DEVELOPMENT AND CHARACTERISATION OF TITANIUM- AND VANADIUM-CARBIDE REINFORCED IRON BASE COMPOSITE

XU HONG, FU SIJING, WANG JING and DING YICHAO

Mechanical Engineering Department
Chengdu Electromechanical College
Chengdu 610031
P. R. China

Abstract

Titanium- and vanadium-carbide reinforced iron base composite was produced by a novel process which combines in situ synthesis with powder metallurgy technique using Ti, Fe, FeV and carbon powder. The microstructure of the titanium- and vanadium-carbide reinforced iron base composite was studied by scanning electron microscopy (SEM) and X-ray diffraction (XRD); with the help of differential thermal analysis (DTA), the reaction mechanism of the Fe-Ti-V-C system was discussed. The results show that the production of an iron matrix composite reinforced by titanium- and vanadium-carbide particulates using the novel process is feasible. Titanium- and vanadium-carbide particles exhibit homogeneous distribution in the pearlite. The reaction mechanism is as follows: first, allotropic change $Fe_\alpha \rightarrow Fe_\gamma$ and the reaction $FeV + C \rightarrow Fe + VC$ at 765.7°C; second, formation of the compound Fe_2Ti at 1058.5°C because of the eutectic reaction between Ti and Fe; third, reaction between carbon and melted Fe_2Ti causing formation of TiC at 1140.4°C; finally, formation of $(Ti, V)C$ due to the solubility between TiC and VC.

1. Introduction

Composite materials with steel matrix and ceramic particle reinforcements provide scope for producing relatively inexpensive wear-resistant materials. Most of the work on iron-based composites has involved titanium-carbide reinforcement, which is introduced in the iron matrix through a powder metallurgy (P/M) route [4]. The typical advantages of the route are raw material savings and low energy costs. In addition, P/M technique allows a higher content of alloying elements and the addition of the ceramic particles [6]. However, the materials produced via this technique generally suffer from the problem of contaminated matrix-reinforcement interfaces. From this view point, the techniques involving the *in situ* generation of the reinforcing phase have emerged as a preferred synthesis route for these materials. *In situ* techniques involve a chemical reaction resulting in the formation of a very fine and thermodynamically stable ceramic phase within a metal matrix. As a result, the reinforcement surfaces are likely to be free from gas absorption, oxidation or other detrimental surface reaction contamination, and the interface between the matrix and the reinforcement bond therefore tends to be stronger [13]. Some of these technologies include exothermic dispersion (XD), liquid-solid or liquid-liquid reactions, and self-propagation high-temperature synthesis (SHS). Among the several techniques available to synthesize metal-matrix composites, SHS and casting technologies are widely used to produce titanium-carbide reinforced iron base composites. However, manufacturing of titanium-carbide reinforced iron base composites using SHS will encounter large difficulties handling the intrinsic porosity during the reaction [8]. Whereas titanium-carbide reinforced iron base composites produced by casting will confront two difficulties: on one hand, the distribution of titanium-carbide in the iron matrix is likely to be uneven because of the density difference between iron and titanium-carbide; on the other hand, the volume fraction of titanium-carbide is limited because of reduction of the steel liquid fluidity at high titanium-carbide levels.

In addition, vanadium-carbide particles are expected to be the reinforcement for steel matrix composites because of their high modulus

of elasticity, high hardness, good wettability, low density and their relative stability with steel matrix. Over the past several years, Wang et al. [10, 11] produced Fe-VC surface composite to improve the service life of components. Ding et al. [5] produced V_8C_7 reinforced iron base composite to improve the wear-resistance of components. However, works concerning vanadium-carbide as an alternative addition to titanium-carbide reinforced iron base composites are scare.

