Achievements in Mechanical and Materials Engineering

COMMENT Worldwide Congress on Materials and Manufacturing Engineering and Technology
16th - 19th May 2005 Gliwice-Wisła, Poland

COMMITTEE OF MATERIALS SCIENCE OF THE POLISH ACADEMY OF SCIENCES, KATOWICE, POLAND
INSTITUTE OF ENGINEERING MATERIALS AND BIOMATERIALS OF THE SILESIAN UNIVERSITY OF TECHNOLOGY, GLIWICE, POLAND
ASSOCIATION OF THE ALUMNI OF THE SILESIAN UNIVERSITY OF TECHNOLOGY, MATERIALS ENGINEERING CIRCLE, GLIWICE, POLAND

13th INTERNATIONAL SCIENTIFIC CONFERENCE ON ACHIEVEMENTS IN MECHANICAL AND MATERIALS ENGINEERING

Structure and properties of Fe-6,67%C alloy obtained by mechanical alloying

R. Nowosielski, W. Pilarczyk

Institute of Engineering Processing Technology and Biomaterials, Silesian University of Technology, ul. Konarskiego 18a, 44-100 Gliwice, Poland

Abstract: The test material was the mixture of Fe and C powders obtained by the mixing of the graphite powder with the iron powder in the proportion of 6,67% C : 93,33% Fe (%mass.). The powders were ground for the following time intervals: 10, 25, 50, 75 and 100 hrs. The microscopic observation of the shape and size of the powdered material grains was carried out by the scanning electron microscope provided within the magnification of 100-2000 times. The changes of the powder structure were tested by means of the X-ray diffractometer.

Keywords: Mechanical alloying, Iron-carbon alloy

1. INTRODUCTION

Thanks to the advance in development of material engineering it is possible to make use of a relatively new method, called mechanical alloying, for fabrication of modern nanomaterials based on iron and carbon, with preset chemical composition and structure. 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. Iron-carbon alloys are fabricated by conventional method, in steelmaking processes, from liquid state. Iron castings are produced in the melting processes with participation of further processes. Steel ingots, after solidification and surface preparation, are subjected to hot working within the temperature range of approx. 1200ºC to approx. 800ºC. Mechanical properties of Fe-C casting alloys are influenced by different circumstances, for instance, oxidation of steel, the presence of non-metallic inclusions and the size of graphite and ledeburyte grains which are formed during solidification while the reduction in their size is difficult to accomplishment [1]. The grain size of the steel fabricated by this means is of the order of micrometers. Since 1990 intensive investigations connected with nanocrystalline materials have been carried out. It has been observed that the deformation in the MA process yields better results in creation of nanocrystalline structure than the conventional method of cold work of materials such as pure metals or alloys [3]. Mechanical grinding (MG) – is one of the techniques which enables to generate the maximum distortion in metallic powders and the largest deformations as well as enables to modify the material structure and properties. It is expected that nanocrystalline structures generated in Fe-C alloys will be conducive to their improvement in great measure.

The increase in the interest in nanocrystalline materials is due to their unusual properties, which, in turn, arise form the grain size being of the order of nanometers (<100 nm).

As mentioned above, iron-carbon alloys which are the basis of constructional materials are of special importance. Fe-C alloy called the scientifists' attention owing to its weight in steel industry. Two-component alloy of iron and carbon is a suitable, model alloy, the components of which have different atomic radii. It is simultaneously an alloy containing carbides and carbon undissolved in α-Fe under thermodynamic equilibrium. Up to now, the interest in fabrication of Fe-C alloys was focused on receiving and investigation of alloys with low mass concentration of carbon. 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 [2]. Mechanical alloying is a new method making it possible to receive high carbon Fe-C alloys (Fe – 6,67% C in mass fraction) with very fine microstructure. Thereby it creates the possibility of manufacture of an advanced material. The grain size of Fe-C alloys fabricated by mechanical alloying is of the order of several dozen nanometers what can suggest that in these alloys also the superplasticity phenomenon occurs. In accordance with the definition, the superplasticity is the phenomenon of unusually large plastic deformation, reaching up to 2000%, observed in certain fine-grained alloys, mostly of eutectic composition. There is the opinion that the main mechanism of the superplastic flow is the slip over grain boundaries and the diffusion forced by the existence of stress fields. It is so called structural superplasticity. From the analysis of literature data it can be concluded that the process of mechanical alloying of iron powders with carbon powders as well as iron, carbon and another elements has remained unknown with full particulars. The areas in which new phases of Fe-C alloys are formed and the mechanism of the mass penetration in this process are also unknown. In the MA process the essential issue is that the structure and morphology of powders as well as their changes on each stage of the process may be defined with the use of predetermined technological procedure of receiving powders with preset phase composition. The establishment of the correct research methodics enables to determine the process-structural model of mechanical alloying. The determination of the relations between the powders structure and mechanical and operational properties of the products fabricated from them is necessary for the proper procedure of material manufacture and indication of the fields of their application [7].

