

## The influence of HEBM on the structure of Fe-0,8%C alloys

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### Materials

#### ABSTRACT

**Purpose:** The microstructure characterization of iron-carbon powder and sinters is the purpose of this paper. The main aim of this work is to determine structure of Fe-0.8% mass. C alloys.

**Design/methodology/approach:** Iron-carbon powder alloys were produced employing the mixture of crystalline iron and graphite powders in suitable weight relation. The powders were mechanical alloyed and sintered. The pressing and free sintering were applied. The X-ray diffraction, scanning electron microscopy and optical microscopy investigations were performed on Fe-0.8% mass. C alloys.

**Findings:** The application of the mechanical alloying method enables to produce nanocrystalline alloys with amorphous phase. The choose of sinter method have an influence on structure of sinters. The free sintering process in the temperature of 1100 degrees causes the crystallization of cementite and forming of voids.

**Research limitations/implications:** The powder metallurgy techniques make it possible to obtain Fe-C massive materials by means of HEBM (High Energy Ball Milling), followed by compacting and sintering. Further investigations should be concentrate on the developing of powder consolidation method and refinement particles during high energy ball milling.

**Originality/value:** The determination of the relations between the powders and sinters structure is necessary for the proper procedure of material manufacture and indication of the fields of their application.

**Keywords:** Nanomaterials; Metallic alloys; Mechanical alloying; Sintering

### 1. Introduction

Among all methods that have been developed to obtain the nanomaterials are the mechanical alloying (MA) and mechanical milling (MM) [4, 12]. The notion of MM is understood as mechanical crumbing and mechanical alloying. During the mechanical grinding the crushing, breaking up, reducing size of particles and strengthening itself the material occurs [2, 8]. The MA process locally in the MM process can occurs. The scope of the MM investigation is to obtain powder material about the required size of small particles, whereas the MA process targets to the synthesis of pure elements powders. During the process, the powders become effectively broken up while the thermodynamical equilibrium is fixed, what results in the making of new, nonequilibrium alloys [9, 13].

This paper presented the following investigation of iron-carbon alloys. Fe-C alloys are the basis of constructional materials. They are of special importance in steel industry. Two-component alloy of iron and graphite is a basic, suitable model alloy [10, 11]. The areas in which new phases of Fe-C system are formed and the mechanism of the mass transmission in MA process are also unknown [3, 14, 15].

### 2. Experiment

The Fe-0.8 %mass. C powder alloys were produced by High Energy Ball Milling process. This process was conducted in a

high energy SPEX 8000 mill of the shaker type under inert argon atmosphere. The tests were made with pure, crystalline iron (99.5% in purity) and graphite (99.9% in purity) powders in suitable weight relation. Particle size were less than  $45\mu\text{m}$ . Two types of ball mill were used: 12 balls diameter 12 mm and 48 balls diameter 6 mm. The powders were ground 5 and 50 hours. Ball to powder ratio was 13:1.

The powder were cold pressed and sintered [1, 5, 6, 7]. Sintering was performed at the temperature of  $1100^\circ\text{C}$  for 1 hour at nitrogen atmosphere.

The changes of the powder and sinters phase composition were tested by means of the X-ray diffractometer of the Philips PW 1140 with digital registration. The crystallites' size was measured by the Scherrer's method basing oneself on the diffraction records.

The microscopic observations of the shape and size of the powder particles were carried out by means of the scanning electron microscope. Voids observations of sinters were made by optical microscopy and scanning electron microscopy.

### 3. Results and discussion

The X-ray analysis proved the changes occurring in the mechanical alloying and sintering process. The diffraction patterns of the Fe-0.8 %mass. C powders milled 5 and 50 hours showed the dependence of changes in the structure on the grinding time (Fig. 1.). All X-ray peaks become wider and their intensity decreases. The widening of peaks in comparison with the initial Fe-0.8 %mass. C powder is connected with the size reduction in the powder grains and formation of the amorphous phase. The diffraction patterns recorded for the powder alloyed for 5 and 50 hours shows the peak characteristic for Fe- $\alpha$ . When the time of MA increases, phase compositions no changes. The diffraction patterns recorded for the sinters (Fig. 2. and Fig. 3.) presents the peaks characteristic for Fe- $\alpha$  and Fe<sub>3</sub>C.

