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Production of (V-B) Reinforced Fe Matrix Composites

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***Abstract*—**Metal matrix composites (MMCs) have gained a considerable interest in the last three decades. Conventional powder metallurgy production route often involves the addition of reinforcing phases into the metal matrix directly, which leads to poor wetting behavior between ceramic phase and metal matrix and the segregation of reinforcements. The commonly used elements for ceramic phase formation in iron based MMCs are Ti, Nb, Mo, W, V and C, B. The aim of the present paper is to investigate the effect of sintering temperature and V-B addition on densification, phase development, microstructure, and hardness of Fe–V-B composites (Fe-(5-10) wt. %B – 25 wt. %V alloys) prepared by powder metallurgy process. Metal powder mixes were pressed uniaxial and sintered at different temperatures (ranging from 1300 to 1400ºC) for 1h. The microstructure of the (V, B) Fe composites was studied with the help of high magnification optical microscope and XRD. Experimental results show that (V, B) Fe composites can be produced by conventional powder metallurgy route.

***Keywords*—**Hardness, Metal matrix composite (MMC), Microstructure, Powder Metallurgy.

I. Introduction

METAL matrix composites (MMCs) have gained a considerable interest in the last three decades [1], [2]. The use of Metal Matrix Composites (MMCs) is considered as an excellent combination of hard ceramic reinforcements and ductile metallic matrix, which makes them a promising material for wear-resistance applications [3]. The composite generally has more superior characteristic than those of each of the individual components. [4]. MMCs are widely used in several industrial areas such as aerospace, automotive and electronics [5], [6]. It has been observed that properties of MMCs are greatly influenced by the nature of reinforcement and its distribution in the metal matrix [7]. Properties of the composites are also influenced by the chemical nature of components, morphology of particles, their distribution and interface reactions [8]. Particle size, however, is an important factor which is directly related to the strength of the composites [9], [10]. There have been significant advancements in the processing techniques to control the microstructure and resulting mechanical properties of MMCs [11], [12]. Conventional powder metallurgy production route often involves the addition of reinforcing phases into the metal matrix directly, which leads to poor wetting behavior between ceramic phase and metal matrix and segregation of reinforcements. The commonly used elements for ceramic phase formation in iron based MMCs are Ti, Nb, Mo, W, V and C, B [13]-[15]. Ceramic phase could also be formed by crystallization or by precipitation in supersaturated solid solution. Each of the elements added into iron changes the enthalpy of element by formation of an alloy phase [16]. Generally, the binary Fe-B system is used to develop the metal matrix composites [17]. It is very well known that the hardenability of steel increases by increasing boron addition. However, the solubility limit of boron in iron is very low, either in austenite or in ferrite and more addition could induce the formation of boride phases. Moreover, carbon is almost dissolved in borides, thus the properties of matrix can be adjusted by carbon content. This offers a unique possibility to increase hardness of reinforcement in MMC by using borides and at the same time adjust the properties of the matrix by levelling the carbon content. Gou [18] found that the solubility of boron in iron can be changed with the addition of other elements such as Cr, Mo and V. It was also found that adding Cr element changes the toughness of boride particles (Fe, $Cr)_2B$ in the system significantly as well as mechanical properties of the matrix [19], [20]. The effect of chromium has clearly shown that the abrasive resistance and higher toughness significantly changes the wear resistance [15]. The aim of the present paper is to investigate the effect of sintering temperature on relative density, phase identification, microstructure and hardness of V-B reinforced Fe matrix composites (5-10 wt.% B; 75 wt.% Fe-25 wt.% V), prepared by powder metallurgy process.

