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Microhardness and Microstructure of In-Situ Formed Fe-50%TiC Composites by Different Heating Methods

Melih KOÇYİĞİT[1] H. Erdem ÇAMURLU[1*]

[1]*Makine Mühendisliği Bölümü, Akdeniz Üniversitesi, 07058, Kampüs, Antalya, Türkiye*

Keywords

Volume Combustion Synthesis
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Abstract

The aim of this study is to fabricate in-situ TiC particle reinforced Fe matrix composites via volume combustion synthesis (VCS) through heating by two different sources. One group of reactant pellets was ignited by heating in an induction furnace (IF). The other group was ignited via heating by using a tungsten inert gas (TIG) torch. Thus, the differences in the microhardness and microstructure of the obtained composites could be compared. Fe, C and Ti elemental powders were used to obtain composites that contained 50 vol. % TiC in the Fe matrix. In the repeated experiments, the ignition temperatures of the IF pellets were found to be in 1164-1184 ºC range. The formation of composites was verified by X-ray diffraction (XRD) analyses, where it was seen that the products were composed of TiC and Fe with trace impurity phase. Scanning electron microscope (SEM) examinations showed that the in-situ formed TiC particles were regularly distributed in matrix in both series. The TiC particles obtained by TIG heating were about 5 times larger than the particles obtained by induction heating. Microhardness values of the samples were higher in IF series as compared to TIG series. It was shown that 50 vol. % TiC particle reinforced Fe matrix composites could be obtained by both heating methods. TIG was found to be a much practical method, when compared to conducting VCS in a furnace.

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Author ID (ORCID Number)

M. Koçyiğit, 0000-0003-2933-0436
H. E. Çamurlu, 0000-0003-3170-4492

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1. INTRODUCTION

Materials consist of different type of constituent that have different mechanical and thermal properties, are called as composites. Metals and polymers are mostly used as the matrix of a composite. They are preferred because of their ductility. On the other hand, ceramics are commonly used as reinforcement materials in metal matrix composites due to their high hardness (Koczak et al., 1993). Moreover, in-situ production of the composites has some superiorities such as thermodynamic stability at elevated temperatures, cleanliness of the matrix-reinforcement interfaces, even distribution of the particles in the matrix when they are compared to the ex-situ methods (Tjong & Ma, 2000).

Fe has been a popular matrix material since it has advantages such as low cost and ease in accessibility. Carbides (TiC, SiC, etc.) borides (TiB_2, etc.) and oxides (Al_2O_3, etc.) are in the list of reinforcements for Fe matrix composites. Mechanical and physical properties such as high hardness, low density and good wettability with Fe, render TiC a suitable reinforcement (Emamian et al., 2011).

Combustion synthesis is a relatively simple technique that exploits the self-maintaining characteristic of the exhothermic reactions. This method may be applied in two forms. In the first one, reaction travels from one side to the other, after the ignition from one side. This sort of synthesis is called self-propagating high temperature synthesis. In the second one, as a result of even heating, as opposed to ignition from one side, a

*Corresponding Author, e-mail: erdemcamurlu@gmail.com

complete reaction occurs in the total of the green pellet. Thus, this kind is called the volume combustion synthesis (Rogachev, 2017).

Combustion mechanism of Fe-Ti-C system was investigated intensively by different researchers (Fan et al., 1999; Jing & Yisan, 2007; Kocyigit & Camurlu, 2022; 2023). Choi et al. (1992) introduced the requirements of exothermic reactions in combustion synthesis in three steps. First, reaction needed to have high exothermicity. Second, there should be a liquid or vapor form for pre-reaction diffusion to occur. Third, heat loses must be less than heating rate. Accordingly, in some studies it was suggested that Fe-Ti eutectic temperatures play an important role on the reaction mechanism of the Fe-Ti-C system (Fan et al., 1999). In literature, there are studies that focus on the effect of the in-situ produced TiC particles, on the mechanical properties. It was reported that by Rahimi-Vahedi et al. (2018) that addition of TiC provided rise in hardness of the composite which was produced via combustion synthesis.

