

The Structural Properties of Fe-Ti-B Based Alloys Produced by Mechanical Alloying

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In the present study, the production of Fe-Ti-B based alloys was realized and their structure and properties were investigated. Mechanical alloying proceeds by the continual cold welding and fracturing of the constituent mixture of Ti+4B+5Fe powder when subjected to the large compressive forces of a high speed mill. The powder charge together with 7 mm diameter steel balls were loaded into a tool steel grinding container at approximately 350 RPM for 20 h. The samples were shaped as cylinder of $\text{Ø}15 \times 8$ mm dimensions by uniaxial pressing at 450 MPa. Then, the green body materials were produced by sintering at 1100 °C for 1–4 h in argon atmosphere. The morphology of composite materials was investigated by optical microscopy and scanning electron microscopy and phase analysis was realized by x-ray diffraction analysis. The bulk densities of the materials were measured using by Archimedes method. Also, the micro-hardness of the samples was measured by Vickers indentation technique. As a result, Fe, iron boride (FeB, Fe₂B) and titanium boride (TiB₂) phases were detected in the phase analysis of the Fe-Ti-B based materials. The hardness of the materials was measured between 1107 HV_{0.05} and 1551 HV_{0.05}, depending on sintering time. The densities of the samples were determined between 4.205 g/cm³ and 4.219 g/cm³.

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1. Introduction

The exothermic dispersion process technology was used with mechanical alloying and sintering process to produce a new modified composite having TiB₂ and Fe₂B reinforcement particulates. This process utilizes a mixture of powders of the ferrous alloy powders components with a third metallic component. Heating of this mixture causes the exothermal interaction between the components. Steel-based material is a kind of metal material of extensive application, and has such merit as low price, no pollution in the fabrication, convenience in the reuse, and good process ability. Moreover, its mechanical and physical properties can be adjusted by heat treatment. TiB₂ particles are selected because they possess a very high hardness, high melting point, high strength, excellent corrosive resistance, good thermodynamic stability as well as good thermal conductivity [1].

Recently, MA has often been used to synthesize the metal carbides, borides and silicides, which provide a novel route to prepare fine-grained ceramic powders. MA is an alternative solid state technique by which novel materials may be synthesized from elemental or pre-alloyed powders [2–4].

It is well established that the incorporation of hard ceramic particulates (e.g. carbides and borides) to ferrous matrices can significantly improve certain material properties, such as wear resistance, toughness and

strength. Therefore, ceramic particulate reinforced Fe and steel matrix composites have received considerable attention in recent years. Titanium diboride (TiB₂), possessing outstanding features such as high melting point (2790 °C), high hardness (33 GPa), high modulus (530 GPa) and low density (4.451 g/cm³), is another potential reinforcement. Generally, there are several methods of fabricating the particulate reinforced steel matrix composites, such as powder metallurgy (PM), conventional melting and casting, carbo-thermic reduction, self-propagating high-temperature synthesis (SHS) (also called combustion synthesis) and alumino-thermic reduction [5, 6].

A ternary system, which gains the attention of scientists, is the Fe-Ti-B system because of the formation of TiB₂ particles. These particles have ideal properties for being used as a reinforcing material, such as high hardness and melting point, good corrosion resistance and thermal conductivity. The majority of studies focus on the Fe-TiB₂-TiFe₂ region of the ternary diagram, while the Fe-TiB₂-Fe₂B region remains unexplored, although it presents a great potential [7].

In present study, fabrication, structural and mechanical properties of the in-situ TiB₂ and Fe₂B reinforced steel matrix composites by the mechanical alloying treatment of the powders and sintering process was studied.

2. Experimental procedure

Ferrous boron, ferrous niobium and Armco-iron (ASC.100.29) powders were used in the study. The nominal compositions (wt.%) of ferrous boron and ferrous titanium alloys used in the study were as follows: 19.63%

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B, 0.44% C, 0.05% Al, 0.98% Si, balance Fe and 68.4% Ti, 0.13% C, balance Fe, respectively. Ferrous boron and titanium were grounded by ring grinder and resieved to be 100 μ m grain sizes. At the first step of the study, the mixture of Ti+4B+5Fe powders was mechanically alloyed in the attritor at 350 rpm for 20 h in argon atmosphere. The steel balls with 7 mm diameter were used as a grinding media with a ball-to-powder ratio of 5:1. At the second step, mechanically alloyed powders were shaped as cylindrical coupons at the dimensions of 15 mm in diameter and 8 mm in thickness by uniaxial dry pressing under the pressure of 450 MPa. Polyvinyl alcohol was used as a binder material. After shaping, samples were dried at 80 °C for 24 h in an oven. Dried samples were sintered in an electrical furnace with a heating rate of 6 °C/min at 1100 °C for 1–4 h in the Ar atmosphere. Then, the samples were cooled down to room temperature in the furnace.

