



# Controlling conditions for synthesis of iron–titanium carbide using two different titanium precursors



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## ABSTRACT

Preparation of Fe–TiC composite from mixtures of carbon black and two different titanium bearing minerals (black sand ilmenite and natural rutile) was studied. Milled (mechanically activated) and unmilled carbon containing mixtures were prepared and then heated at temperatures 1200 °C and 1300 °C for 3 h under an inert atmosphere. The reaction progress, as well as reaction products, was evaluated using thermogravimetric analysis (TGA-DTA), X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), and energy dispersive X-ray spectroscopy (EDX). The feasibility of producing Fe–TiC composites from titanium bearing materials mixed with carbon black was proved. Fe–TiC could be produced by carbothermic reduction of mechanically activated black sand ilmenite containing mixtures milled for 50 h and heated up to 1200 °C. On the other hand, 60 h milling followed by heating up to 1300 °C was needed in case of natural rutile containing mixture. The morphology of the Fe–TiC produced from black sand ilmenite showed a homogeneous distribution of Fe and TiC enriched areas, while the Fe–TiC produced from natural rutile showed intense distribution of TiC phase with traces of iron and lower titanium oxide.

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## 1. Introduction

Recently, particle reinforced iron matrix composites have been a focus within the scope of new materials. Due to the fine wettability with liquid iron, TiC particles with high hardness and chemical stability are widely used as a reinforcing phase for iron matrix composites [1–2].

TiC can be synthesized by direct reaction between Ti and carbon under vacuum at high temperature of 1900 °C–2900 °C [3], thermal plasma synthesis [4], chemical vapor deposition (CVD) [5], and self-propagating high temperature synthesis (SHS) [6–8]. A major technology for TiC is based on reduction of rutile (TiO<sub>2</sub>) by carbon black in a temperature range from 1700 to 2100 °C [9–12]. Reduction of TiO<sub>2</sub> by carbon is highly endothermic and proceeds under standard conditions at 1289 °C [13]. However, the actual titanium carbide production needs much higher temperatures because of kinetic barriers. Many studies have indicated that the reactivity of reactants in chemical reactions can be increased by mechanical activation through high-energy ball milling due to decreasing crystallite size within individual particles and intermixing of phases, which increases the interfacial surface area [14–15]. These enhancements lead to reactions occurring at lower

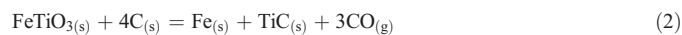
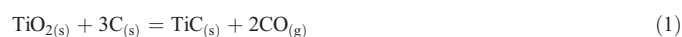
temperature and/or at greater rates than for simple physical mixtures [16–17].

The present paper describes a one-step process to produce Fe–TiC composites directly from different titanium bearing materials and carbon black via mechanical activation followed by reduction reactions under argon atmosphere at relatively lower temperature. The effects of mechanical activation time and reduction temperature on the formation of iron–titanium carbide were also studied.

## 2. Experimental

The starting materials were black sand ilmenite and natural rutile with mean particles size (*BT-2001 (Liquid) laser particle size analyzer*) of 128 and 133 μm, respectively. Typical chemical composition analysis of these materials (weight percent of dry material) was carried out by X-ray fluorescence (XRF) analysis, and given in Table 1. High purity carbon black powder (6 μm) of more than 99% was used as the reductant.

Each material was first mixed with an appropriate amount of carbon black (C/O molar ratio was kept at 1.5) to provide sufficient carbon for full reduction of oxides of titanium and iron present in the raw materials according to the following reactions:



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**Table 1**  
Chemical analysis of used ores.

Raw materials (wt.%)	TiO <sub>2</sub>	FeO	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	MnO	V <sub>2</sub> O <sub>5</sub>	MgO	CaO
Black sand ilmenite	43.1	38.79	14.76	0.26	1.15	–	0.5	2.42	–
Natural rutile	86.65	–	8.57	0.59	2.4	0.14	0.38	0.48	0.16

