Journal of Achievements in Materials and Manufacturing Engineering

VOLUME 18
ISSUE 1-2
September–October
2006

The Fe-C alloy obtained by mechanical alloying and sintering

R. Nowosielski, W. Pilarczyk *
Division of Nanocrystalline and Functional Materials and Sustainable Pro-ecological Technologies, Institute of Engineering Materials and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland
* Corresponding author: E-mail address: wirginia.pilarczyk@polsl.pl

Received 15.03.2006; accepted in revised form 30.04.2006

Materials

ABSTRACT

Purpose: The main aim of this work was to determine structure and properties massive Fe-C materials obtained by mechanical alloying and sintering.
Design/methodology/approach: The results of experiments on the fabrication of powders materials and solid materials using pure iron and graphite powders are presented. The powders of the Fe-C 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 materials were presented.
Findings: The laboratory tests show that, by using the mechanical alloying method, one can produce powder of Fe-6.67% mass.C alloy with intentional chemical constitution and desirable structure. The structure of the 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 microstructure possible. The hardness of the sinters was 1300 HV.
Research limitations/implications: The mechanical alloying method is one of the techniques which enables to improve property of Fe-C alloys. It is possible by refinement of structure and modification of phases composition. Nanocrystaline size of grain is advisable to make it in correct technology of producing massive materials with nanocrystaline structure. All of the presented experiments in this article are conducted on a laboratory scale. At the present time, all over the world, 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 nanomaterials will take place not only in the laboratory and move to the industry.
Originality/value: The nanomaterials have an unusual mechanical, physical and chemical properties.
Keywords: Nanomaterials; Mechanical alloying; Sintering; Iron-carbon alloy

1. Introduction

Nanomaterials are the most widely studied group of materials. Synthesized by mechanical alloying, they are often non-equilibrium.

Iron-carbon alloys are of great importance in technology of steel and in industry. So essential importance of iron and its 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.

High carbon alloys were treated on a limited scale only. It follows from the sublimation of carbon and from difficulties in preparation of carbides (Fe_3C, Fe_7C_3) by conventional methods.

Mechanical alloying is a new method making it possible to obtain massive high carbon Fe-C alloys with very fine microstructure content-related approach to the issue worked out and described by authors [1, 3, 6].

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

2. Experimental procedure

The investigations on Fe-6,67%mass.C alloy were conducted. The test material was the mixture of iron and graphite powders in suitable weight relation. The mechanical alloying process was conducted in a high-energy SPEX 8000 Certi/Prep Mixer/Mill of the shaker type under inert gas (argon) atmosphere. The powders were mixed for the 25 and 150 hrs. BPR ratio was equal 2:1. The sintering process was conducted by using the PPS method (Pulse Plasma Sintering method [5, 7]) (Table 1). After consolidation, the final dimensions of samples 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 sinters was carried out by means of the LEICA optical microscope. Then one performed the measurements of the hardness with the Vickers method.

3. Results

The phase X-ray analysis proved the changes occurring in the mechanical alloying process. The diffraction pattern recorded for the powder 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 versus the time of grinding are shown in Fig. 1 and Fig. 2.

The sinters containing the Fe_3C and α-Fe were obtained from the mixture with iron and graphite. The diffraction pattern recorded for the sinters (Fig. 3. and Fig. 4.) shows the peaks characteristic for Fe_3C and α-Fe. The phase changes and the results of the structural examinations of the powders were reported earlier [1, 2].

The optical microstructure of the Fe-6,67%mass.C sinter produced of powders after 25h mechanical alloying and sintering one is presented in Fig. 5. The structure is 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. 6. Fig. 6 shows the equiaxial and round grains frequently. Fig. 7 shows the microstructure of the sintered materials. The structure of the sinter is characterized with the quantity of voids. There appear small concentrations of large, approx. 0,5 - 1μm void

The average hardness of the sinters was 1300 HV.