The microstructure of sintered materials was observed by using optical microscope (Nikon Epiphot) and scanning electron microscope (SEM) (Jeol JSM 6060 LV) to the samples which were ground on silicon carbide papers to 1200 grit, and then progressively polished with, 0.3 μ m Al₂O₃ paste. An x-ray diffractometer (Rigaku XRD/D/MAX/2200/PC) with CuK α radiation was used to analyze the constituent phases in the microstructure. The bulk densities of the materials were measured by Archimedes method. Also, the micro-hardness of the samples was measured by using Future Tech FM 700 microhardness tester by Vickers indentation technique with 50 g load.

3. Results and discussions

Figure 1 shows the SEM image and EDS analysis of the mechanically alloyed Ti+4B+5Fe powders. As shown on Fig. 1a, the powder was established well ground and agglomerated structure, and EDS analysis (Fig. 1b) shows that the elemental distribution of the powder which was including Fe, Ti and B beside trace element of Al which was coming from the ferrous alloys. Figure 2a and b shows optical and SEM images of the mechanically alloyed and sintered Ti+4B+5Fe alloys at 1100 °C for 4 h. Optical and SEM micrographs include well distributed different color grain structure phases. X-ray diffraction analysis showed that the phases formed in the sintered sample at 1100 °C for 1 h are Fe₂B, FeB, TiB₂ and Fe. It is clear from the X-ray diffraction analysis that the alloy produced from the mechanically alloyed powders includes different boride phases and Fe and so, microstructure of the produced samples includes these phases' grains as different contrast. EDS analysis supported the X-ray diffraction analysis as shown in Fig. 2c, d and e. Possible reactions took place during the sintering process are follows [8–10].

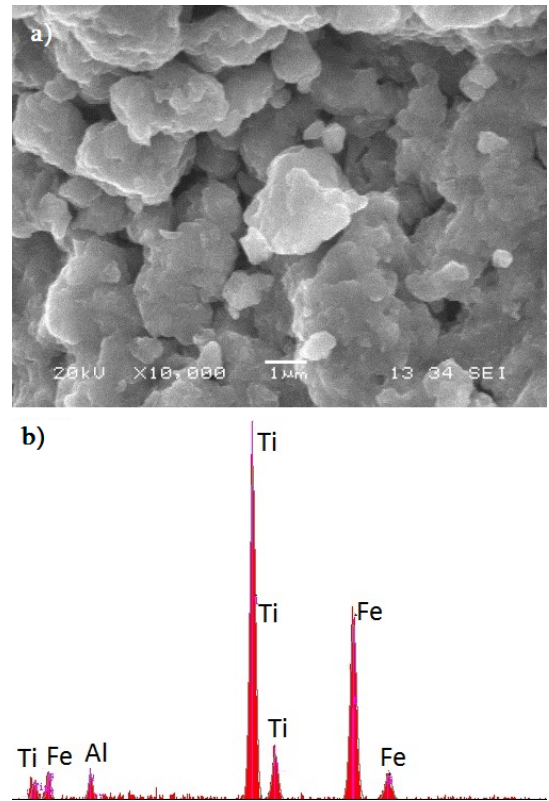
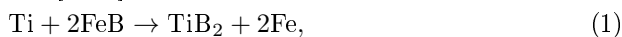


Fig. 1. SEM images and EDS analysis of mechanically alloyed Ti+4B+5Fe powders.

Ferrous boron includes FeB phase and ferrous titanium includes 68.4%Ti by wt. As a result, TiB₂ and Fe₂B phases realized from the ferrous alloys. Raghavan's phase diagrams of Fe-Ti-B demonstrate that the phases took place in the diagram of Ti+4B+5Fe concentration are Fe₂B, TiB₂ and Fe. As shown from Fig. 3 that the presence phases in the study includes FeB phase. It is probable that the reactions to be realized in the powder mixture will be continue during sintering process at 1100 °C over 1 h sintering time up to used up the FeB phase in.