2. OBJECTIVE OF THE INVESTIGATIONS

The main objective of the work was the attempt of the application of the MA process to manufacture the Fe – 6,67mass.% C alloy and the estimation of the effect of the process run time on changes in the structure and morphology of powders. The scope of the investigation comprised the execution of the mechanical alloying process and the qualification of phase changes occurring in the test alloy. The work results allow to determine the optimum time for the conducting the mechanical alloying process in order to obtain the material with the preset phase composition and structure.

3. EXPERIMENTS

The mechanical alloying process was conducted in a high-energy SPEX 8000 CertiPrep Mixer/Mill of the shaker type under inert gas (argon) atmosphere. The test material was the mixture of Fe and C powders obtained by the mixing of the graphite powder, 99,99% in

purity, with the iron powder, 99,5% in purity, in the proportion of 6,67% C : 93,33% Fe (by weight). The powders were ground for the following time intervals: 10, 25, 50, 75 and 100 hrs. The microscopic observation of the shape and size of the powdered material grains was carried out by means of the OPTON DS 540 scanning electron microscope. The changes of the powder structure were tested by means of the X-ray diffractometer of the DRON-2. The crystallites size was measured by the Scherrer's method basing oneself on the diffraction records.

4. RESULTS OF THE INVESTIGATIONS

From the tests carried out on the scanning electron microscope it results that the average grain size of the Fe – 6,67mass.% C powder decreases together with the increased time of grinding, as shown in Fig. 1. The Fig. 1 shows the sequent powder structures after 10, 25, 50, 75 and 100 hours of grinding. During the process, the powders become effectively broken up while the equilibrium between the cracking and joining is fixed, what results in the relatively stable grain sizes of the powder after the prolonged time of grinding (see Fig. 1 – after 100 hours). The particles of the powder collide with the grinding media, walls of the container and also with themselves. The particles of the powder are cold pressure welded and their morphology changes from spherical to more lamellar as well as more fine particles are generated which cluster around the larger ones. The large aggregations are crushed whereas smaller ones which can resist deformations without cracking are joined in larger systems. During the mechanical alloying the crystallites are flattened, joined, crushed and rejoined many times. That gives rise to considerable changes in the microstructure of the material and in the chemical composition. The phase X-ray analysis proved the changes occurring in the MA process. The X-ray diffraction patterns of the powders showed also the dependence of changes in the structure on the time of grinding. The diffraction records of powders vs. the time of grinding are shown in Fig. 2. The diffraction pattern recorded for the powder ground for 10 hours shows the peaks characteristic for α-Fe, Fe_3C, whereas none X-ray peak originated from the graphite was observed. When the grinding time increases, all X-ray peaks become wider and their intensity decreases. The widening of peaks is connected with the size reduction in the powder grain as well as with the presence of considerable stresses resulting from the intensive plastic strains occurring during the grinding. The amorphous phase became visible in the last sample after 100 hours of mechanical alloying. It appeared in the form of a wide peak for angles 2θ equal about 52,4°. The powdering of the crystalline structure is increased together with the increase in the grinding time, what brings about the rendering the alloy to be partially amorphous again. The volume fraction of the amorphous phase increases progressively.

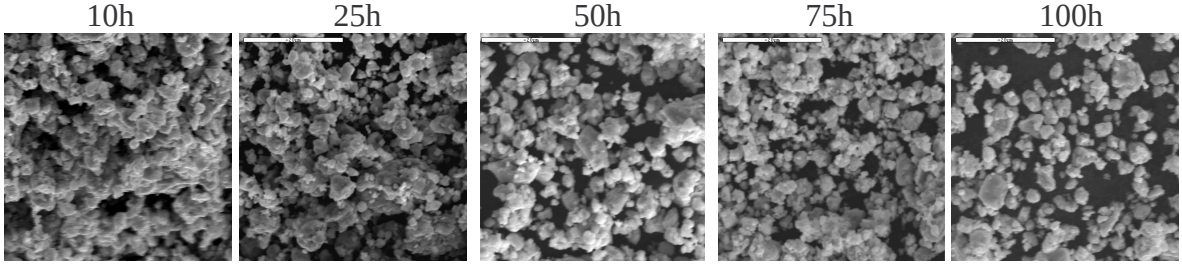

Figure 1. Structure of Fe - 6,67mass.% C powder after 10, 25, 50, 75 and 100 hours of mechanical alloying (magnification 2000 X)