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II. Experimental Procedure

The composites samples were prepared by powder metallurgy from Fe, V and B powders. Carbonyl iron powder having 99, 5 wt.% purity and particle size of 45 μm are used as starting material. Vanadium powder with a particle size of 45 μm and boron particles with ≤10 μm size was used as the reinforcement powders. The Fe (V) composites containing 5 wt.% and 10wt. % boron were fabricated by a PM method. The powders were blended for 45 min in a T_2F Turbula® mixer, with 2 wt.% zinc stearate added before blending to obtain a uniform and homogenous mixture. The mixed powders were cold pressed into pin specimens under a pressure of 250 MPa. These specimens were sintered at 1300°C and 1400°C in a tube furnace in an argon gas atmosphere for 1 h. The zinc stearate was heated up to 500°C and held for 5 min. and a brown product was produced and heated up to the sintering temperature followed by a holding time of 1 h and cooled to room temperature (5°C/min). The dimensions of cylinder specimens were 12 mm in diameter and 3 mm in height. The theoretical, measured and resulting relative density of the sintered specimens was measured using the Archimedes' principle. The hardness of the pure alloy and reinforced composites were measured by Vickers hardness

(VHN) method and the mean of at least eight readings was taken. A X-ray Diffractometer (Rigaku D-MAX 2200/PC) with a monochromatic Cu-Kα radiation (λ = 1.5408 Å) was used over a 2θ angle from 20° to 80° to characterize the crystal structure of the sintered compacts. The surface microstructures of the samples were obtained through Nikon® MA200 optical microscope with Clemex vision Image Analyser.

III. Results And Discussion

A. Density Measurement and Hardness

The bulk and relative density of the sintered materials were determined using Archimedes' water displacement method, as specified by European Standard EN 99 (ISO 10545-3, 1991) [20]. Theoretical and relative densities of the 5-10 wt. % boron and 25 wt. % vanadium reinforced iron based composites are shown in Table I.

TABLE I
THEORETICAL, MEASURED AND RELATIVE DENSITY OF THE (V, B) REINFORCED FE MATRIX COMPOSITES

Material Types (wt. %)	Theoretical density (g/cm^3)	Measured density (%) 1300°C	Measured density (%) 1400°C	Relative density (%) 1300°C	Relative density (%) 1400°C
Fe-25V	7,292	6,1	6,755	83,65±0,4	92,63±0,5
Fe-25V-5B	6,5748	5,74	6,531	87,3±0,2	99,3±0,6
Fe-25V-10B	5,984	5,181	5,906	86,58±0,5	98,9±0,4

Vickers Micro hardness tests were performed on three samples for each unique composition. Eight measurements were conducted on each specimen. Vickers Microhardness of composites was determined using a Schimadzu® MHV tester under an applied load of 1 kg-f. For hardness tests the average hardness values and standard deviation were calculated. The results of hardness test on the all materials are shown in Fig. 1. At 1300°C sintering temperature, although the relative density is around 85%, the hardness values tend to increase due to increasing amount of B in matrix to form more ceramic phases around grain boundaries. By increasing temperature, the densification was almost completed and all hardness values were increased from 70 VHN to 290 VHN for Fe (V) matrix and from 110 VHN to 390 VHN for 10 wt.% B; 75 wt.% Fe-25 wt.% V containing composites, respectively. This situation can be attributed to the increasing boron content and dissolution of more Fe-B phases into grains and grain boundaries.

