Journal of Alloys and Compounds 628 (2015) 113–120

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jalcom

Investigation on the reactions sequence between synthesized ilmenite and aluminum

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ARTICLE INFO

Article history:
Received 22 May 2013
Received in revised form 4 December 2014
Accepted 27 December 2014
Available online 31 December 2014

Keywords:
Ilmenite
Aluminum
Fe–Al_2O_3/TiC composites
Phase transitions

ABSTRACT

Fe–Al_2O_3/TiC composites have features of which we can refer to: good strength, toughness, wear resistance, high temperature strength and light weight. It can also be used as cutting tools. Some researchers have used ilmenite, aluminum and carbon to produce these composites. However the formation mechanism and reaction sequences are not clear enough. In this research, the first step of reactions was investigated by studying the binary phase between aluminum and ilmenite. At first, ilmenite was synthesized, and then the critical temperatures due to the reactions occurring in the ilmenite–aluminum powder mixture were determined by DTA. The milled and pressed samples of synthesized ilmenite and aluminum with molar ratio of 1:2 were heat treated at 740 °C for 3, 12, 15, 24 and 72 h and at 1000 °C for 24 h. The final products were analyzed with XRD, SEM and EDS. The results show that after the melting of aluminum, the reaction between ilmenite and molten aluminum starts, leading to formation of Fe and TiO_2. It is found that the tendency of FeO to reduce with aluminum is more than TiO_2. Therefore, FeO reduces to Fe while the TiO_2 remains unchanged. By consumption of the raw materials, Fe and TiO_2 react with remained aluminum, resulting in the formation of spherical Fe_2Ti in the TiO_2 and Al_2O_3 matrix.

1. Introduction

Strength enhancement, toughness, wear resistance, high temperature strength, and decrease in weight are the benefits of using ceramics as reinforcements in metals. Common ceramic materials used for the enforcement of Fe matrixes include: TiC, Al_2O_3, ZrO_2, TiN_2, Si_3N_4, B_4C, VC, etc. [1]. On the other hand, Al_2O_3–TiC composites are used as cutting tools and adding a metal such as Fe can help improve the stiffness [2,3]. These composites can be produced by various methods [1], among which, the combustion synthesis method requires the least energy [4]. Several research [5–8] have been performed using ilmenite as a raw material, for the production of Fe–Al_2O_3/TiC composites by combustion synthesis method, due to the higher price of titanium, compared to ilmenite. Eq. (1) shows the main reaction leading to the formation of Al_2O_3–TiC/Fe composite from ilmenite, aluminum and graphite.

$$FeTiO_3 + 2Al + C = Fe + TiC + Al_2O_3 \quad (1)$$

There are few researches available to clarify the mechanism of the above reaction [5–9]. Although, in these few researches [5–9], it was showed that, in the $FeTiO_3$–Al–C system, even where carbon is available, the reduction reactions start with the reaction of aluminum and ilmenite [5–7,9]. In other words, the first step of reactions in the ilmenite, aluminum and graphite system is the reaction of ilmenite and molten aluminum. However, there is not any agreement on the product phases, their formation sequence and the mechanism. Therefore, in this research a simplified binary system of $FeTiO_3$–Al was investigated in order to determine the first step reactions mechanism. Thereupon, the molar ratio of the ilmenite and aluminum was selected according to the raw materials ratios in Eq. (1).

A variety of ilmenite to aluminum ratio is used by different researchers, as is explained in the following: In some of the researches, carbon is used according to the following equations:

$$FeTiO_3 + 2Al + C = Fe + TiC + Al_2O_3 \quad \text{(Willis [9])} \quad (2)$$

$$3FeTiO_3 + 7Al + 3C = 3Al_2O_3 + 3TiC + Fe_3Al \quad \text{(Zou [7])} \quad (3)$$

In other research, a binary system of ilmenite and aluminum has been investigated according to the following equations:

$$FeTiO_3 + (8 + x)Al \rightarrow Al_2O_3 + Al_3Fe + Al_3Ti + xAl \quad \text{(Sleziona [10])} \quad (4)$$

$$FeTiO_3 + 2Al = Al_2O_3 + Fe + Ti \quad \text{(Sleziona [11])} \quad (5)$$

$$FeTiO_3 + 7Al \Rightarrow FeAl_2 + TiAl_3 + Al_2O_3 \quad \text{(Welham [12])} \quad (6)$$

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http://dx.doi.org/10.1016/j.jallcom.2014.12.206
0925-8388/

It can be seen that at different ilmenite to aluminum ratios, different products are obtained. It does not have expressed comprehensive and detailed mechanisms in the researches. Therefore, in this research, to determine the first step reaction mechanism of Eq. (1), an ilmenite to aluminum ratio of 1:2 was used.

Furthermore, in this work, according to the previously published methods [13–15], synthesized ilmenite was prepared and used instead of ilmenite concentrate. There are some impurities available in the ilmenite concentrate. The effect of these impurities on the reaction mechanism is not known. Therefore, it was necessary to synthesis the ilmenite. Production of synthesis ilmenite is the milestone of the experiments in this paper.

Then ilmenite and aluminum were mixed according to the molar ratio of Eq. (1) ($FeTiO_3$: Al = 1:2) to be examined the possible interactions of ilmenite and aluminum, as the first step reactions of the ilmenite, aluminum and graphite system.

