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Preparation of Fe–TiC Composites by the Thermal-Explosion Mode of Combustion Synthesis

A. Saidi,[a] A. Chrysanthou,[b] J. V. Wood[c] & J. L. F. Kellie[d]

[a]Department of Materials Engineering, Isfahan University of Technology, Isfahan, Iran
[b]Department of Materials Science and Engineering, University of Surrey, UK
[c]Department of Materials Engineering and Materials Design, University of Nottingham, Nottingham, UK
[d]London and Scandinavian Metallurgical Company Limited, Rotherham, UK

Abstract: Titanium carbide ferrous matrix composites were produced by means of the thermal explosion mode of combustion synthesis. The combusting behaviour of the powder mixtures was investigated. The effect of the processing variables on the lattice parameter and the composition of TiC was examined. The influence of the carbide composition on the wetting of TiC by the matrix and the subsequent microstructure are discussed. © 1997 Elsevier Science Limited and Techna S.r.l.

1 INTRODUCTION

High density carbide components can be produced by mixing TiC powder with a binder phase such as iron and nickel. Iron-based TiC metal matrix composites are currently produced by powder metallurgy techniques and are commercially available under a number of trade names such as 'ferrotic' and 'ferrotitanit'. Liquid-based routes are potential alternatives for the production of Fe–TiC composites. Self-sustaining High temperature Synthesis (SHS) also provides a unique method for the production of this composite. An even dispersion of particulate TiC in an iron matrix can be obtained by the SHS reaction between titanium, iron and carbon. The mechanism of this process has previously been demonstrated by the present authors.[1]

Titanium carbide has a cubic NaCl-type structure which has an extraordinarily wide composition range between $TiC_{0.49}$ and $TiC_{0.95}$.[2] It has been demonstrated that the properties of TiC depend strongly on its C/Ti ratio, which in turn is a function of the lattice parameter.[3–5] Most of the investigations concerning the effect of TiC composition on its mechanical properties have focused on C/Ti atomic ratios ranging from 0.8 to 1 and therefore very little information is available on the properties of TiC with carbon content less than 44 mol%. The wettability of TiC by liquid metals is considered as an important physicochemical property when titanium carbide is going to be used in the production of composites or cemented carbides. Ramqvist[6] has demonstrated that a change in C/Ti ratio from 1 to 0.49 can decrease the wetting angle between TiC_x and copper from 108° to zero (i.e. complete wetting is achieved by $TiC_{0.49}$). This observation has been related to the stability of the carbides such that the stronger the inter-atomic bonds in the carbide the lower the tendency to break and interact with the liquid metal. This relationship has been studied by Morozova[7] who suggested that the wettability of TiC_x with varying carbon contents follows a trend identical with that for the heat of formation. However, both studies used enthalpy of formation (ΔH°) values rather than Gibbs energy data (ΔG°) to compare thermodynamic stability. The relationship between wettability and carbide stoichiometry, for Cu–TiC_x, is illustrated in Fig. 1.

A knowledge of the parameters which control the stoichiometry of titanium carbide in Fe–TiC, and a method for quick estimation of that, are practically very useful. The objective of the present work was, therefore, to investigate the effect of the processing variables on the titanium carbide lattice parameter and the combined carbon in Fe-TiC produced by combustion synthesis.

2 EXPERIMENTAL PROCEDURE

The reagents used in this research were powders of titanium, iron, and carbon which were kindly supplied by the London and Scandinavian Metallurgical Company Limited. Titanium powder had a purity of 99% and an average particle size of 45 μm. Iron powder was 98% pure with a particle size between 100 and 250 μm. Carbon black with a mean particle size of 35 nm was used. Reactant powder mixtures with iron compositions ranging from 0 to 80 wt% and C/Ti atomic ratios ranging from 0.4 to 1 were prepared for the following basic reactions:

$$Ti + C + x(Fe) = x(Fe) + TiC \quad (1)$$

$$Ti + x(C) + 0.3Fe = 0.3Fe + TiC_x \quad (2)$$

Pellets 11.3 mm in diameter were compacted in a ceramic die, using a pelletising pressure of about 4 MPa. The pellets were heated to ignition in a graphite crucible, using an induction furnace. The furnace was evacuated to a level of 0.15 Pa and back filled with argon to atmospheric pressure. For each run a temperature profile was obtained using an Ircon-Modline two-colour pyrometer together with a data acquisition system capable of taking output voltage data at a rate of 500 readings per second. These facilities made it possible to measure and analyse the temperature change of the sample during the reaction which normally lasted for less than 2 s. Product identification was carried out by means of X-ray diffraction analysis, using Cu-K_α radiation. Lattice parameter of TiC was worked out using the Bragg law for the cubic system:

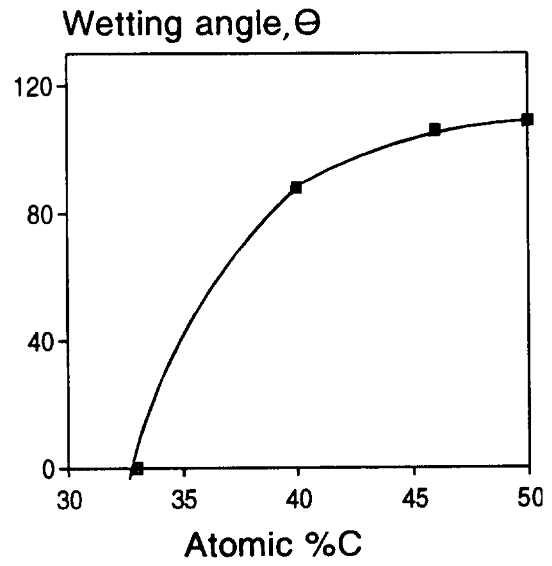

Fig. 1. Wettability of TiC by liquid Cu at 1100 °C vs carbon composition.[7]

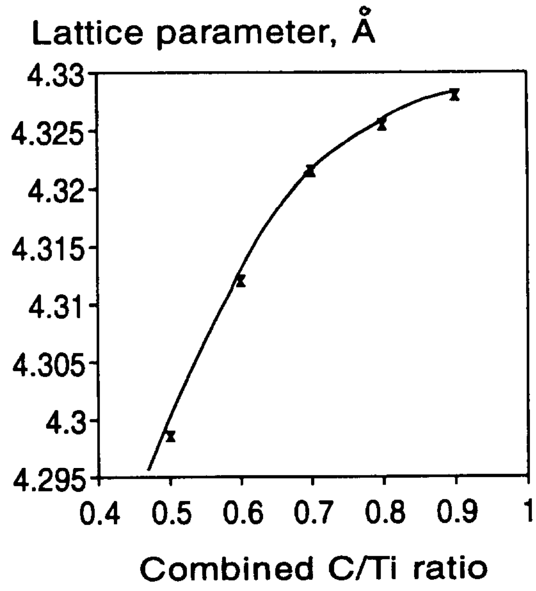

Fig. 3. The variation of the lattice parameter of TiC with composition.[9]

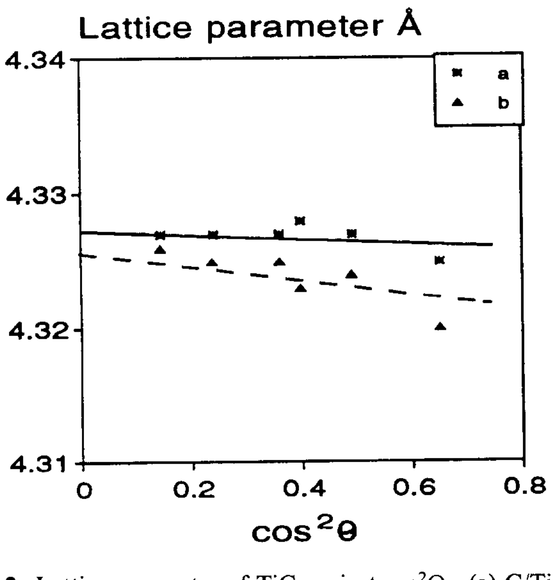

Fig. 2. Lattice parameter of TiC against $\cos^2\Theta$. (a) C/Ti = 1; (b) C/Ti = 0.6.