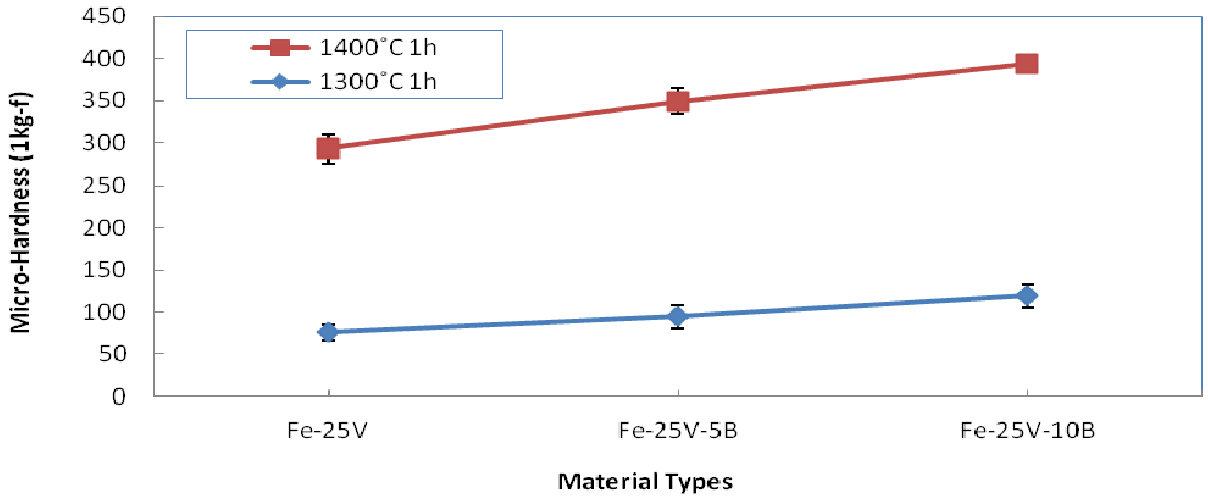

Fig. 1 Hardness of (V, B) reinforced Fe matrix composites

B. XRD Analysis

The room-temperature x-ray diffraction patterns recorded for Fe matrix composites added with 25 wt.% V and 5-10 wt.% B sintered at 1300-1400°C are shown in Figs. 2 and 3. All the peaks are indexed according to the PDF files No. 85-1410, 18-0664 and 75-1065. In Fig. 2 the formation of (Fe, V) B solid solution in all the intermediate compositions is evidenced and peak shift corresponds to the change in the Fe/V ratio. It was found that a small amount of Fe_2B as a minor phase and Fe as a major phase are present in the composite specimens. This shows that a slight reaction between iron and boron due to the low sintering temperature occurs. As seen from Fig. 2, increasing boron content also leads to the formation of FeB and Fe_2B phases due to the dissolution-precipitation mechanism.

In Fig. 3 (a), pure Fe (V) solid solution phase can be seen in sintered sample at 1400°C for 1h. Fig. 3 (b) represents the increase of boron in composite, since the B content increased up to 5 wt.%, FeB phase occurs predominantly by a diffusional reaction. By increasing boron to 10 wt.%, another Fe-B phase Fe_2B is also formed due to solution limits of boron in Fe. Thus, in this Fe-V-B composite system sintering

mechanism can be considered as reactive liquid phase sintering. By solution-reprecipitation mechanism in liquid phase sintering, FeB and Fe_2B phases are found as dispersed in Fe-V matrix.

C. Microstructural Analysis

Microstructure of the composite was examined using Nikon® MA200 optical microscope with Clemex vision Image Analyser. After grinding and polishing, the composite samples were etched with 2 vol % nitric acid-alcohol solution.

Fig. 4 is a combination of temperature and boron content evaluation in microscope image for the (V, B) reinforced Fe matrix composites after sintering 1300 and 1400°C for 1h.