In some studies, on the formation of Fe matrix surface composites and coatings in literature, tungsten inert gas (TIG) heating method was used (Xinhong et al., 2009). The intense heat, which is created by the TIG torch, brings about the possibility of local heating on the surfaces of metals. Most studies concerning TIG heating, involve heating and melting of a powder mixture on the surface of a substrate. Thereby a surface composite is formed (Xinhong et al., 2009). Mechanical properties such as microhardness and wear resistance were reported to increase by incorporation of Fe-TiC surface composites via TIG heating (Zhao et al., 2019).

Although there are studies in literature on Fe-Ti-C system, no study was encountered on comparison of microhardness and microstructural properties of in-situ Fe-TiC composites which were produced by different heat sources such as induction furnace heating and TIG heating. In this study, in-situ formed TiC reinforced Fe matrix composites were produced by combustion synthesis from elemental fine Fe, Ti and graphite powders via both induction furnace heating and TIG heating processes. Unlike the studies in literature, in the present study TIG was not used for melting the products or formation of a coating. It was used solely for initiating the exothermic reaction in a preformed pellet of powder mixture. Effects of the two heating methods on microstructure and microhardness were compared. It was shown that 50 vol. % TiC particle reinforced Fe matrix composites could be obtained by both methods. TIG was found to be a much practical method, when compared to conducting VCS in a furnace.

2. MATERIAL AND METHOD

In-situ formed TiC-Fe composites were produced through VCS. Ignition of the samples were accomplished by two different methods. The first method involves heating in an induction furnace (IF) and in the second method the samples were heated by a tungsten inert gas (TIG) torch.

Elemental iron, titanium and carbon (as graphite) were employed to produce the in-situ Fe-50 vol.% TiC composites. In Table 1, the designation of the sample and contents of the starting materials are given. The same starting compositions were used for both induction furnace heating (IF) and tungsten inert gas (TIG) heating experiments according to Reaction (1). Prepared mixtures were pressed in a die. The green pellets were quite loose. The green pellet can be seen in Figure 1a.

$$Ti + C + xFe \rightarrow TiC + xFe \quad (1)$$

Table 1. *TiC contents and sample codes of the obtained composites and compositions of the starting powder mixtures*

TiC ratio of the Fe-TiC Composite (Vol. %)	**Designation**	**Initial Mixtures**		
		Fe (g)	**Ti (g)**	**C (g)**
50	**IF50/TIG50**	1.6689	0.8352	0.2094

2.1. Thermodynamic Calculations

Adiabatic temperatures (T_{ad}) of the systems that contained 50 vol. % TiC was calculated by HSC Chemistry 9. T_{ad} of a reaction is the maximum temperature that the products will get as a consequence of the developed exothermic energy, in the state that there is no heat loss from the yields. The Gibbs free energy (ΔG) of the Reaction (1) and of the other reactions which may occur in the system were calculated by the HSC 9.

2.2. Heating Processes

The IF50 series was heated in an induction furnace. Before the experiments, vacuum was applied to the system and then argon gas was supplied. A pyrometer was used for monitoring the temperature of the sample during heating. The exothermic reaction started by the escalation of temperature, which was taken as the ignition temperature, T_i. In induction heating, power was turned off by the time the ignition occurred. The temperature of the sample continued to increase due to the released heat and reached the combustion temperature, T_c.

The TIG50 was heated by a TIG torch (40A electrical current). In TIG heating, heating was continued for 10s since it was sometimes not possible to distinguish the ignition moment. Therefore, higher temperatures are expected to be attained by TIG heating than induction heating. The argon gas blow of the TIG torch prevented the oxidation of the reactants and the products. The argon gas blow was continued until the products were cooled to room temperature. In TIG heating, there is also an instant heating and occurrence of the reaction in the whole volume of the sample at once. Therefore, it is believed that TIG heating can be defined as VCS.

2.3. Characterization

The products were sanded with 180 - 3000 number sandpapers. Micro structures were examined by optical microscopy and scanning electron microscope which had a field emission gun (FEI). Microhardness was determined by a Micro Vickers Hardness Tester (THV-1DTe). XRD analyses were conducted on product pellets by a Rigaku Smartlab Unit by Cu-Kα cathode tube.