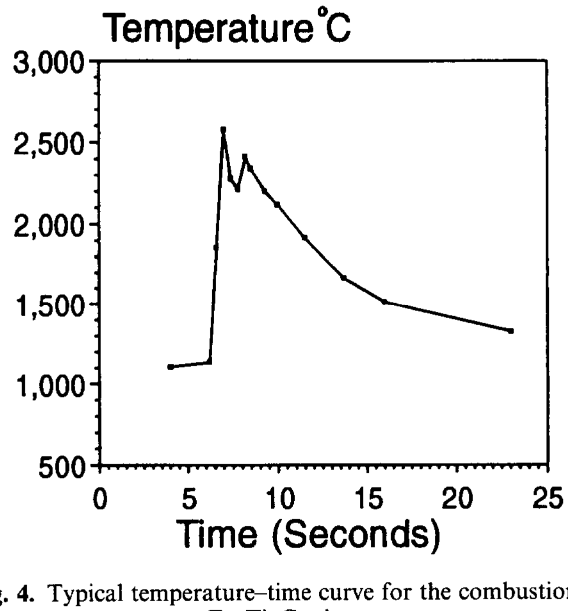

Fig. 4. Typical temperature–time curve for the combustion of a Fe–Ti–C mixture.

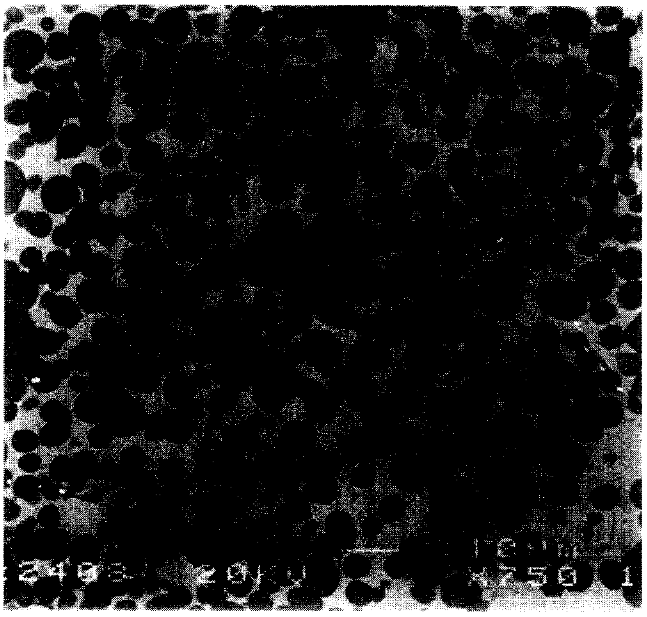

Fig. 5. Back scattered electron micrograph of Fe–TiC composite. Dark particles are TiC and white matrix is iron.

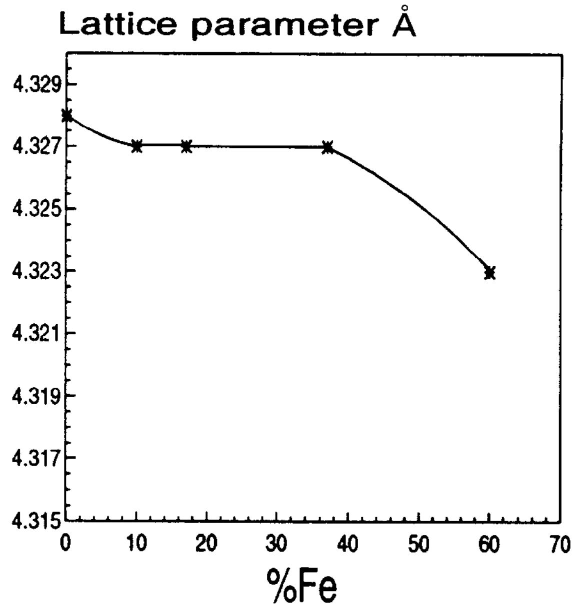

Fig. 6. Lattice parameter of TiC against iron composition.

$$a = d\sqrt{(h^2 + k^2 + l^2)} \quad (3)$$

For the precise parameter measurement, as described by Cullity,[8] lattice parameter, a, was plotted against $\cos^2\Theta$ and the extrapolation of the best line was used to work out the parameter when Θ approaches zero (Fig. 2). The composition of TiC was predicted using the relation between C/Ti ratio and the lattice parameter (Fig. 3).