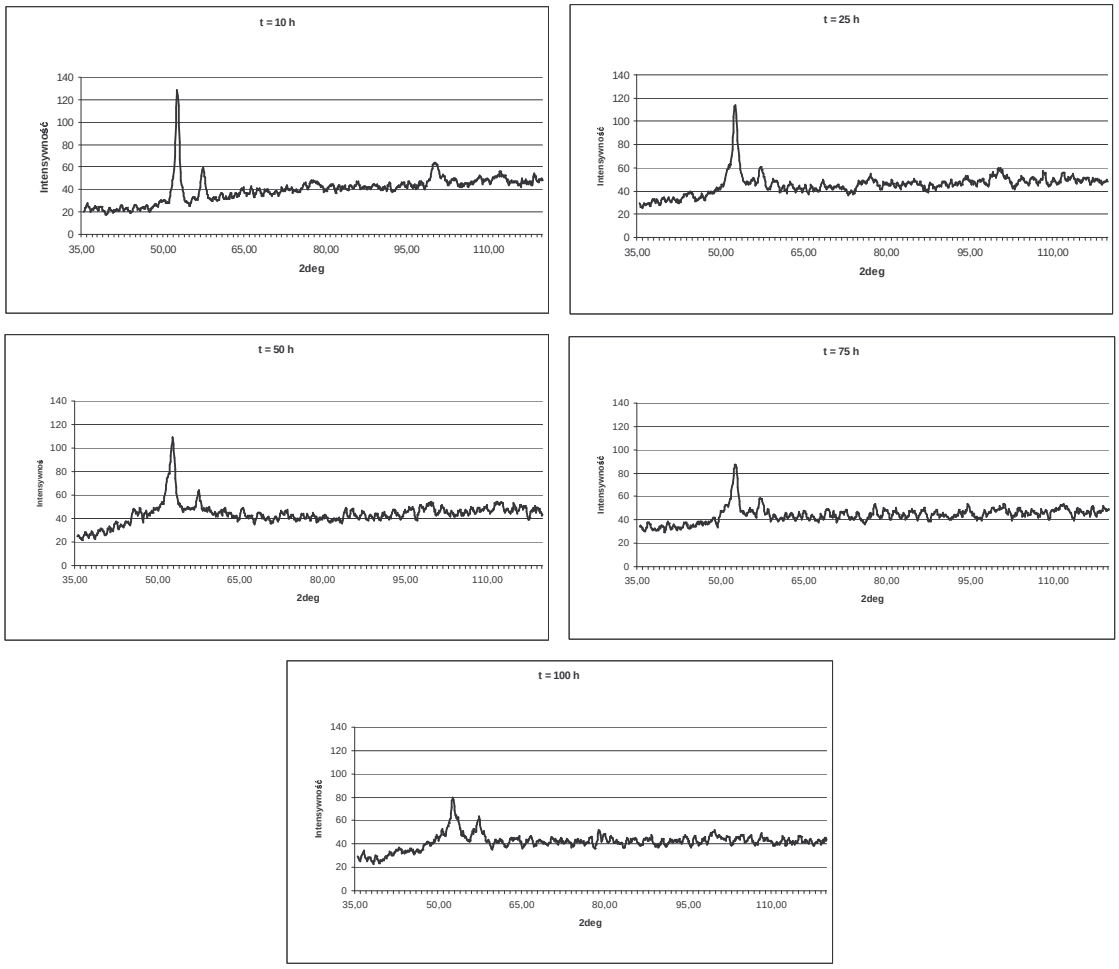

Figure 2. The phase X-ray diffraction patterns of Fe – 6,67mass.% C alloy vs. the grinding time: 10, 25, 50, 75 and 100 hours

The diffraction patterns obtained on the basis of the phase X-ray analysis allowed, applying the Sherrer's method, to determine the crystallite size in the powder. As it was observed, the crystallite sizes decreased and the powder structure became more homogeneous. The results of calculations are presented in Table 1.

Table 1.
Crystallite size vs. time of grinding

	Time of grinding, hours	Crystallite size, nm
1	10	14,7
2	25	13,8
3	50	12,8
4	75	7,9
5	100	5,0

The crystallite size of the Fe – 6,67% C alloy decreases systematically when the time of mechanical alloying increases. After 25 hours of grinding, the crystallite size was estimated as being 13,8 nm and after 100 hours as being 5 nm.