In the present work, we highlight the fabrication of high dense titanium- and vanadium-carbide reinforced iron base composite by the new method, and this article gives particular attention to the reaction principle and the microstructure of the final product.
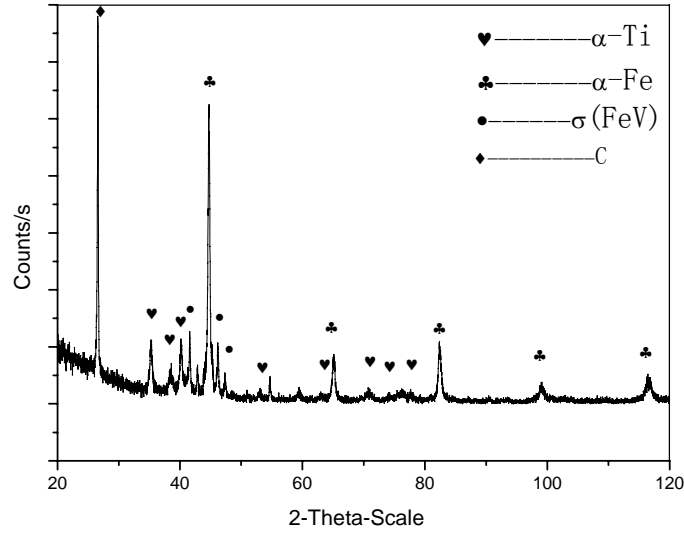
2. Experimental Procedure

The starting powders were Ti, Fe, Ferrovandium (Fe-50wt%V) and carbon black. The powders were mixed in a composition of 19.5wt.% Ti, 17wt.% FeV, 56.5wt.% Fe and 7wt.% C. Powders mixing was done by planetary ball (QM-1SP, China) milling at $180 \, \text{rev} \, \text{min}^{-1}$ for 24 h. The jars for powders mixing were made of stainless steel. The balls for milling, which were made of stainless steel, were weighed, to achieve a ball to powder ratio of 6:1. After mixing, the powder was characterised by X-ray diffraction (XRD) using Cu-Kα radiation. Samples of 16 mm diameter $\times 10$ mm height were manufactured by uniaxial die pressing at 350 MPa. Sintering was performed in 1400°C in a vacuum furnace for 1 h followed by furnace cooling.

Microstructure was examined by a JSM-5900LV scanning electron microscopy (SEM). Phase identifications in room temperature and high temperature were carried out on a Philips X-ray diffractometer and a D/max 2400 X-ray diffractometer, respectively. In addition, when phase identifications in high temperature were done, the mixing powder was put on a Pt foil. Differential thermal analysis (DTA) of the specimen was performed using NETZSCH STA 449C equipment. The sample was heated at 10°C/min up to a maximum temperature of 1300°C and cooled at the same rate to room temperature. The entire test process was conducted in high-purity argon gas using a flow rate of 150 ml/min.
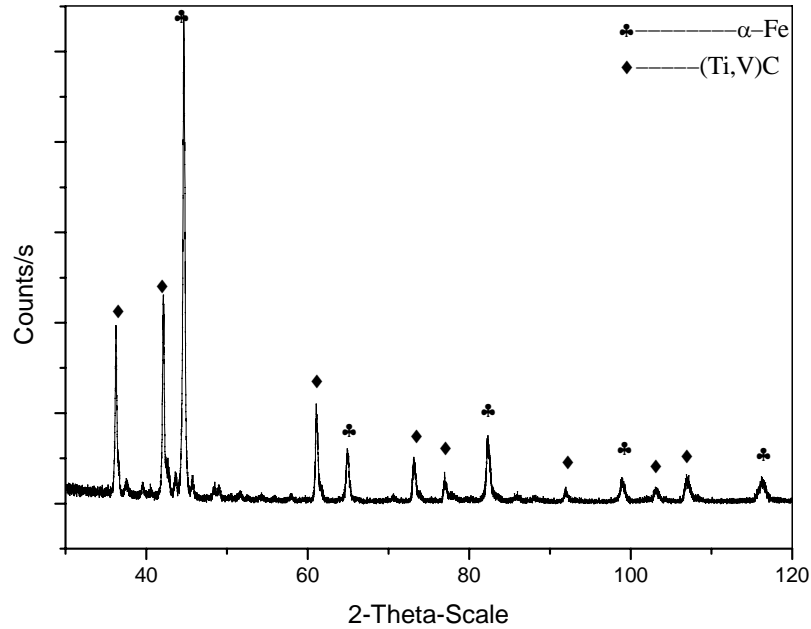
3. Results and Discussion

3.1. Phase identification

Figure 1 shows the characteristic XRD pattern of initial powder mixture. It has been detected the presence of α-Fe, α-Ti, σ (FeV) and C (graphite).