3 RESULTS AND DISCUSSION

The reaction of titanium and carbon was self-sustaining for all the samples which contained up to 80 wt% iron. Reactions were fairly violent in the form of an explosion. A typical temperature–time curve is shown in Fig. 4. It is evident that after ignition, the temperature reaches its highest value within 1–2 s and the reaction is assumed to be complete within this period. Titanium carbide produced by this method was spherical and was evenly distributed in the iron matrix.

Figure 5 shows a typical microstructure of stoichiometric Fe–TiC composite containing 22 wt% iron (e.g. sample 7 in Table 1). Sub-stoichiometric samples (e.g. sample 9) resulted in a relatively different microstructure with an uneven TiC particle size and distribution.

Table 1 shows the calculated lattice parameter and the predicted C/Ti ratio in TiC produced by reactions 1 and 2. For samples containing up to 37 wt% iron the lattice parameter of the carbide had a value of about 4.327 Å with little or no variation. This value corresponds to a C/Ti atomic ratio of about 0.9. This observation is illustrated in Fig. 6, which shows a decrease in the carbide lattice parameter for samples containing in excess of 37 wt% Fe. The lower C/Ti ratio of the more diluted samples may be caused by the lower combustion temperature as reported in Table 1.

Table 1. Sample specifications and experimental results

Sample	wt% Fe	C/Ti atomic ratio in green sample	Combust. temp. °C	Lattice parameter (Å)	x in TiC_x
1	0	1	2620	4.328	0.9
2	10	1	2580	4.327	0.9
3	17	1	not available	4.327	0.9
4	37	1	2558	4.327	0.9–(0.89)*
5	60	1	2286	4.323	0.75
6	80	1	1440	4.321	0.65
7	22	1	2540	4.327	0.9–(0.91)*
8	23	0.8	not available	4.327	0.9
9	24	0.6	1946	4.325	0.8–(0.75)*
10	25	0.4	1600	4.323	0.75

*The values in brackets were measured by chemical analysis.