Basing oneself on the literature data, one can suppose that during the first stage of grinding the graphite becomes amorphous and simultaneously the plastic deformation of the iron powders takes place. Having analyzed the results of the works [6, 1], it can be assumed that at this stage of the MA the carbon content in the particles of the α-Fe phase is low. The crystal lattice of iron becomes deformed. During the further grinding, the cracks and joining of particles are repeated. The amorphous carbon adherent to the surface of the crystal lattice of iron diffuses into its interior. The diffusion of carbon atoms into the crystal lattice of iron proceeds and the solid solution of carbon in iron is formed. Together with the prolongation of the grinding time the content of carbon atoms in their solid solution in iron increases and the supersaturated solution of carbon in iron appears. According to the publications [6, 1], the supersaturated solid solution of carbon in iron becomes progressively amorphous at the further stage of the MA process. At this stage, the rendering the Fe-C alloy amorphous and the diffusion of carbon atoms into the crystal lattice of iron run parallel. Finally, the carbon content reaches the solubility limit and the iron and carbon powders become amorphous.

5. CONCLUSIONS

The results of the investigation show that the application of the mechanical alloying process for grinding of iron and carbon powders enables to fabricate nanocrystalline alloys in amorphous matrix of pure elements. The MA process allows to obtain finely-dispersed carbide phase in ferrite matrix. The process can be applied, among many other methods, for the production of a new nanocomposite material, namely Fe-Fe_3C.

As the time of mechanical alloying increases, the crystallites size is reduced and progressively the alloy becomes amorphous.

The powdered nanocomposite material, after forming and sintering, can be applied for the manufacture of the material characterized by high parameters such as: hardness, tensile strength and wear resistance.

REFERENCES

1. Rochman Nurul Taufiqu, Kawamoto K., Sueyoshi H., Nakamura Y., Nishida T.: “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), s. 367-372.
2. Wang G. M., Campbell S. J., Calka A., Kaczmarek W. A.: “Ball-milling of Fe-C (20-75% Fe)”. NanoStructured Materials, Vol.6, 1995, s. 389-392.
3. Umemoto M., Liu Z. G., Masuyama K., Hao X. J., Tsuchiya K.: ”Nanostructured Fe-C alloys produced by ball milling”. Scripta mater. 44, 2001, s. 1741-1745.
4. Shabashov V. A., Korshunov L. G., Mukoseev A. G. i in.: “Deformation-induced phase transitions in a high-carbon steel”. Materials Science and Engineering A346, 2003, s. 196-207.
5. Rochman Nurul Taufiqu, Kawamoto K., Sueyoshi H., Nakamura Y., Nishida T.: “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), s. 367-372.
6. Yelsukov E. P., Dorofeev G. A., Zagainov A. V., Vidanova N. F., Maratkanova A. N.: “Initial stage of mechanical alloying in the Fe – C system”. Materials Science and Engineering A369, 2004, s. 16-22.

7. Formanek B., Maciejny A., Szopiński K., Olszówka – Myalska A.: „Kompozytowe materiały proszkowe otrzymywane metodą mechanicznego stopowania". Inżynieria materiałowa, 3 – 4, 1999, s. 137 – 143.
8. Goodwin T. J., Yoo S. H., Matteazzi P., Groza J. R.: "Cementite – iron nanocomposite". NanoStructured Materials, Vol.8, 1997, s. 559-566.
9. Hidaka H., Tsuchiyama T., Takaki S.: "Relation between microstructure and hardness in Fe-C alloys with ultra fine grained structure". Scripta mater. 44, 2001, s. 1503-1506.
10. Tokumitsu K., Umemoto M. Materials Science Forum 183, 2001, s. 360 – 362.
11. Tokumitsu K., Umemoto M. Materials Science Forum 479, 2002, s. 386 – 388.
12. Jurczyk M.: „Mechaniczna synteza". Wydawnictwo Politechniki Poznańskiej, 2003.
13. Shabashov V. A., Korshunov L. G., Mukoseev A. G. i in.: "Deformation-induced phase transitions in a high-carbon steel". Materials Science and Engineering A346, 2003, s. 196-207.
14. Cao H.S.: "Determination of mechanical properties of consolidated nanocrystalline iron – carbon alloys Fe-0,5%C and Fe-4,5%C by means of echography and acoustic microscopy". Materials Letters 58, 2004, s. 3564 – 3568.
15. Suryanarayana C.: "Mechanical alloying and milling". Progress in Materials Science 46, (2001), s. 1 – 184.
16. Liu Z.G., Fecht H.J., Xu Y., Yin J., Tsuchiya K., Umemoto M.: "Electron – microscopy investigation on nanocrystal formation in pure Fe and carbon steel during ball milling". Materials Science and Engineering A362, 2003, s. 322-326.