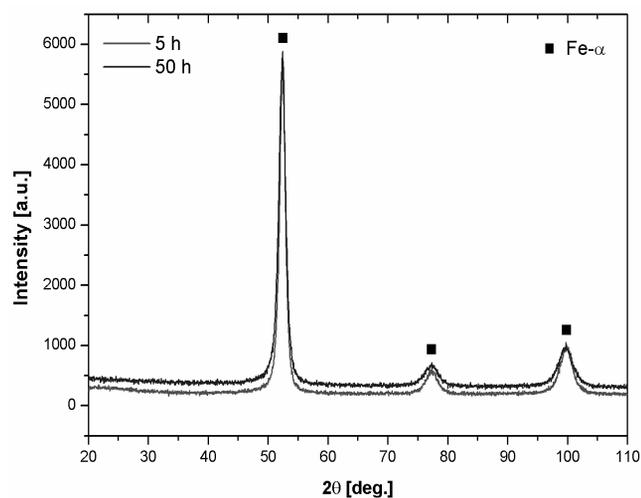


Fig. 1. X-ray diffraction patterns of Fe-0,8 %mass. C alloys after 5, 50 h of MA process

The crystallites' size of Fe-0.8 %mass. C alloy increased after the sintering. After MA, the crystallites' size was estimated as being about 20 nm and after sintering as being about 75 nm.

Observation of morphology of powders after 5 h and 50 h allows size reduction of powder particles (Fig. 4., Fig. 5.)

Microstructure observations of sinters were made by optical microscopy (Fig. 6., Fig. 7.) and scanning electron microscopy (Fig. 8., Fig. 9.).

Fig. 8. and Fig. 9 show the microstructure of the sintered materials. The structure is characterized with the large quantity of voids. It is result of applied sintering method. The investigation results have shown that it is difficult to obtain high-density sinters of low porosity. The impulse plasma sintering method [1, 6, 7] is more efficient than free sintering used in this experiment.

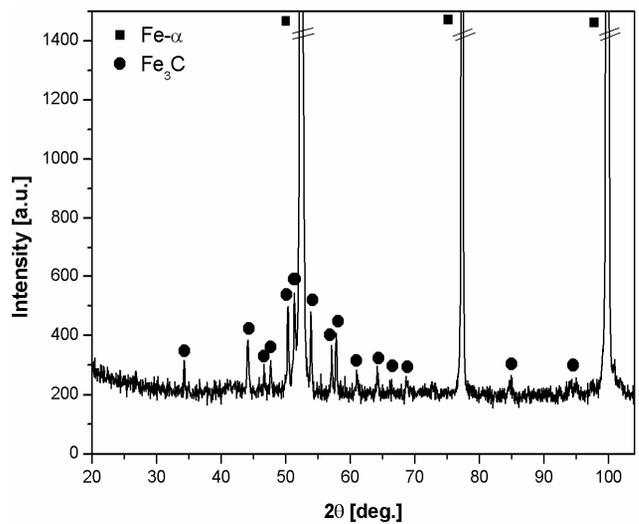


Fig. 2. X-ray diffraction patterns of Fe-0.8 %mass. C alloys after MA (5 h) and sintering

