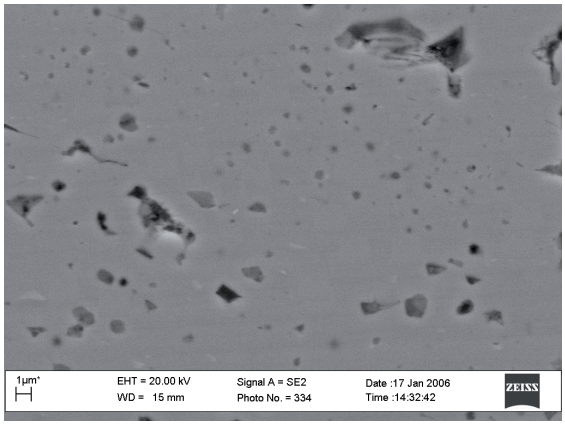

Fig. 18. Image of the voids of the sintered Fe-6.67%mass.C powder obtained by 150h of MA and sintering

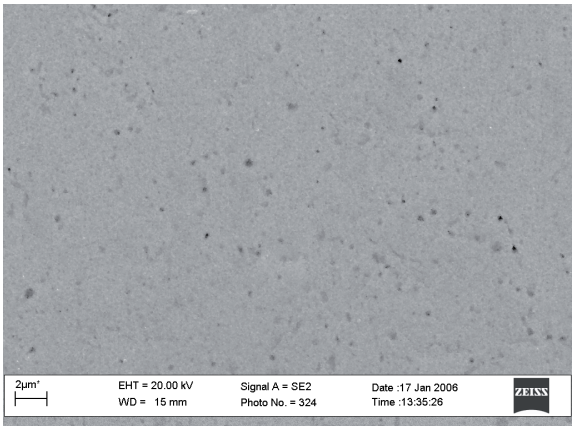

Fig. 19. Image of the voids of the sintered Fe-0.4%mass.C powder obtained by 25h of MA and sintering

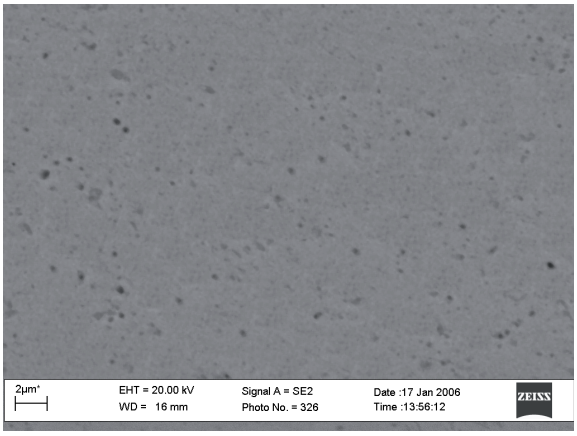

Fig. 20. Image of the voids of the sintered Fe-0.4%mass.C powder obtained by 25h of MA and sintering

Table 6.
Hardness of the sinters Fe-6.67% mass. C and Fe-0.4% mass. C

Samples	Hardness (μHV)
Fe-6.67% mass. C, 25h	1300
Fe-6.67% mass. C, 150h	1300
Fe-0.4% mass. C, 25h	250
Fe-0.4% mass. C, 150h	250

Table 7.
Real and theoretical density of the sinters Fe-6.67% mass. C and Fe-0.4% mass. C

Samples	Fe-α [vol. fraction %]	Fe_3C [vol. fraction %]	theoretical density [g/cm^3]	real density [g/cm^3]
Fe-6.67% mass. C, 25h	0.34 ± 0.29	99.66 ± 0.29	7.670714	7.44 ± 0.02 (96.99% theoretical density)
Fe-6.67% mas. C, 150h	0.15 ± 0.31	99.85± 0.31	7.670315	7.42 ± 0.02 (96.74% theoretical density)
Fe-0.4% mass. C, 25h	97.2 ± 1.31	2.8 ± 1.31	7.874120	7.80 ± 0.02 (99.05% theoretical density)
Fe-0.4% mass. C, 150h	90.0 ± 1.35	10 ± 1.35	7.859	7.76 ± 0.02 (98.74% theoretical density)

4. Conclusions

1. The mechanical alloying method and the impulse-plasma sintering method permit to obtain massive Fe-6.67% mass. C and Fe-0.4% mass. C materials. The structure of the sinters is fine-grained and homogeneous.
2. The sintering process in the temperature of 900 degrees causes the further crystallization of cementite. In sintered materials Fe_3C and α-Fe phases are present.
3. The average hardness of the sinters was 1300 HV and 250 HV for the Fe-6.67%mass.C and Fe-0.4%mass.C alloys adequately.
4. The density was 96% and 99% of the theoretical value of the sinters Fe-6.67%mass.C and Fe-0.4%mass.C alloys adequately.

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Additional information

The presentation connected with the subject matter of the paper was presented by the authors during the 14th International Scientific Conference on Achievements in Mechanical and Materials Engineering AMME'2006 in Gliwice-Wisła, Poland on 4th-8th June 2006.

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