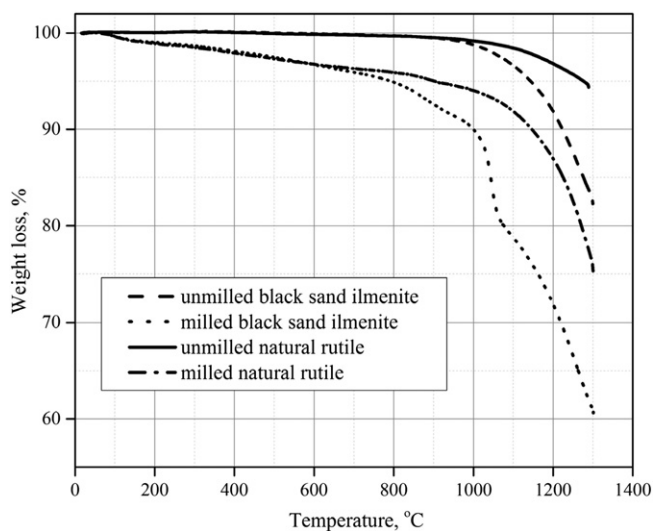


These mixtures were milled in a vertical planetary ball mill with an agate cell containing silicon nitride balls of 20 mm diameter. A weight ratio of ball to powder (BPR) of 10:1 and rotational speed of 180 rpm were maintained throughout the study. The powder mixtures were milled for various milling duration (30–60 h) with intermittent sampling. A portion of the milled samples was reduced at 1200 and 1300 °C for 3 h under an argon atmosphere. The reduction products were analyzed by XRD (XRD, Bruker axs D8 Advance, Germany with Cu K<sub>α</sub> radiation = 1.5406 Å) and its morphology was investigated by field emission scanning electron microscope (FESEM, Quanta, FEG 250).

### 3. Results and discussion

#### 3.1. Thermogravimetric analysis

The carbothermic reduction process is often monitored by thermogravimetric analysis (TGA, Netzsch STA 409 C/CD); variation of weight loss of samples during heating is an indication of reaction kinetics. Fig. 1 shows thermogravimetric curves of thermally treated milled and unmilled mixtures of both black sand ilmenite as well as rutile containing mixtures. The samples were heated 10 °C/min from room temperature up to 1300 °C under a helium atmosphere. The results show that the actual weight loss for unmilled samples starts at 800 °C reaching 1.22 and 0.82 wt.% decrease during heating up to 1000 °C for black sand and rutile mixtures respectively; this ineffective weight loss may be due to moisture liberation. A significant weight loss was observed only at relatively higher temperatures between 1000 °C and 1300 °C, irrespective of the sample composition, up to 17.36 wt.% for ilmenite, and



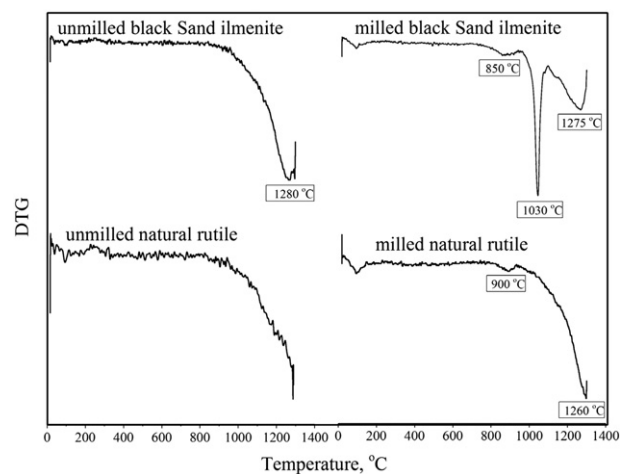
**Fig. 1.** Thermogravimetric curves of two different mixtures: black sand ilmenite-carbon and natural rutile-carbon on helium atmosphere up to 1300 °C before and after milling.

only 5.62 wt.% for rutile. This weight loss may be due to gasification of carbon at a high temperature range according to Eqs. (4) and (5):



Unlike the unmilled titanium bearing mixtures, the milled mixtures started to react quite early at 100 °C, which is indicated by the early mass loss. When the sample temperature reached 1000 °C, 10% mass loss was observed for black sand ilmenite and 5.97% mass loss for the natural rutile containing mixtures. The increase in weight loss continued up to 1300 °C with maximum values of 39.51 and 24.98 wt.% respectively. This progress in weight loss due to mechanical activation effect led to decreasing the mean particle size from 128 μm to 7 μm for black sand ilmenite and 133 μm to 14 μm for natural rutile. This decrease in particle size promoted the chemical reaction due to intermixing of reactant phases and active surface area.

Fig. 2 shows the differential thermographs of the two mixtures before and after milling. The thermographs have similar shapes up to 1000 °C for two unmilled mixtures. On rising the temperature beyond 1000 °C, only one endothermic peak was observed in the black sand ilmenite mixture at 1280 °C, while the natural rutile mixture didn't show any noticeable thermal changes. On the other hand, after mechanical activation different thermal changes were observed. For the black sand ilmenite mixture, three endothermic peaks at 850 °C, 1030 °C and 1275 °C were observed, which may be due to the aforementioned solid and gaseous reduction reactions [18]. A sharp endothermic peak was observed at 1030 °C which may be due to metallization of iron. Also, natural rutile mixture shows two endothermic peaks at 900 °C and 1260 °C, which may be due to formation of unstable oxygen-deficient Ti<sub>n</sub>O<sub>2n-1</sub> (n > 3), phase with a lower oxidation state of Ti



**Fig. 2.** Differential thermal analysis curves of two different mixtures: black sand ilmenite-carbon and natural rutile-carbon before and after milling under helium atmosphere up to 1300 °C.

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