3. RESULTS AND DISCUSSION

3.1. T_{ad}, T_i and T_c

According to the calculations which were made in this study, the adiabatic temperature of the reaction for the formation of the composite that contained 50 vol. % TiC is 1535 ^oC. This is the melting point of Fe. It was seen that the heat formed by the reaction is capable of heating the products to the melting point of iron. It can be inferred that some of the iron melts.

The combustion temperatures that were recorded during the formation of samples IF50 in different trials are presented in Table. 2. It can be seen that the measured T_c are below the calculated T_{ad} values. The difference probably arises from the loss of heat from the products through the substrate and the crucible during VCS.

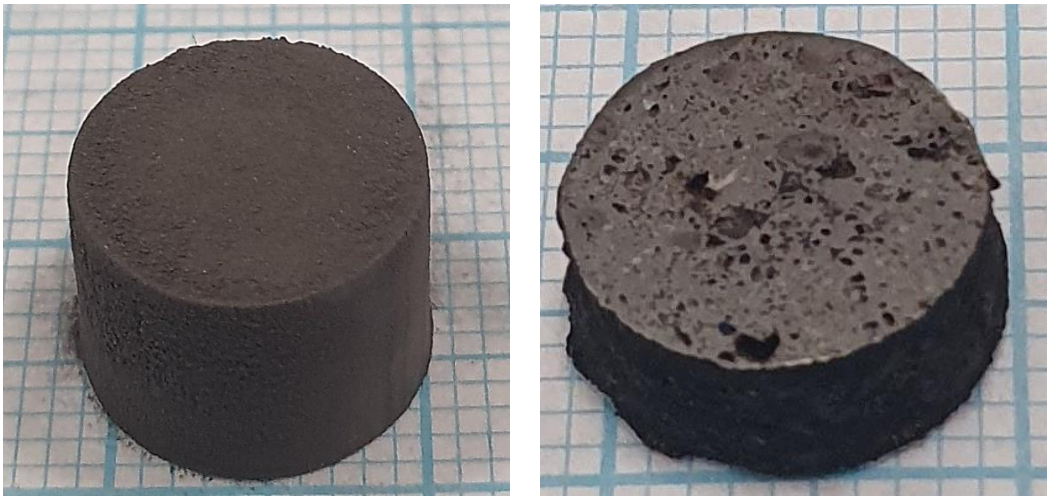
Table 2. *T_i and T_c of IF50 samples*

Sample	Ignition Temperature, T_i (^oC)	Combustion Temperature, T_c (^oC)
IF50 (Trial 1)	1164	1358
IF50 (Trial 2)	1175	1358
IF50 (Trial 3)	1184	1338

The ignition temperatures of the samples were between 1164-1184 ^oC. The closeness of the obtained results indicates the accuracy of the temperature measurements. In combustion synthesis, ignition mostly takes place by the formation of liquid phase in the system. In Fe-Ti there are two eutectic reactions at 1085 ^oC (Ti-FeTi) and the at 1290 ^oC (Fe_2Ti-Fe) (Murray, 1981). The measured combustion temperatures are slightly above the

eutectic reaction at 1085 °C. It can be suggested that after diffusion of Fe atoms into Ti particles, as a result of the eutectic reaction of Ti-FeTi, a liquid phase forms and ignition takes place. Ti and C dissolves and TiC particles precipitates in the formed liquid (Fan et al., 1999).

In this study, ignition of the reactant pellet was conducted by both induction furnace (IF) heating and TIG heating. After the combustion synthesis reactions in both systems, the product pellet remained intact and maintained its integrity (Figure 1). This observation indicated that TIG heating was utilized successfully for the ignition of the pellet.



(a) (b)

Figure 1. *Images of* ***a)*** *IF50 green pellet,* ***b)*** *IF50, after VCS (Polished)*

Gibbs free energy values of some of the compounds, in the Fe-Ti-C system are given in Figure 2. It can be seen that TiC is the most feasible compound in the system, with the lowest free energy values. Formation of Fe_3C is not expected due to its high free energy of formation. Fe_2Ti and FeTi are possible products, with negative Gibbs free energies. Their formation may be possible at points where C concentration is low and Reaction (1) is not complete (Kocyigit & Camurlu, 2023).

