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Role of nano-size TiO_2 particles on the crystallite size of microwave—Combustion synthesized Al_2O_3/TiC composite

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ABSTRACT

The main goal of the current study is to clarify the effect of TiO_2 particles size (micro and nano) on the crystallite size of microwave—combustion synthesized Al_2O_3/TiC composite. Al, C, both nano- and micron TiO_2 have been mixed mechanically and milled using a planetary ball mill. The milled samples have been pressed before exposing to microwave with different powers. In order to validate the formation of Al_2O_3 and TiC phases, the samples have been analyzed by X-ray diffraction. Crystallite sizes of produced TiC and Al_2O_3 in samples containing micron TiO_2 are about 46.6–87.9 and 63.8–208.8 nm respectively and in samples containing nano-TiO_2 are about 38.2–68.7 and 54.4–99.5 nm respectively. It means that using nano-TiO_2 as one of the raw materials leads to formation of nano-structure Al_2O_3/TiC composite. Microstructure of produced composites has been evaluated using a scanning electron microscope (SEM). Microscopic evaluations show that Al_2O_3 phase in micron samples has a regular shape, unlike needle shape in nano-samples.

1. Introduction

Al_2O_3/TiC composite with special properties such as hardness, toughness, chemical stability, good strength, electrical conductivity and excellent wear resistance [1] has a wide application in cutting tools, wear resistance coating and computer pieces such as magnetic head sliders [2]. Currently, it is manufactured primarily by pressure less sintering or hot pressing the direct mixtures of Al_2O_3 and TiC powders [3], mechanical alloying [4], pulsed laser deposition (PLD) [1], etc. Nowadays, combustion synthesis (CS) because of having advantages such as simplicity, a relatively low energy requirement and feasibility of using low-cost raw materials has been one of the most important techniques for fabrication of cermet composites [5] such as Al_2O_3/TiC composite. In this method, the synthesis is obtained through an extremely rapid self-sustaining process driven by the large heat release by the internal energy of the reactants [6].

According to the literature survey done by the authors there are many articles focused on synthesis of alumina/titanium carbide composites. The effect of different factors such as fabrication methods, using different heating sources, role of particle size of initial powders on produced Al_2O_3/TiC composite, effect of green density, extra aluminum and electric field on density of products, the role of carbon sources, cooling rate and preheating temperatures on microstructure of this composite and the effect of various parameters such as mechanically activation, microwave power, green density and so on, on the combustion behavior of this composite were studied in previous articles [7–10]. Also based on literature survey, there are a few papers concentrated on the use of microwave energy to synthesize Al_2O_3/TiC composite. Besides the role of nano-size TiO_2 and different factors such as mechanically activation, the influence of the use of diluent materials (for example aluminum) and green density on crystallite size of Al_2O_3/TiC composite has not been under attention. Thus the main goal of the current study is to fill the literature gap appeared in this field. Considering the possibility of production nano-structure composite is another target of this study.

Because of the interrelationship between the effective parameters, the analysis of the results is a labor and time consuming work, thus, Taguchi robust design method of system optimization with L_9 orthogonal array was used to determine the best level of each factor [9]. This method explained in more detail elsewhere [10].

2. Experimental details

2.1. Materials

In order to produce Al_2O_3/TiC composites, flake-like C with 99.93% purity, Al particles with irregular shape, less than 45 μm and 99.4% purity were purchased. Nano- and micron TiO_2 particles with average particle size of about 70 nm and 0.6 μm respectively were used as raw materials. Purity of nano- and micron TiO_2 was 90% and 99.94% respectively. Al_2O_3/TiC composites were produced by mixing of powders

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Table 1
Main controlling factors and their levels.

Factors	Levels		
	1	2	3
Green density (% ideal density)	58	61.5	64.5
Excess Al (mole)	0	1	2
Ball milling time (h)	0	0.5	1
Power of microwave (W)	600	900	–

based on Eq. (1):

$$3TiO_2 + 3C + (4+x)Al = 3TiC + 2Al_2O_3 + xAl \quad (1)$$

The exact reason of using extra aluminum can be attributed to the fact that the temperature increases during combustion synthesis process and based on the literature survey, it was found that a diluent (extra Al or Al_2O_3) can be used because of controlling the temperature.

It has been empirically suggested that combustion reactions will not become self-sustaining unless $T_{ad} \geq 1800$ K [8]. The calculated theoretical adiabatic temperature of this reaction is 2546 K using thermodynamics data [8,10]. It means that according to merzhanove constant, Al_2O_3/TiC composite can produce under combustion synthesis condition.