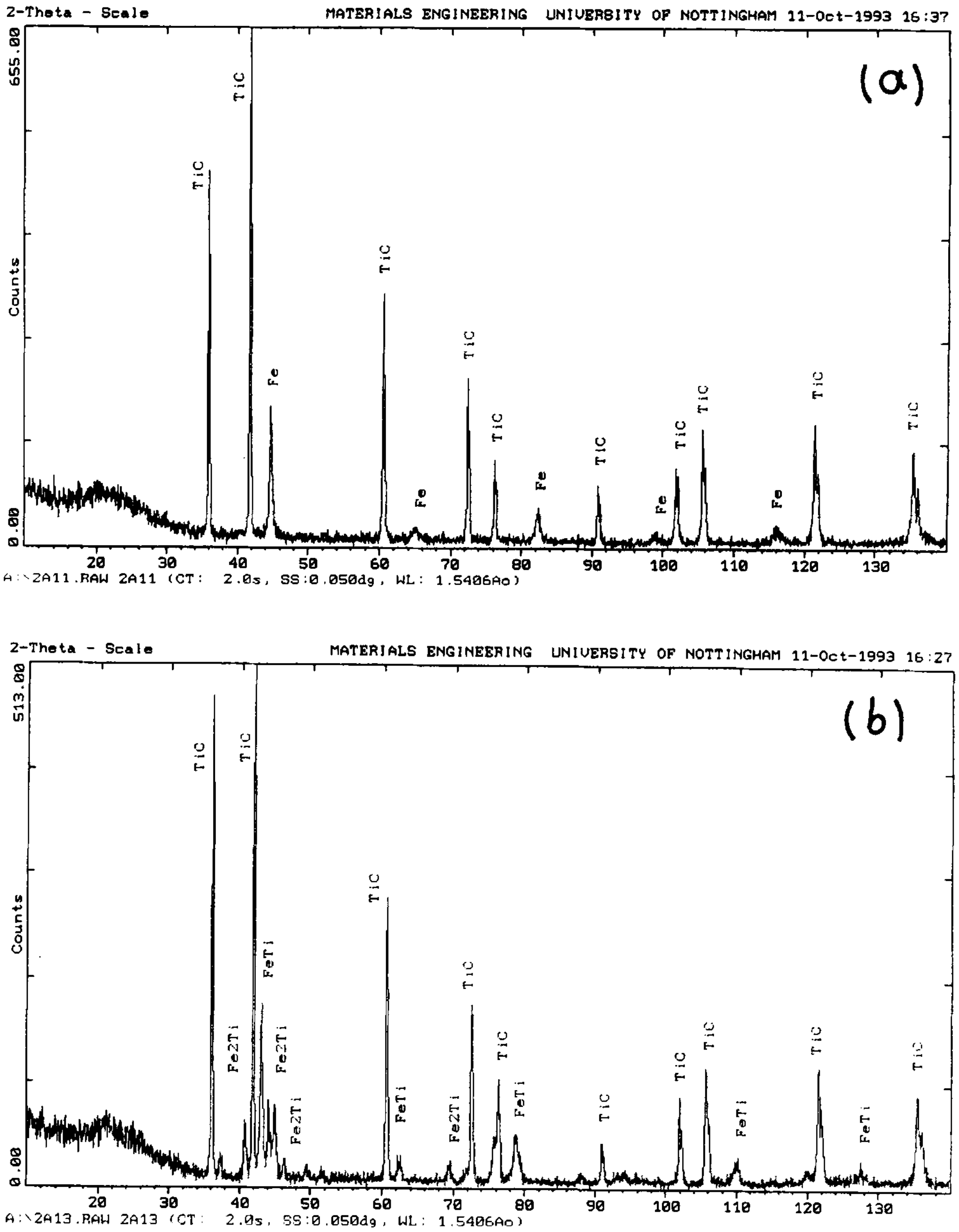

Fig. 7. X-ray diffraction pattern: (a) sample 7; (b) sample 9.

A study of reaction 2 showed that this also was self-sustaining for C/Ti ratios between 0.4 and 1. A typical temperature profile similar to that of reaction 1 was obtained. As shown in Table 1, the lattice parameter and so C/Ti ratio in TiC were not exactly dictated by the carbon composition of the starting mixture. In fact, the deficiency of carbon in the sample results in some un-reacted Ti in the form of FeTi and/or Fe_2Ti as shown by the results of X-ray diffraction analysis in Fig. 7.

The total and free carbon contents of some of the combusted samples were measured by chemical analysis. Using these data, the combined C/Ti ratio was calculated as presented in Table 1, which shows the predicted and measured C/Ti ratios to be in good agreement.

4 CONCLUSIONS

Fe–TiC composites varying in iron content of up to 80 wt% can be produced by means of the thermal explosion mode of combustion synthesis, using Fe, Ti and carbon black powders as the starting materials. The reaction was self-sustaining even when the C/Ti ratio in the mixture was well below the theoretical stoichiometric ratio (e.g. C/Ti = 0.4). The reaction yielded an even dispersion of particulate TiC in an iron matrix. For iron contents in the Fe–TiC composite up to 37 wt% the lattice parameter of TiC remained more or less constant. When the iron content exceeded this value, a significant drop in the carbide lattice parameter, and therefore the combined C/Ti ratio, took place. Similar results were observed when the amount of carbon black in the starting material was varied below the theoretical stoichiometric amount to form $TiC_{1.0}$. This behaviour was attributed to the fact that an increase in the iron content or a decrease in the carbon content resulted in a decrease in the combustion temperature and therefore in the combined C/Ti ratio.

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