Figure 4 shows the density and micro-hardness of the samples sintered at 1100 °C for 1–4 h. As shown from the figure that the increase in the sintering time caused to increase of densities of the sintered samples. As known, the density of the sintered materials increased with increase in sintering time [11]. It is possible that the increase in sintering time caused to decrease of porosity and realized the reactions took place in the powder mixture.

Vickers micro-hardness test was realized on the sintered samples. As shown from Fig. 4, increase in sintering time caused to increase the formation of boride phases and decrease of the porosity in the sintered samples. The samples produced include Fe and borides of Ti and Fe. So it's an in-situ composite structure. The hard boride phases increment will be caused to increase of the materials hardness [12]. The hardness of the Fe₂B and TiB₂ phases is between 1500–1700 HV [13] and 2900 HV

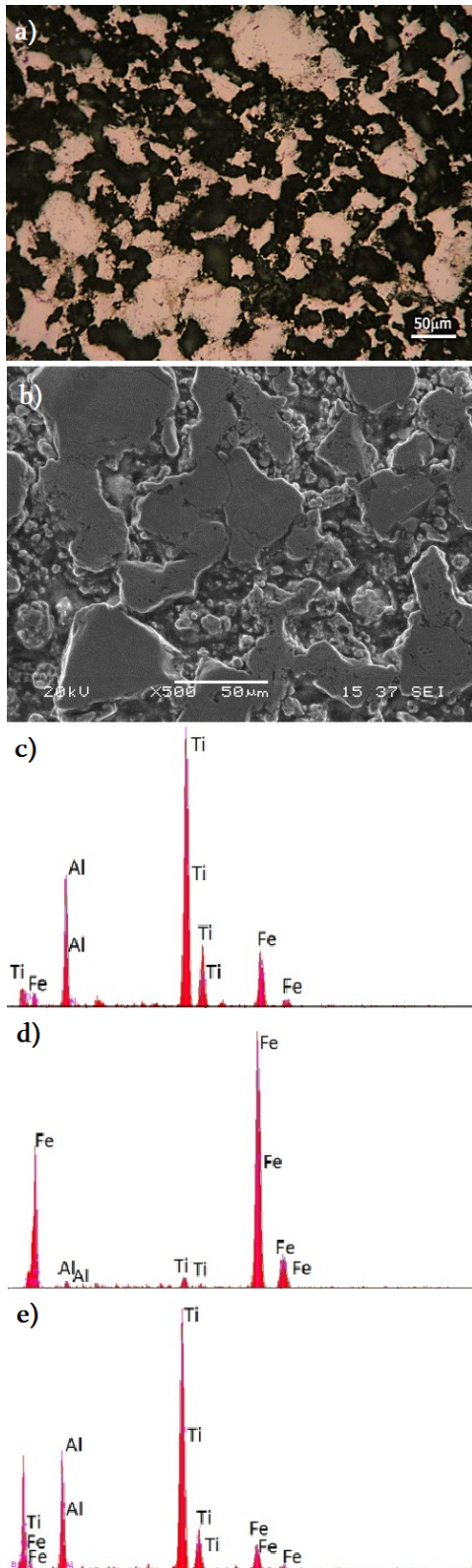


Fig. 2. a) Optical and (b) SEM micrographs of Ti+4B+5Fe alloys at 1100 °C for 4 h and (c, d and e) EDS analysis of marked on SEM image as 1, 2 and 3, respectively.

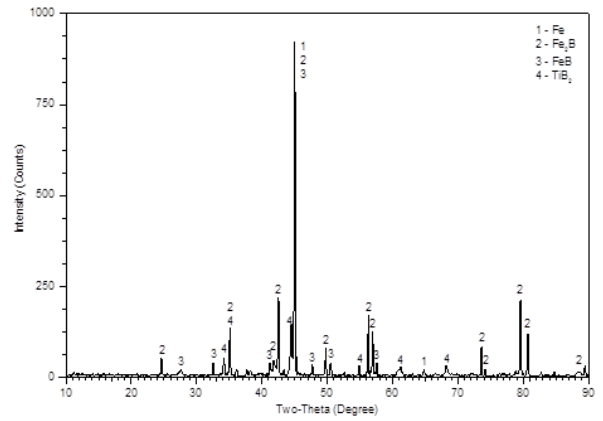


Fig. 3. X-ray diffraction analysis of Ti+4B+5Fe alloys at 1100 °C for 1 h.

to 3400 HV [14].