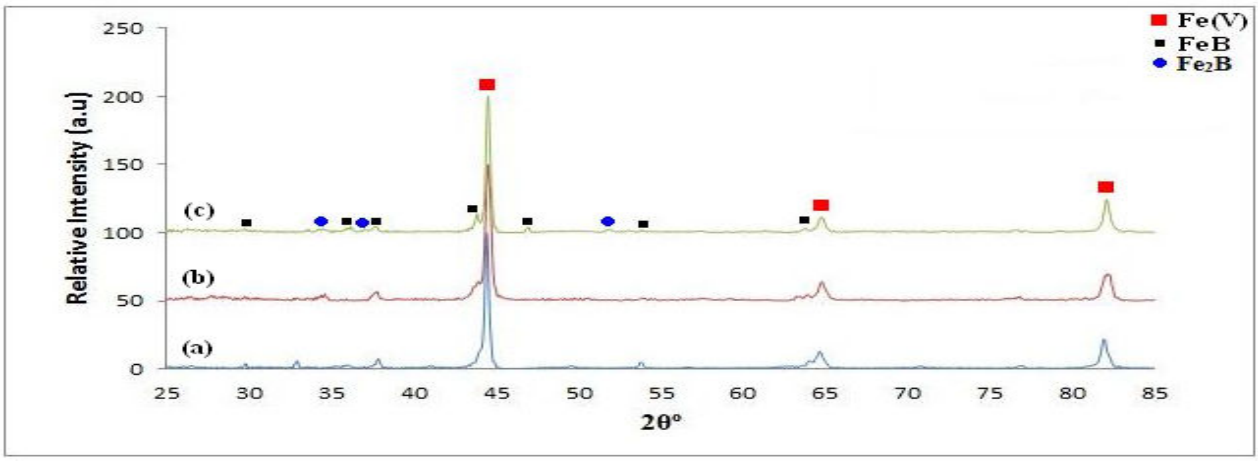

Fig. 2 XRD patterns of Fe based composites with 25 wt. %V and 5-10 wt. % B sintered at 1300°C for 1 h. (a)Fe-25V, (b) Fe-25V-5B, (c) Fe-25V-10

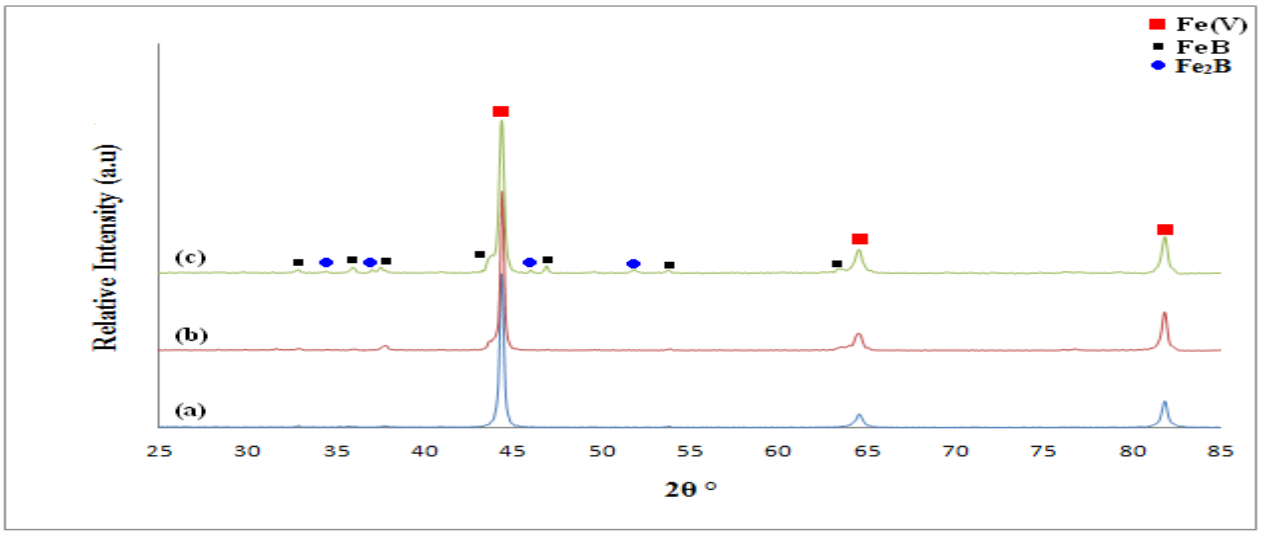

Fig. 3 XRD patterns of Fe based composites with 25 wt. %V and 5-10 wt. % B sintered at 1400°C for 1 h. (a)Fe-25V, (b) Fe-25V-5B, (c) Fe-25V-10B