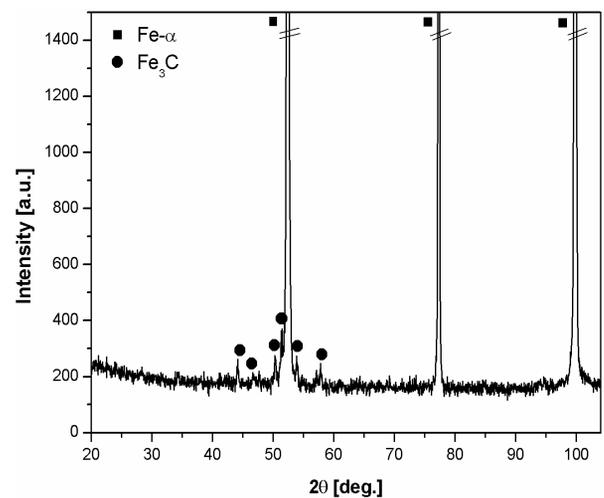


Fig. 3. X-ray diffraction patterns of Fe-0.8 %mass. C alloys after MA (50 h) and sintering

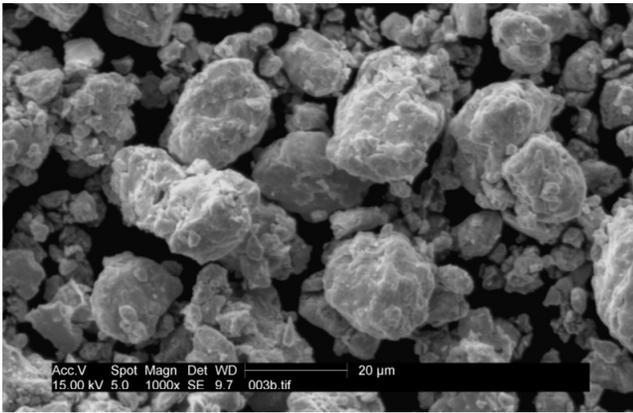


Fig. 4. SEM images of the studied Fe-0.8 %mass. C powder after 5 h of MA

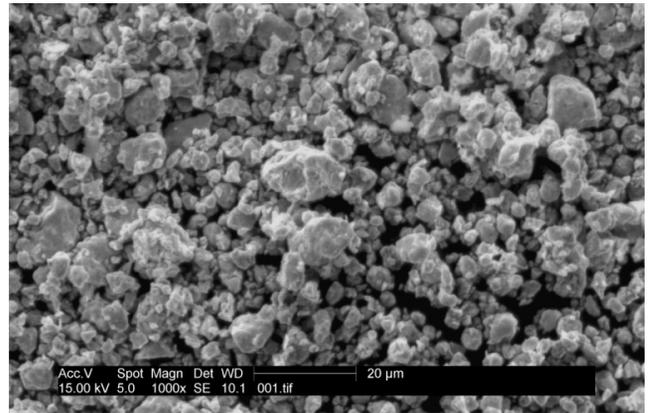


Fig. 5. SEM images of the studied Fe-0.8 %mass. C powder after 50 h of MA

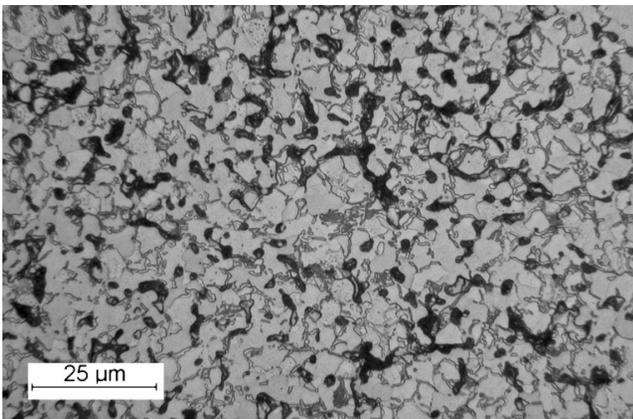


Fig. 6. Image of the microstructure of the sintered Fe-0.8 %mass. C powder obtained by 5 h of MA and sintering (magn. 400x)