Table 1.
Parameters of the sintering of powders

Parameter	Stage I	Stage II	Sample
Temperature	100ºC	900ºC	Fe-6,67%mass.C-25h Fe-6,67%mass.C-150h
	100ºC	900ºC	
Time	180s	90s	
Pressure	60MPa	60MPa	
Chamber pressure	$5 \cdot 10^{-2}$ Pa	$5 \cdot 10^{-2}$ Pa	

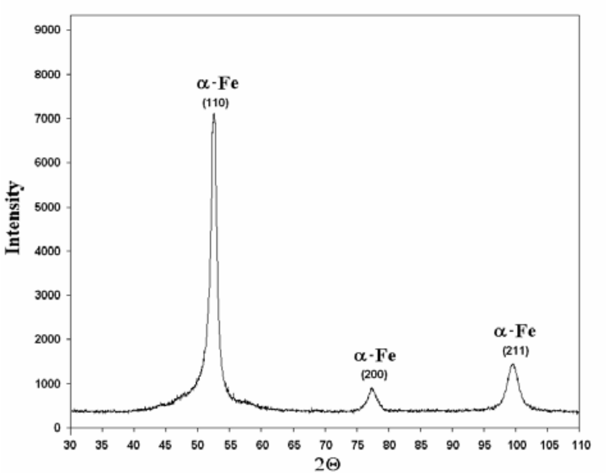

Fig. 1. The X-ray diffraction patterns of Fe-6,67% mass C powder alloy vs. the grinding time 25 hours

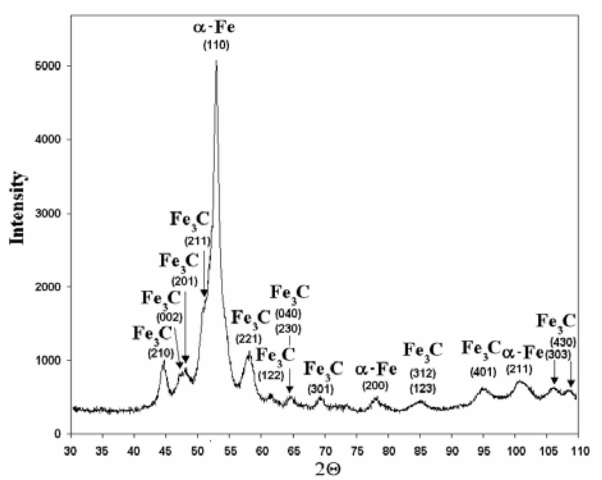

Fig. 2. The X-ray diffraction patterns of Fe-6,67% mass C powder alloy vs. the grinding time 150 hours

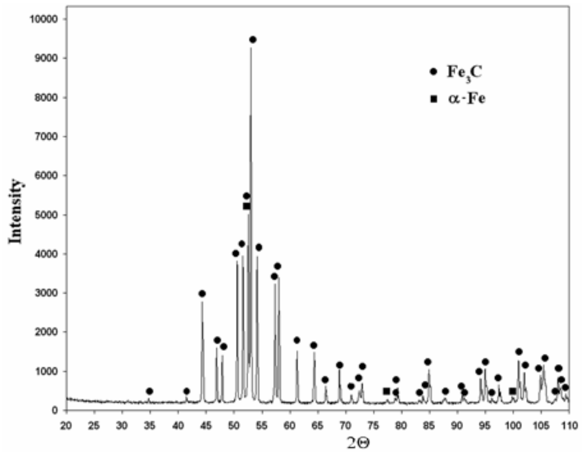

Fig. 3. The X-ray diffraction patterns of the Fe-6,67% mass C powder after mechanical alloying (25 h) and pulse-plasma sintering

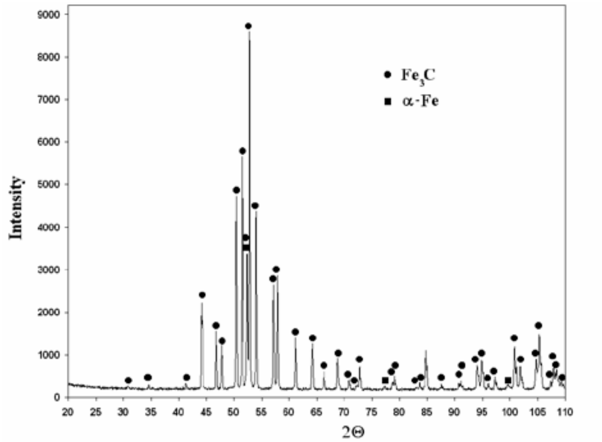

Fig. 4. The X-ray diffraction patterns of Fe-6,67% mass C powder after mechanical alloying (150 h) and pulse-plasma sintering