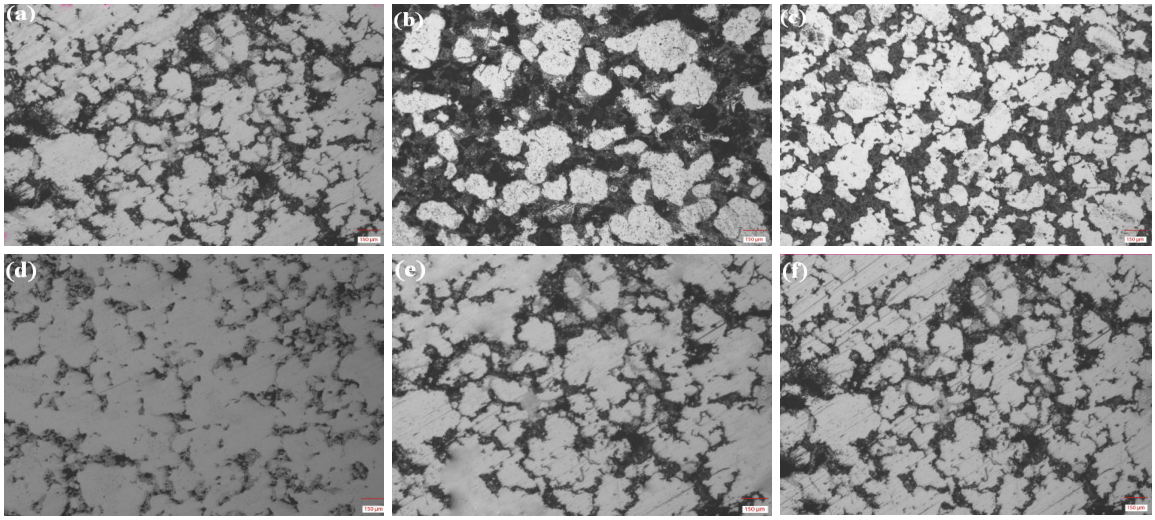

Fig. 4 Microscope images of the (V-B) reinforced Fe matrix composites. (a) Fe-25V (1300)°C, (b)Fe-25V-5B (1300)°C, (c)Fe-25V-B (1300)°C, (d)Fe-25V (1400) °C, (e) Fe-25V-5B (1400) °C, (f) Fe-25V-10B (1400)°C

The dark areas are considered as Fe (V)-B particles due to the lower amount of B and the lighter region is Fe(V) matrix and pores are seen as black regions. Vanadium and boron particles can be seen as uniformly dispersed in matrix. Figs. 4 (a)-(c) clearly show that the presence of Fe rich phase in a diameter of 150 μm ‘‘islands” can be seen well as a unique characteristic of rearrangement stage of liquid phase sintering. This means that all other constituents are dissolved from this Fe(V) islands. Porosity reduction and microstructure refinement were observed in Figs. 4 (d)-(f) as the sintering temperature increased from 1300 to 1400°C. Negligible amount of porosity is present in the specimens sintered at 1400°C. It is worth to note that the microstructures with higher temperature presented the (V, B) rich phase well distributed into the Fe rich phase in a more homogeneous and refined manner. Strong Fe-B precipitates can also be observed for the composite alloys as proved by XRD analysis (Fig. 4 (f)).

IV. Conclusion

The effects of reinforcement addition on sintering behavior, phase identification, microstructure and hardness of V-B reinforced Fe matrix composites are investigated. Using a conventional process which combines *in situ* reactive production with powder metallurgy technique, iron base composites reinforced by (V, B) particles were produced. The vanadium and boron particles formed in situ ceramic phases which are uniformly dispersed in the matrix. Additionally, with the increasing of boron ratio, morphology of (V, B) Fe particles transforms from irregular to spherical shapes due to higher amount of dissolution-precipitation characteristics specified by binary phase diagrams. Increased densification with increasing sintering temperature, reach maximum for the specimens sintered at 1400°C. The reasons might be as follows: with the increase of sintering temperature, the solution of V and B in Fe are increased by increasing temperature which intensifies the dissolution–precipitation of (V, B) Fe particles and increases the amount of liquid phase. As a result, densification increases due to the increasing flow ability of Fe by B increase in alloy. Because of increasing total ceramic phase content, not only in grains but also in grain boundaries, the micro hardness of B reinforced Fe-V composites increases.