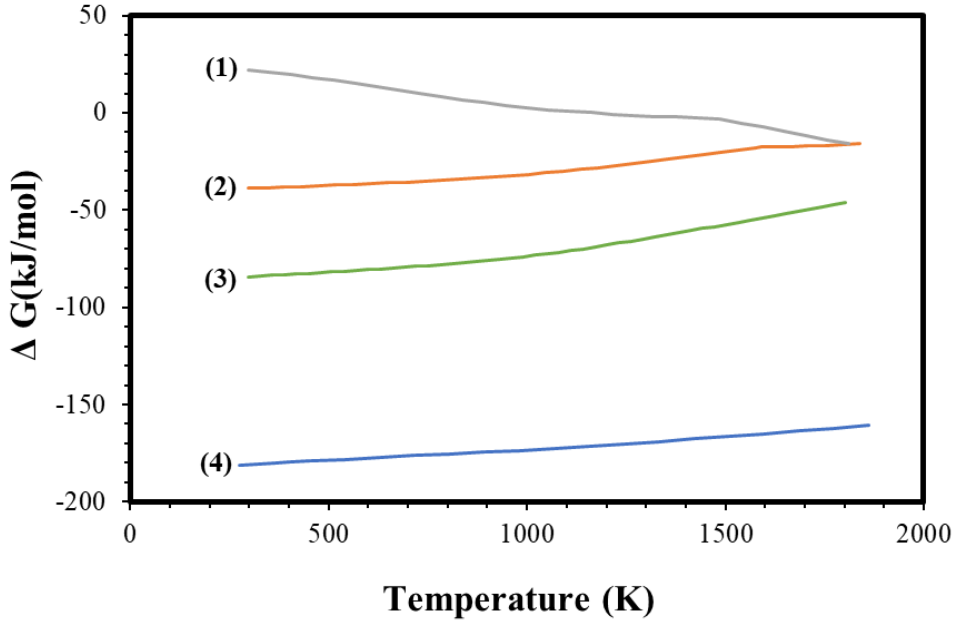


Figure 2. *Gibbs free energy of some species in Fe-Ti-C reaction system;*
(1) *Fe_3C;* ***(2)*** *FeTi;* ***(3)*** *Fe_2Ti;* ***(4)*** *TiC*

3.2. XRD Analyses

XRD pattern of the starting mixture of Fe-50 vol.% TiC composite is presented in Figure 3a. It can be seen that the pattern contains Fe and Ti peaks. After the VCS reaction, the Fe peaks remain, on the other hand, the peaks of the Ti phase disappear in the XRD pattern of the VCS products (Figure 3b). This indicates the consumption of the Ti phase. The Fe peaks in the XRD pattern of the products indicate that the matrix phase is composed of Fe in the composites, as intended, with trace amount of impurity phase, Fe_2Ti. According to Gibbs free energy calculations, formation of Fe_2Ti is possible, however it is not a stable product when C is present. Trace amount of this phase may have formed in regions where carbon is limited and TiC formation was not possible (Kocyigit & Camurlu, 2023). There is also the possibility of C loss from the system., which would lead to a surplus of Ti in the system. It can be seen in the XRD pattern of the products that the peaks of the TiC phase appear, indicating the formation of the TiC particles in the products.