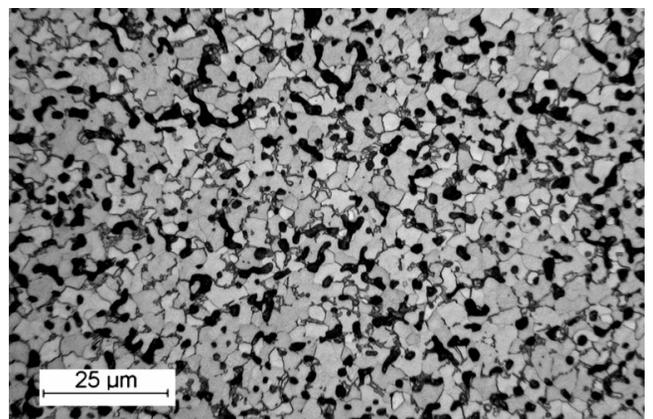


Fig. 7. Image of the microstructure of the sintered Fe-0.8 %mass. C powder obtained by 50 h of MA and sintering (magn. 400x)

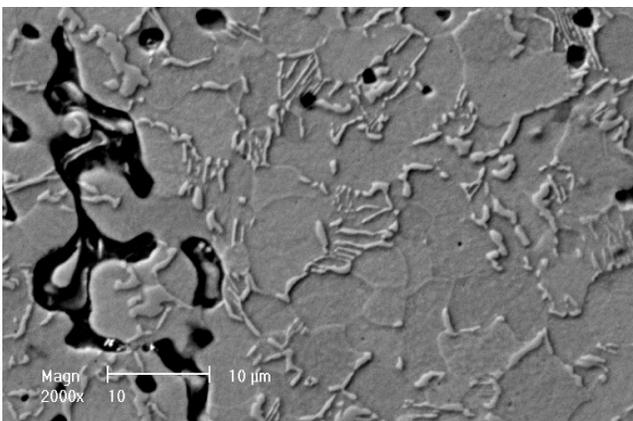


Fig. 8. Image of the microstructure of the sintered Fe-0.8 %mass. C powder obtained by 5 h of MA and sintering (SEM, magn. 2000x)

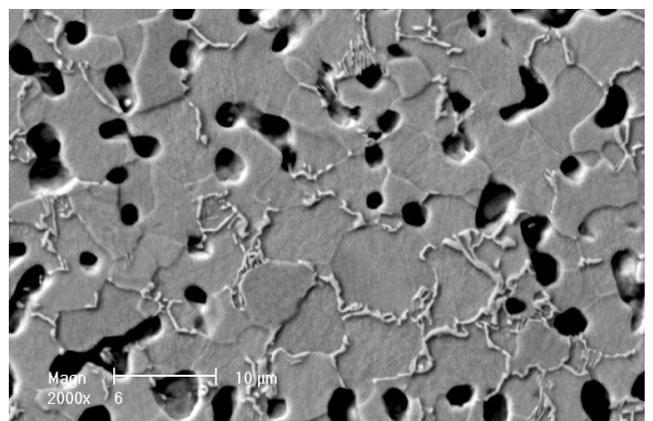


Fig. 9. Image of the microstructure of the sintered Fe-0.8 %mass. C powder obtained by 50 h of MA and sintering (SEM, magn. 2000x)

## 4. Conclusions

The main aim of this work was to determine structure of mechanical alloyed and sintered Fe-0.8 %mass. C alloys. One expected the small particles and nanocrystallites.

The investigation of the Fe-0.8 %mass. C powder alloy allowed to draw the following conclusion:

- The mechanical alloying method permit to obtain the crystallites' size of Fe- $\alpha$  phase above 100nm;
- The sintering free process in the temperature of 1100°C for 1 hour at nitrogen atmosphere causes the further crystallization of cementite. The X-ray analysis revealed the presence of Fe- $\alpha$  phase in powder alloy and Fe- $\alpha$ , Fe<sub>3</sub>C phases in sintered materials;

The studies have indicated the importance of following scientific research in the sintered powder materials. The obtained voids appeared to be much larger then expected.

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