References

[1] Tjong S C and Ma Z Y 2000 *Mater. Sci. Engg.* 29 49.
[2] Rabiei A, Vendra L and Kishi T 2008 *Compos. Part A: Appl. Sci. Manuf.* 39 294.
[3] Acosta, P., Jimenez, J. A., Frommeyer, G., Ruano, O. A., Microstructural characterization of an ultrahigh carbon and boron tool steel processed by different routes. Materials Science and Engineering, 1996, vol. A206, pp. 194-200.
[4] Krishan K. Chawla, N. Chawla, “Metal-Matrix Composites” vol 016 , 2004.
[5] Aldas K and Mat D M 2005 *J. Mater. Proc. Technol.* 160 289
[6] Mehdi Rahimian, Naser Ehsani, Nader Parvin and Reza Baharvandi Hamid 2009 *J. Mater. Proc. Technol.* 209 5387.
[7] Roy D, Ghosh S, Basumallick A and Basu B 2006 *Mater. Sci. Eng.* A415 202.
[8] Reddy B S B, Rajasekhar K, Venu M, Dilip J J S, Das Siddhartha and Das Karabi 2008 *J. Alloys Compd.* 465 97.
[9] Wei Yueguang 2001 *Acta Mechan. Sinica* 17 ISSN 0567-7718
[10] Chen S H and Wang T C 2002 *Acta Mechan.* 157 113.
[11] Shen Y L and Chawla N 2001 *Mater. Sci. Engg.* A297 44.
[12] Pagounis E, Talvitieb M and Lindroos V K 1996 *Compos. Sci. Technol.* 56 1329.
[13] Choteborsky, R., Hrabe, P., Muller, M., Savkova, J., Jirka, M., Navratilova, M.: Effect of abrasive particle size on abrasive wear of hardfacing alloys, Research in Agriculture Engineering. 2009, vol. 55, no. 3, pp. 101-113, ISSN1212-9151
[14] Choteborsky, R., Hrabě, P., Muller, M., Valek, R., Savkova, J., Jirka, M.: Effect of carbide size in hardfacing on abrasive wear, Research in Agriculture Engineering. 2009, vol. 55, no. 4, pp. 149-158, ISSN1212-9151.
[15] Zhang, J., Gao, Y., Xing, J., Ma, S., Yi, D., Yan, J.: Effects of Chromium Addition on Microstructure and Abrasion Resistance of Fe–B Cast Alloy, Tribology Letters. DOI 10.1007/ s11249-011-9823-5.
[16] Kootsookos, A., Gates, J. D.: The role of secondary carbide precipitation on the fracture toughness of a reduced carbon white iron, Materials Science and Engineering. 2008, vol. A490, pp. 313-318.

[17] Liu, Z., Chen, X., Li, Y., Hu, K.: High Boron Iron-Based Alloy and Its Modification, Journal of Iron and Steel Research, International. 2009, vol. 16, no. 3, pp. 37-42.
[18] Gou, Ch., Kelly, P. M.: Boron solubility in Fe-Cr-B cast irons, Materials Science and Engineering. 2003, vol. A352, pp. 40-45.
[19] Ma, S., Xing, J., Liu, G., Yi, D., Fu, H., Zhang, J., Li, Y., Effect of chromium concentration on microstructure and properties of Fe–3.5B alloy, Materials Science and Engineering. 2010, vol. A527, pp. 6800-6808.
[20] http://www3.ipc.org.es/guia_colocacion/info_tec_colocacion/los_materiales/baldosas/caract_fis_qui/