2. Experimental

The synthesized ilmenite was prepared based on the following reaction:

$$Fe + Fe_2O_3 + 3TiO_2 = 3FeTiO_3 \quad (7)$$

3.78 g of iron powder (99.5%, <10 μm, from Merck company), 5.41 g TiO_2 (98%, <45 μm, prepared from Crimea Titan PJSC) and 10.81 g Fe_2O_3 (96.5%, <64 μm, from Crimea Titan PJSC company) were mixed for 10 min in a fast mill at 400 rpm with an alumina jar, including 16 alumina balls of 2 cm diameter. The ball to powder ratio (BPR) was (5:1). After the milling procedure, the mixed powder was pressed in a mold to obtain a pellet with a diameter of 1 cm under 3.4×10^6 Pa pressure to obtain an appropriate contact between the powders. In the next step, the samples were heat treated for 48 h at 1100 °C.

To prevent the oxidation of the products, heat treatment was done under argon atmosphere. The argon gas was passed through heated pure Cu at a temperature of 550 °C to eliminate any O_2 present in the argon gas. Ascarite (sodium hydroxide coated silica) and Drierite (anhydrous calcium sulfate) have also been used to eliminate the CO_2 and H_2O presented in the argon gas as impurities, respectively.

A quartz tube was used in a furnace tube to prevent the samples from oxidation. If quartz tube was suddenly removed from the furnace (at 1100 °C), it would have been cracked or fractured. Therefore, to remove the sample, it was cooled to 800 °C by reducing the furnace temperature and then was removed from the furnace.

The prepared ilmenite powder was sieved (<74 μm) and mixed with at aluminum powder (99.5%, <45 μm) to a molar ratio of 1:2. The powder mixture was milled for 10 min using a BPR of 5:1 at 400 rpm. After the milling procedure, the mixed powder was pressed in a mold under 3.4×10^6 Pa to obtain pellets of 1 cm diameter and 5 mm height. The pellets were finally heat treated at 740 °C between 1 to 72 h and at 1000 °C for 24 h.

To determine the critical temperatures of the system, DTA (NETZSCH STA 409 PC/PG) was used. XRD technique was used for the phase analysis of the product using a PHILIPS model PW 1800 machine with Cu Kα radiation ($\lambda = 1 \cdot 54$ Å) under

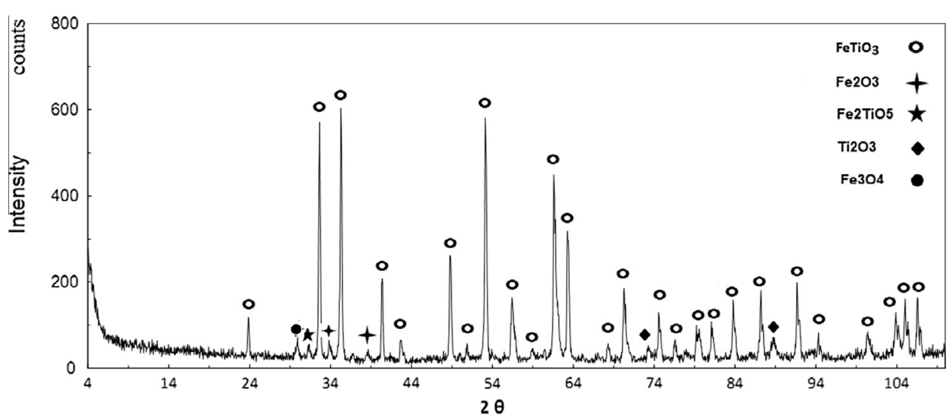

Fig. 1. The XRD pattern of the prepared synthesized ilmenite.

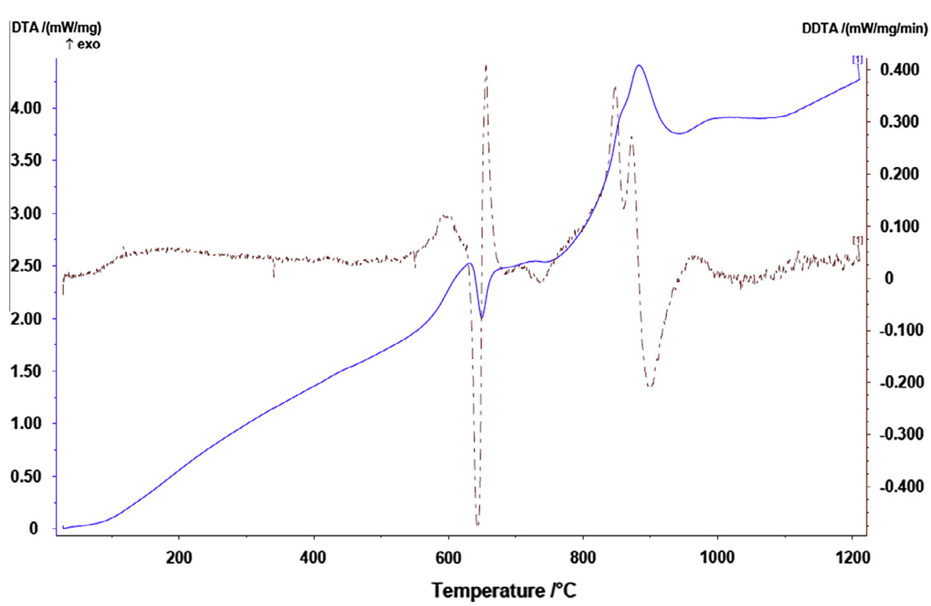

Fig. 2. DTA result for the powder mixture of ilmenite and aluminum ($FeTiO_3$: Al, 1:2).