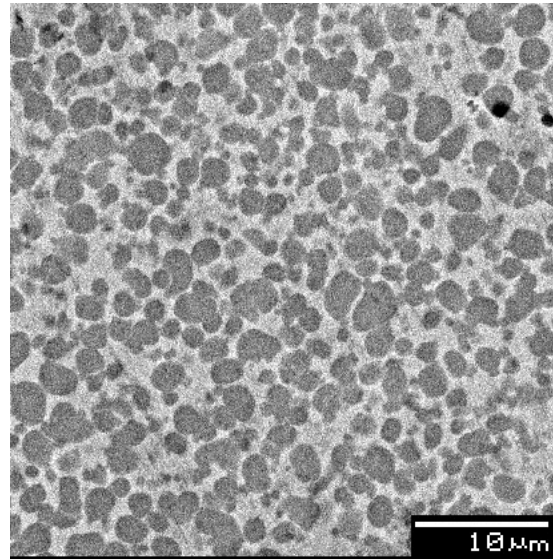**Figure 1.** X-ray diffraction pattern of initial powder mixture.

The diffraction pattern for the titanium- and vanadium-carbide reinforced iron base composite sintered at 1400°C is shown in Figure 2. The bcc-Fe binder was unchanged with respect to the original powder, while the formation of (Ti, V)C solid solution is seen in the composite.
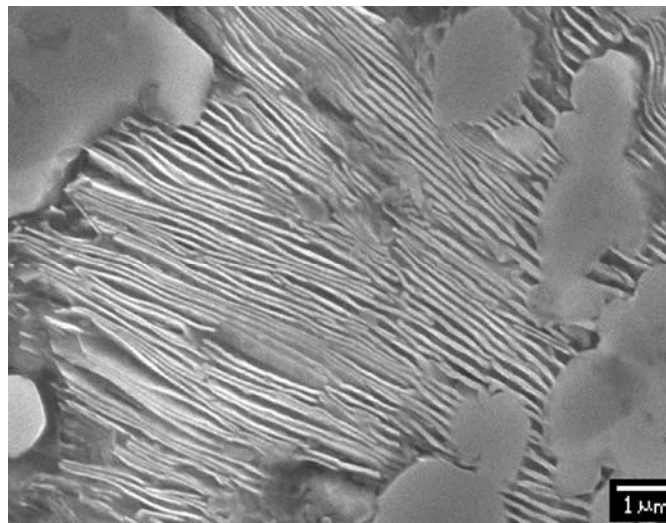
Figure 2. X-ray diffraction patterns of titanium- and vanadium-carbide reinforced iron base composite sintered at 1400°C.

3.2. Microstructure of iron base composite

Figure 3 shows the scanning electron micrograph of titanium- and vanadium-carbide reinforced iron base composite. The gray areas are (Ti, V)C particles and the lighter region is α-Fe matrix, while the dark areas are voids. It can be seen that (Ti, V)C particles are uniformly dispersed in the matrix. Figure 4 shows the scanning electron micrograph of titanium- and vanadium-carbide reinforced iron base composite etched by 4% nitric acid and alcohol solution. It can be seen that the matrix microstructure of composite is pearlite.
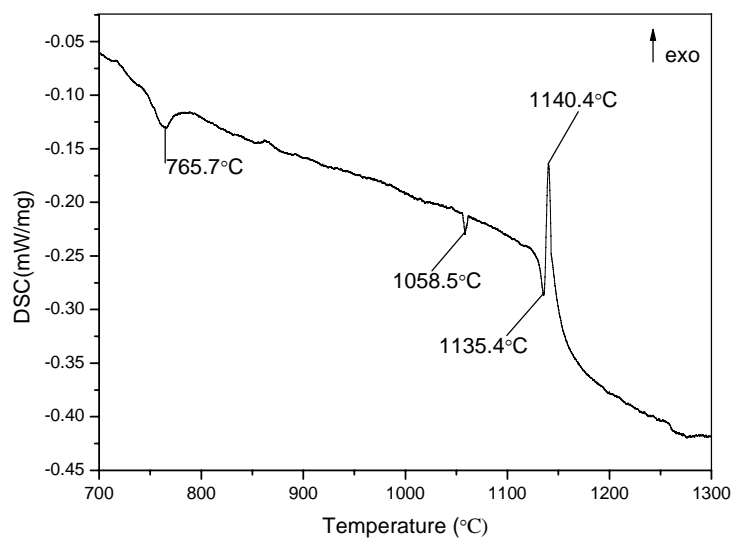
Figure 3. Microstructure of titanium- and vanadium-carbide reinforced
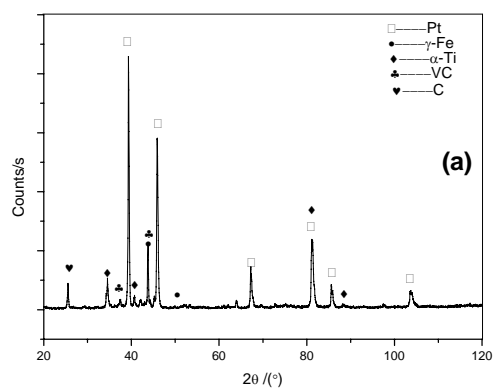iron base composite sintered at 1400°C.