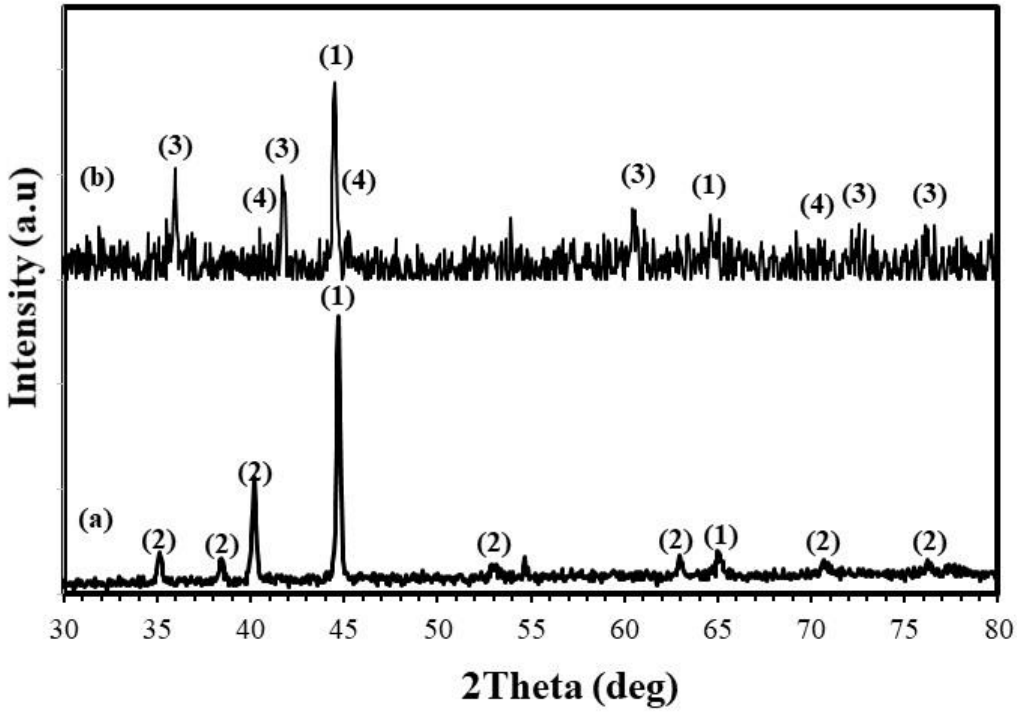


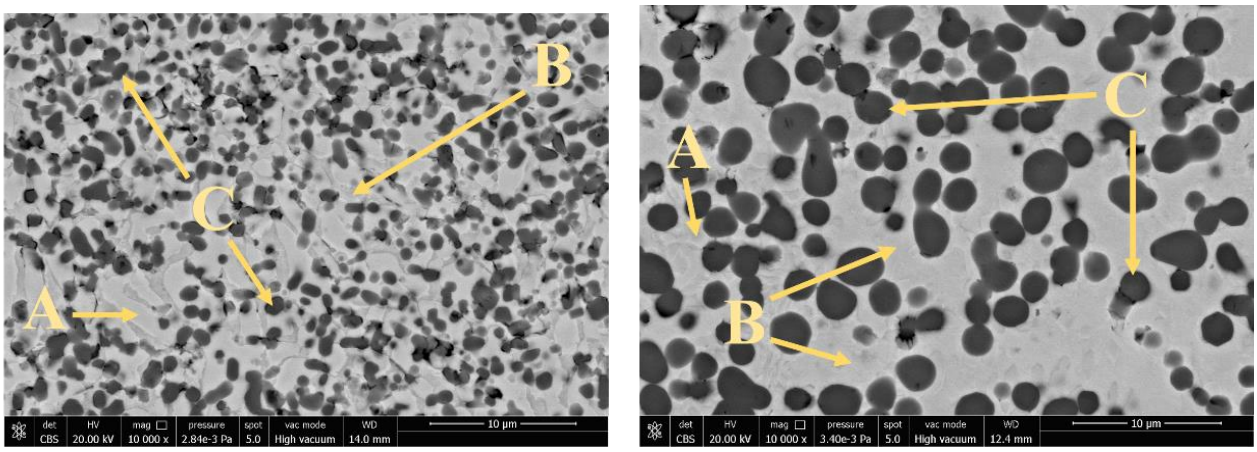
Figure 3. *XRD pattern of the* ***a)*** *IF50 initial powder mixture,* ***b)*** *IF50 composite formed after VCS by induction heating;* ***(1)*** *Fe,* ***(2)*** *Ti,* ***(3)*** *TiC,* ***(4)*** *Fe_2Ti*

3.3. Microstructure

SEM micrographs of the composites containing 50 vol. % TiC are presented in Figure 4. Product in Figure 4a was obtained by induction furnace heating and product in Figure 4b was obtained by TIG heating. In these images, the dark-colored round particles are TiC (marked as C in the Figure 4) as verified by the EDS analyses. Homogenous distribution of TiC particles was seen to take place in the composites which were obtained by both of the heating methods.

