The Effect of TiO₂ on Synthesizing Fe-TiC Composites

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Abstract

The ratio of ilmenite (FeTiO₃) to titanium dioxide (TiO₂) was varied to synthesize Fe-TiC composites by carbothermal reduction of ilmenite ore under gas atmosphere at the temperature 1500°C. Ratio of Fe to TiC showed that it can be controlled through adjustment of the reactant materials. The standard Gibbs energy minimization method was used to calculate the equilibrium composition of the reaction species. The products were characterized using SEM and XRD.

1. Introduction

Ceramic-metal composites or cermets are widely used for the manufacture of high performance wear parts and cutting tools. Titanium carbide (TiC) is one of the most suitable reinforcement in iron matrix composite due to its high hardness and high chemical stability and also it can be wet by iron matrix [1].

The most common used process for titanium carbide production is direct reaction of Ti with graphite under vacuum at high temperature range of 1900°C - 2700°C [2]. The producing of Fe-TiC composite by direct mixing of Fe and TiC powder is relatively expensive. Also, there are some difficulties to disperse reinforced particles of TiC evenly in the matrix Fe. From these reasons many synthesis routes to produce Fe-TiC were studied and proposed such as carbothermal reduction [3], thermal plasma synthesis [4], casting [5], self-propagating high temperature synthesis (SHS) [6]. The thermal plasma synthesis has vary high operating cost; on the other hand casting, SHS process required expensive precursor. The carbothermal reduction of ilmenite ore with activated carbon is considered as less expensive to produce *in-situ* Fe-TiC.

In this study, thermodynamics model for carbothermic reduction of ilmenite was developed to compare the experimental results of the synthesis Fe-TiC composites. The result composition of final product affected from varying amount of adding TiO₂ were investigated.

2. Theoretical Background

Thermodynamic equilibrium calculations were performed using a computer program base on Gibbs energy minimization method [7] to determine the most stable compositions of the materials as a function of temperature. This method is based on the fact that systems will achieve equilibrium at the lowest possible energy level. Hence, the total Gibbs energy for a system is as a minimum at equilibrium. The equilibrium
composition and distribution for the system FeTiO$_3$-C was calculated elsewhere [3].

The overall chemical reaction for FeTiO$_3$-nTiO$_2$-C system and can be expressed as:

$$\text{FeTiO}_3(s) + n\text{TiO}_2(s) + (4+3n)C(s) \rightarrow \text{Fe}(s) + (1+n)\text{TiC}(s) + (3+2n)\text{CO(g)}$$ (1)

Where $n =$ number of added TiO$_2$ moles

The equilibrium compositions of FeTiO$_3$-2TiO$_2$-C systems at different temperatures were calculated using Gibbs energy minimization method and the results are shown in Figure 1.

![Fig. 1 Gibbs energy minimization plots of FeTiO$_3$-TiO$_2$-C system in an argon atmosphere.](image)

It can be seen from Figure 1 that thermodynamically, it is feasible to fabricate iron matrix composite reinforced with TiC using carbothermal reduction process. When temperature increases, ilmenite decomposes to FeO and TiO$_2$. FeO is reduced by carbon to Fe and remain as iron phase throughout the increasing temperature to 2000°C. At the temperature about 1300°C, TiO$_2$ is reduced by carbon and produces TiC. The reaction will continue as the temperature increases until complete reaction at about 1500°C. Under Ar gas atmosphere that prevent reverses reaction, the result phases at 1500°C can maintain as iron-titanium carbide composite particles until it cool down to room temperature.

3. Experimental

The raw materials used were powder of ilmenite, TiO$_2$ and activated carbon. Each reactant was carefully weighted according to stoichiometric ratio given by equation (1) at $n$ equal to 0, 1, and 2. All precursors were mixed by milling in planetary ball mill for 2 hr. at 250 rpm to ensure the intimate contact of reactants. After milling, samples were heated at 10°C/min to 1500°C in alumina crucible under flowing argon gas (3 mL/min) atmosphere. Soaking at final temperature for 1 hr., then furnace was turned off, allowing the products to cool in the furnace.

The products powder was analyzed using XRD (PHILIPS with Cu K$\alpha$ radiation) and SEM (JSM-5800 LV, JEOL).

4. Results and Discussion

All the experiments with varied mole ratio of ilmenite to TiO$_2$ had completed carbothermal reduction and produced Fe-TiC composites powder shown in Fig 2. The quantitative XRD [8] were used to calculate intensity ratio (%) of each sample and were plot in Fig 3. It shows that the intensity ratio (%) of TiC increase and Fe decrease linearly when added TiO$_2$ into the precursors which match well with the thermodynamic calculations.

The morphology of product mainly are composites powder particles was shown in Fig 4, which is agglomerated of TiC reinforced particles and Fe matrix.

![Fig.2 XRD pattern of products from Varying amount of added TiO$_2$.](image)
5. Conclusions

Iron-titanium carbide composites were produced in-situ from ilmenite powder and activated carbon powder by carbothermal reduction process at 1500°C under Ar gas atmosphere. Experimental results showed that an iron matrix composite with reinforcement phase of TiC was formed. Adding TiO₂ to precursors would produce more TiC reinforced particles in Fe matrix based compared to adding TiO₂ powder. The mole ratio of TiC in products can be increased with adding TiO₂ and mole ratio Fe would be decreased with adding TiO₂.

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7. References