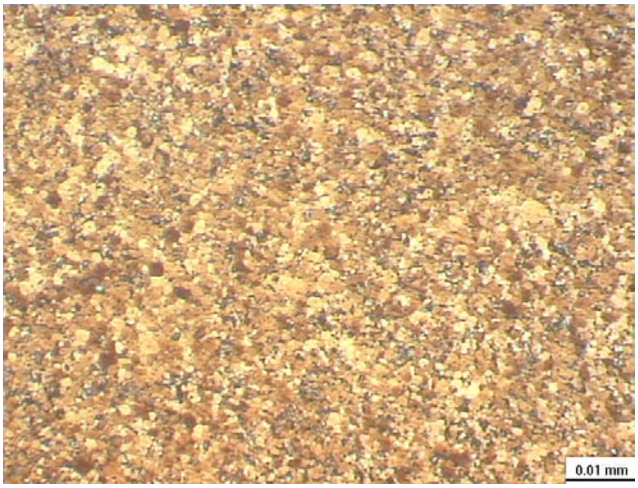

Fig. 5. Image of the microstructure of the sintered Fe-6,67% mass C powder obtained by 25h of MA and sintering (magn. 1000x)

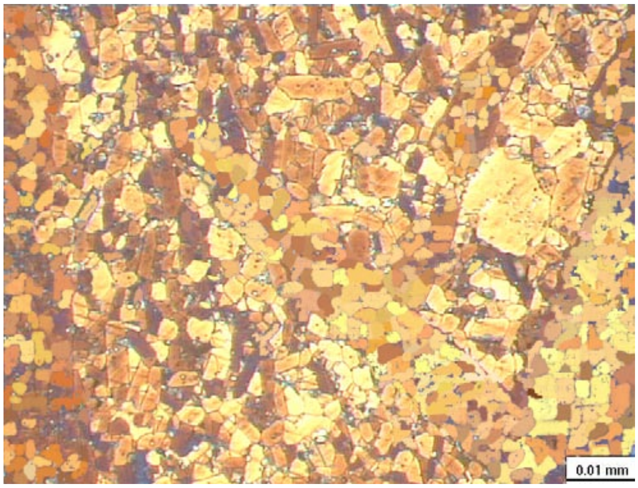

Fig. 6. Image of the microstructure of the sintered Fe-6,67% mass C powder obtained by 150h of MA and sintering (magn. 1000x)

4. Conclusions

1. The mechanical alloying method and the impulse-plasma sintering method permit to obtain massive Fe-C materials.
2. The structure of the sinters is fine-grained and homogeneous.
3. In sintered materials some phases are present, between then Fe_3C and α-Fe.
4. The sintering process in the temperature of 900 degrees causes the further crystallization of cementite.
5. The hardness of the sinters was 1300 HV.
6. Determination of an influence the conditions of the sintering process on massive materials' property requirements of further, subsequent research.

Acknowledgements

The authors would like to thank prof. A. Michalski and dr M. Rosiński (The Warsaw University of Technology, The Faculty of Materials Engineering) for the sintering process. The author are grateful to dr J. Mazurkiewicz (The Silesian University of Technology) for the tests carried out on SEM.

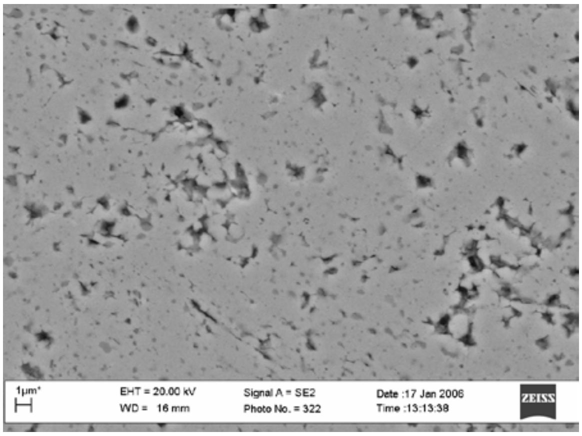

Fig. 7. Image of the voids of the sintered Fe-6,67% mass C powder obtained by 25h of MA and sintering (magn. 10000x)