The sizes of the TiC particles were observed to be smaller in the samples which were ignited by IF heating, than the samples which were ignited by TIG heating. The sizes of the TiC particles were below 1 micrometer in the composites obtained by induction heating. On the other hand, the size of the TiC particles were 3-4 micrometers in TIG heated composite. It can be inferred that the sizes of the formed TiC particles were larger in the composites which were obtained by TIG heating. The difference may have arisen due to higher temperatures attained during TIG heating. In induction heating, power was turned off as soon as the ignition took place. On the other hand, in TIG heating, heating was continued for 10 s since it was not possible to distinguish the ignition moment. Therefore, higher temperatures are expected to be attained by TIG heating. Higher temperature attained in TIG heating may have led to higher rate of TiC grain growth caused by the Ostwald ripening.

According to the EDS analyses, regions that are marked as A (Figure 4) have 25/75 molar Ti/Fe ratio. On the other hand, regions that are marked as B has 7/93 Ti/Fe molar ratio (Table 3). The detected Ti in Fe can be taken as an indication that Reaction (1) was not complete in the system. The unconsumed Ti may have remained in the Fe matrix. Although TiC and Fe major phases were detected by the XRD analyses of the products (Figure 3), trace amount of Fe_2Ti phase was present. Therefore, the XRD results are in accord with the EDS analysis.



(a) (b)

Figure 4. *SEM images of the induction and TIG heated composites* ***a)*** *IF50,* ***b)*** *TIG50 (Magn. 10kX)*

Table 3. *Molar Ti/Fe ratios of the regions that are given in the Figure 4*

Region	**A**	**B**
nTi/nFe	25/75	7/93

3.4. Microhardness

TiC was found to be effective in enhancing hardness, since the microhardness value of unreinforced Fe matrix was 29.8 $HV_{0.2}$. It can be seen in Table 4 that in general, composite that was obtained by induction heating presented slightly higher microhardness values than the composite which was obtained by TIG heating. This difference may be a result of the presence of finer TiC particles in the samples, which were obtained by induction furnace heating. The finer sizes of TiC particles were observed during microstructural examinations (Figure 4). Composites having finer reinforcement particle size generally exhibit higher hardness than the ones having coarser reinforcement (Koczak et al., 1993).

Table 4. *Microhardness Values of IF50 and TIG50*

Sample	**Measurements**					**Average ($HV_{0.2}$)**	**Std. Dev.**
	1.	2.	3.	4.	5.		
IF50	775.3	785.9	710.3	675.2	690.5	727.4	50.2
TIG50	630.9	570.1	702.9	624.4	554.8	616.6	58.5

When the two heating methods are compared, it can be suggested that the TIG heating is a much easier and faster method. TIG was found to be more practical than heating in the induction furnace.

4. CONCLUSION

In this study, the effect of different heating sources (induction furnace (IF) and tungsten inert gas (TIG) welding machine) on microstructure and microhardness of in situ TiC particle reinforced Fe matrix composites was investigated. According to microstructure examinations and XRD results, TiC particles were formed successfully in-situ in the Fe matrix via combustion synthesis by both heating techniques. Trace amount of Fe_2Ti was also detected. The TiC particles were homogeneously distributed in the structure. The TiC particles obtained by TIG heating were about 5 times larger than the particles obtained by induction heating. In general, the samples which were produced by induction furnace heating exhibited higher microhardness values than TIG produced samples. This was attributed to finer reinforcement size of the obtained composites when IF heating was used. It was shown that 50% TiC particle reinforced Fe matrix composites could be obtained by both methods. TIG was found to be a much practical method, when compared to VCS in a furnace.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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