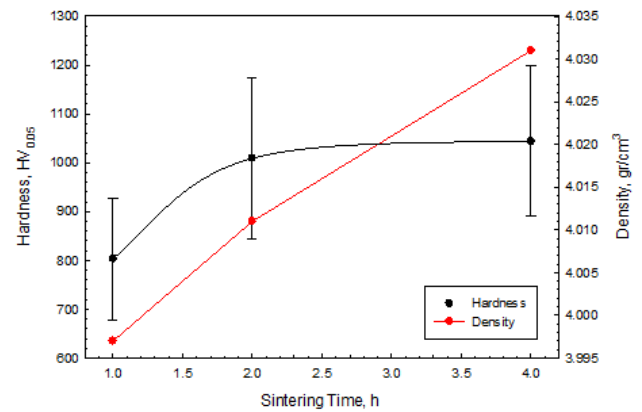


Fig. 4. Density and hardness of the Ti+4B+5Fe alloys sintered at 1100 °C.

4. Conclusions

Based on all of the experimental results obtained in this work, the following conclusions could be drawn:

Mechanically alloyed Ti+4B+5Fe powders produced from the commercial ferrous alloys were established well ground and agglomerated structure.

The powders sintered at 1100 °C can produce in-situ composites with both TiB₂ and Fe₂B as reinforcing particles. Stable phases present in the ternary Fe-Ti-B diagram are formed.

The presence phases in the sintered samples include Fe₂B, TiB₂, Fe and FeB phases.

Increase in the sintering time caused to increase of density of the sintered samples. Increase in sintering time caused to increase the formation of boride phases and decrease of the porosity in the sintered samples.

Increase in the sintering time caused to increase of the hardness of the sintered in-situ composites of TiB₂, Fe₂B and Fe. Increase in sintering time caused to increase the

formation of boride phases and decrease of the porosity in the sintered samples, and so the hardness of the in-situ composite increased.

Acknowledgments

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References

- [1] Z. Xue, J. Kuang, H. Fu, *Mat.-wiss. u. Werkstofftech* **39**, 557 (2008).
- [2] W. Tang, Z. Zheng, Y. Wu, J. Wang, J. Lü, J. Liu, *Trans. Nonferrous Met. Soc. China* **16**, 613 (2006).
- [3] T.S. Ward, W. Chen, M. Schoenitz, R.N. Dave, E.L. Dreizin, *Acta Materialia* **53**, 2909 (2005).
- [4] J.J. Sunol, A. Gonzalez, J. Saurina, L. Escoda, P. Bruna, *Materials Sci. and Engineering A* **375**, 874 (2004).
- [5] Y. Wang, Z.Q. Zhang, H.Y. Wang, B.X. Ma, C.Q. Jiang, *Materials Sci. and Engineering: A* **422**, 339 (2006).
- [6] Y. Wang, H.Y. Wang, K. Xiu, H.Y. Wang, Q.C. Jiang, *Materials Letters* **60**, 1533 (2006).
- [7] M. Darabara, G.D. Papadimitriou, L. Bourithis, *Surface & Coatings Technology* **201**, 3518 (2006).
- [8] J. Balogh, L. Bujdosó, T. Kemeny, I. Vincze, *Journal of Physics: Condensed Matter* **9**, 503 (1997).
- [9] I. Campos-Silva, M. Ortiz-Dominguez, C. Tapia-Quintero, G. Rodriguez-Castro, M.Y. Jimenez-Reyes, E. Chavez-Gutierrez, *Journal of Materials Engineering and Performance* **21**, 1714 (2012).
- [10] D. Demirskyi, Y. Sakka, *Journal of the European Ceramic Society* **35**, 405 (2015).
- [11] S.J.L. Kang, in: *Sintering: Densification, Grain Growth and Microstructure*, Oxford: Elsevier Butterworth Heinemann, 2005.
- [12] Y. Li, M.L. Swartz, R.W. Phillips, B.K. Moore, T.A. Roberts, *Journal of Dental Research* **64**, 1396 (1985).
- [13] S.O. Yilmaz, M. Ozenbas, *Journal of Mater Science* **44**, 3273 (2009).
- [14] G. Hilz, H. Holleck, *Materials Science and Engineering A* **139**, 268 (1991).