Figure 4. SEM micrograph of titanium- and vanadium-carbide reinforced
iron base composite etched by 4% nitric acid and alcohol solution.
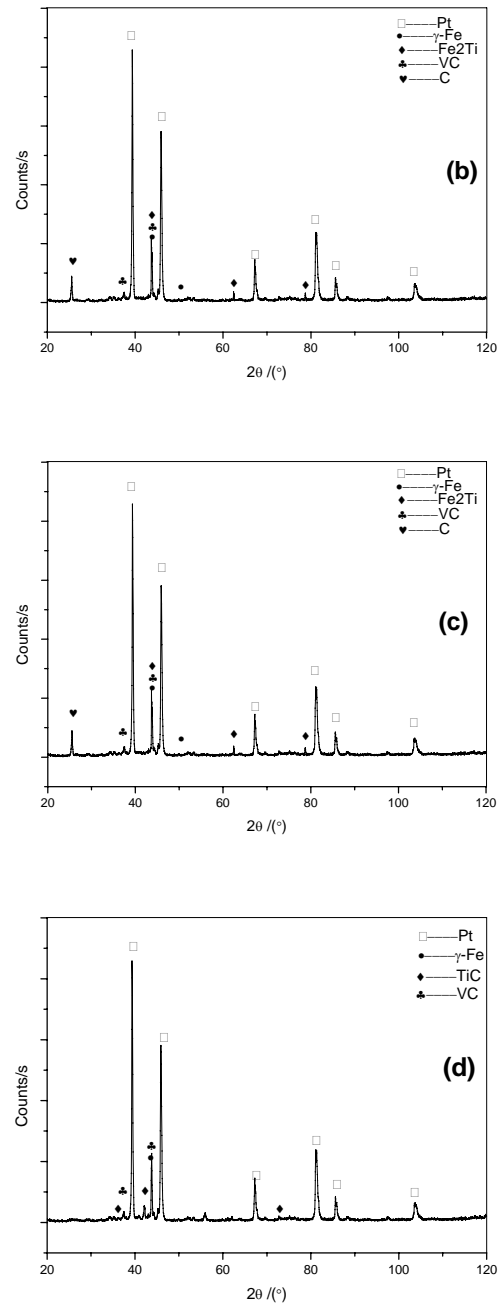
3.3. Reaction mechanism