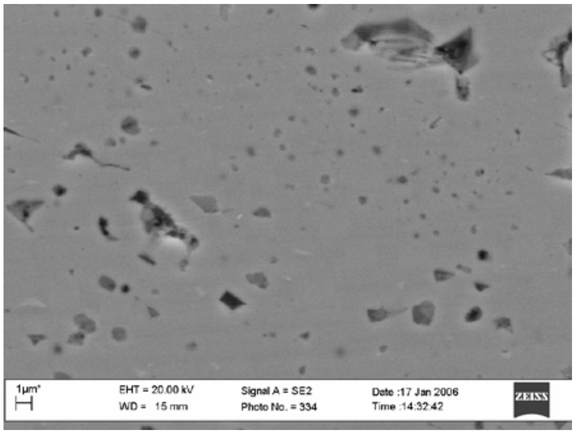

Fig. 8. Image of the voids of the sintered Fe-6,67% mass C powder obtained by 150h of MA and sintering (magn. 10000x)

References

[1] R. Nowosielski, W. Pilarczyk, Structure and properties of Fe-6,67%C alloy obtained by mechanical alloying, Journal of Materials Processing Technology 162-163 (2005) 373-378.

[2] R. Nowosielski, W. Pilarczyk, Structure and properties of metallic powders $Co_{78}B_{11}Si_{11}$ obtained by mechanical alloying, Proceedings of the 12th Scientific International Conference „Achievements in Mechanical and Materials Engineering" AMME'2003, Gliwice-Zakopane, 2003, 675-680.

[3] M. Jurczyk, Mechanical Alloying, Published by Poznan University of Technology, 2003, (in Polish).

[4] J. Nowacki, Sintered metals and composites with metallic matrix, WNT, Warsaw, 2005, (in Polish).

[5] A. Michalski, M. Rosiński, J. Jaroszewicz, D. Oleszak, Sintering of nanocrystalline powders by high current electric impulses, Archives of Materials Science 24 (2003) 547-560.

[6] T. Lou, B. Ding, X. Gu, G. Li, Z. Hu, Mechanical alloying of Fe-Nb-C materials, Materials Letters 28 (1996) 129-132.

[7] A. Michalski, J. Jaroszewicz, M. Rosiński, The synthesis of NiAl using the pulse plasma method with the participation of the SHS reaction, International Journal of Self-Propagating High-Temperature Synthesis 12 (2003) 237-246.

[8] N.T. Rochman, K. Kawamoto, H. Sueyoshi, Y. Nakamura, T. Nishida, Effect of milling temperature and additive elements on an Fe-C system alloy prepared by mechanical alloying, Journal of Materials Processing Technology 89-90 (1999) 367-372.

[9] G.M. Wang, S.J. Campbell, A. Calka, W.A. Kaczmarek, Ball-milling of Fe-C (20-75% Fe), NanoStructured Materials, 6 (1995) 389-392.

[10] H. Hidaka, T. Tsuchiyama, S. Takaki, Relation between microstructure and hardness in Fe-C alloys with ultra fine grained structure, Scripta Materiala 44 (2001) 1503-1506.

[11] L. Lü, M.O. Lai, S. Zhang, Modeling of the mechanical alloying process, Journal of Materials Processing Technology 52 (1995) 539-546.

[12] L. Lü, M.O. Lai, S. Zhang, Diffusion in mechanical alloying, Journal of Materials Processing Technology 67 (1997) 100-104.

[13] C. Suryanarayana, Mechanical alloying and milling, Progress in Materials Science 46 (2001) 1-184.

[14] P. Matteazzi, Gerard Le Caër, A. Mocellin, Synthesis of nanostructured materials by mechanical alloying, Ceramics International 23 (1997) 39-44.

[15] M. Jurczyk, Nanomaterials, Published by Poznan University of Technology, 2001, (in Polish).

[16] J.W. Kaczmar, K. Pietrzak, W. Włosiński, The production and application of metal matrix composite materials, Journal of Materials Processing Technology 106 (2000) 58-67.