Figures 5 and 6 show the DTA curve and XRD patterns of Fe-Ti-V-C system at different temperatures. The DTA curve indicates only one exothermic peak with the maximum at 1140.4°C. Furthermore, there are three endothermic peaks with minima at 765.7°C, 1058.5°C and 1135.4°C, respectively. According to XRD result in Figure 6(a), it can be concluded that the first endothermic peak with a minimum at 765.7°C arises from the reaction $FeV + C \rightarrow VC + Fe$ and allotropic change $Fe_\alpha \rightarrow Fe_\gamma$, Aigner et al. [1] think that the decrease of allotropic change $Fe_\alpha \rightarrow Fe_\gamma$ from 912°C (value for pure iron) is mainly caused by carbon diffusion in the iron. The allotropic change $Fe_\alpha \rightarrow Fe_\gamma$ is endothermic, and the formation heat is 919.6 KJ/mol [3]; whereas the reaction $(FeV + C \rightarrow VC + Fe)$ is exothermic, and the formation heat is 207.45 J/mol [12]. Therefore, the first endothermic peak at 765.7°C in Figure 5 was observed. Based on the Fe-Ti phase diagram, the second endothermic peak (minimum at 1058.5°C) is thought to arise from the eutectic reaction between Fe and Ti, this result is in agreement with that reported by Berns and Wewers [2]. The exothermic peak with a maximum at 1140.4°C is attributed to exothermic reaction between melted Fe_2Ti and carbon. These can be confirmed by XRD analysis (see Figure 6(b) and Figure 6(d)). However, XRD analysis also reveals that the Fe-Ti-V-C system at 1135.4°C (see Figure 6(c)) consists of the similar phases compared with that at 1058.5°C. As a result, it is difficult to deduce a reaction that occurred in the Fe-Ti-V-C system at 1135.4°C. Further work is continuing in this area.

Figure 5. Differential thermal analysis thermogram for the Fe-Ti-V-C system.

Figure 6. X-ray diffraction patterns of Fe-Ti-V-C system.
(a)770°C; (b) 1060°C; (c) 1137°C; (d) 1142°C.

Hamar-Thibault et al. [7] think that in ternary V-Ti-C system, TiC-VC mixtures form a continuous solid solution (Ti, V)C at high temperature, as shown in isothermal sections and quasi-binary sections reported by Holleck, and at low temperature, the solid solution (Ti, V)C is stable and no decomposition is observed. In fact the TiC-VC system has a complete solid solution because the carbides share a cubic NaCl type structure and the atomic radius of each metal is not that different, fulfilling the Hume-Rothery condition. The metals can be replaced or moved without jeopardizing the stability of the structure, so their distribution inside the solid solution will locate at random [9].

4. Conclusions

Using a novel process which combines *in situ* reaction with powder metallurgy techniques, an iron base composite, reinforced by titanium- and vanadium-carbide reinforced iron base composite was produced. The (Ti, V)C particles generated *in situ* are uniformly dispersed in the matrix. The reaction mechanism is as follows: first, allotropic change $Fe_\alpha \rightarrow Fe_\gamma$ and the reaction $FeV + C \rightarrow VC + Fe$ at 765.7°C; second, formation of the compound Fe_2Ti at 1058.5°C because of the eutectic reaction between Ti and Fe; third, reaction between carbon and melted Fe_2Ti causing formation of TiC at 1140.4°C; finally, formation of (Ti, V)C due to the solubility between TiC and VC.

References

[1]   K. Aigner, W. Lengauer and P. Ettmayer, J. Alloys Compd. 262-263 (1997), 488.

[2]   Hans Berns and Birgit Wewers, Wear 251 (2001), 1386.

[3]   Ye Dalun, Thermodynamics Handbook of Inorganic Substances (in Chinese), Metallurgy Industry Press, Beijing, 1981.

[4]   K. Das and T. K. Bandyopadhyay, Mater. Lett. 58 (2004), 1877.

[5]   Y. C. Ding, Y. S. Wang, J. Wang and Y. Xian, J. Sichuan. Univ. 39 (2007), 113 (in Chinese).

[6]   E. Gordo, F. Velasco and N. Antón, Wear 239 (2000), 251.

[7]   Sylvaine Hamar-Thibault, Leila Adnane and Rafika Kesri, J. Alloys Compd. 317-318 (2001), 311.

[8]   Patrik Persson, Anders E. W. Jarfors and Steven Savage, J. Mater. Process. Tech. 127 (2002), 139.

[9]   R. Sepúlveda and F. Arenas, Int. J. Refract. Met. H. 19 (2001), 389.

[10]   Y. S. Wang, Z. P. Sun, Y. C. Ding and F. C. Li, Mater. Design 25 (2004), 69.

[11]   Y. S. Wang, Y. C. Ding and J. Wang, Mater. Design 28 (2007), 2202.

[12]   Yisan Wang, Zhiping Sun, Yichao Ding and Fengchun Li, Mater. Des. 25 (2004), 69.

[13]   X. N. Zhang and W. J. Lü, Scr. Mater. 41 (1999), 39.

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