2.2. Orthogonal array

Four controlling parameters (extra Al, milling time, green density and microwave power) were chosen for experiments and maximum level of them is three. According to orthogonal arrays designed by Taguchi, they are respondent to orthogonal array L_9. Therefore in the current study and based on the Taguchi method, the standard orthogonal array, namely L_9 that reduces the number of experiments to 9 was used. Different parameters and levels are given in Table 1. According to L_9 orthogonal array and Table 1, product qualification of each 9 samples, are given in Table 2. In order to compare the effect of TiO_2 particles size, two series of samples were prepared, i.e. a set of 9 samples using micron TiO_2 and another set using nano-TiO_2.

2.3. Sample preparation

Primary mixtures were performed by mixing of powders based on Eq. (1). With replacing $x = 0$, 1 and 2 in Eq. (1), primary mixtures with 0, 1 and 2 mole extra Al were prepared. At next step, based on Table 2, primary mixtures were milled for 0.5 and 1 h in a planetary ball mill using steel balls with 7 and 12 mm in diameter. The ball to powder ratio was 20/1 and frequency of milling was 252 rpm. Milling was done at room temperature. After milling the mixed powders were compressed to achieve some discs with 14.2 mm in diameter and different green densities. Green densities of discs were about 58%, 61.5% and 64.5% of ideal density. Finally, last stage accomplished by exposing the discs under 600 and 900 W microwave energy.

2.4. Characterization

In order to confirm the formation of Al_2O_3 and TiC phases and to determine their crystallite size, X-ray diffraction measurements were performed. A Bruker AXS, D8 Advance diffractometer was performed using Cu Kα radiation. The range of 2θ, step, step time and temperature were 15–80°, 0.025°, 1 s and 25 °C respectively. The wavelength was 0.15418 nm.

2.5. Microscopic evaluation

A scanning electron microscope (SEM, LEO 1450 VP) was used to evaluate the microstructure of produced composite.

Table 3
Produced phases according to the XRD patterns.

Produced phases	Micron samples	Nano-samples
Al_2O_3	√	√
TiC	√	√
Al_4O_4C	√	√
Ti_9Al_{23}	–	√
Ti_5O_9	√	√
TiO_2	√	–
Al	√	√
C	√	-

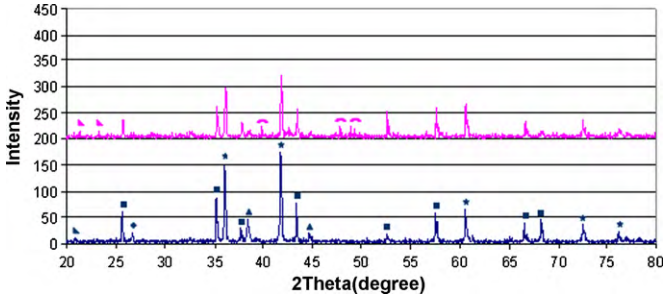

Fig. 1. X-Ray diffractions of sample 6 (upper graph: nano-sample and lower graph: micron sample). (◣) Al_4O_4C, (■) Al_2O_3, (⋆) TiC, (▲) Al, (♦) C and (◠) Ti_9Al_{23}.

3. Results and discussion

3.1. Phase identity

According to X-ray diffraction patterns, both Al_2O_3 and TiC phases are present in all micron and nano-samples. Besides of the desired products, some oxide and intermetallic phases have been seen in several samples, especially in nano-samples because of air atmosphere (Table 3). For example, XRD patterns and SEM micrographs (a: micron and b and c: nano-sample) of sample 6 shown in Figs. 1 and 2 respectively. In Fig. 2, Al_2O_3 and TiC phases are seen as gray and white zones respectively. As seen the shape of Al_2O_3 phase in micron samples is as a particle (Fig. 2(a)) and in nano-samples is looks like a particle and whisker (Fig. 2(b) and (c)). Perhaps the reason can be attributed to the fact that cooling rate of sample surface is much higher than that of the center and leads to form Al_2O_3 phases in surface of nano-samples as particles (Fig. 2(b)), while central Al_2O_3 appears like a whisker (Fig. 2(c)).

Since high temperature can be the main reason for the conversion of particles to whiskers, thus, one may conclude that in nano-samples the incremental combustion temperature is much higher than that of micro samples. Since combustion temperatures depend on green density, extra Al, milling time and power of microwave [10] and crystallite growth is hardly depended on the mentioned parameters. Fig. 3 shows the variation of combus-

Table 2
Preparing conditions of each sample.

Sample no.	Green density (% ideal density)	Extra Al (mole)	Milling time (h)	Power of microwave (W)
1	58	0	0	600
2	58	1	0.5	900
3	58	2	1	600
4	61.5	0	0.5	600
5	61.5	1	1	600
6	61.5	2	0	900
7	64.5	0	1	900
8	64.5	1	0	600
9	64.5	2	0.5	600

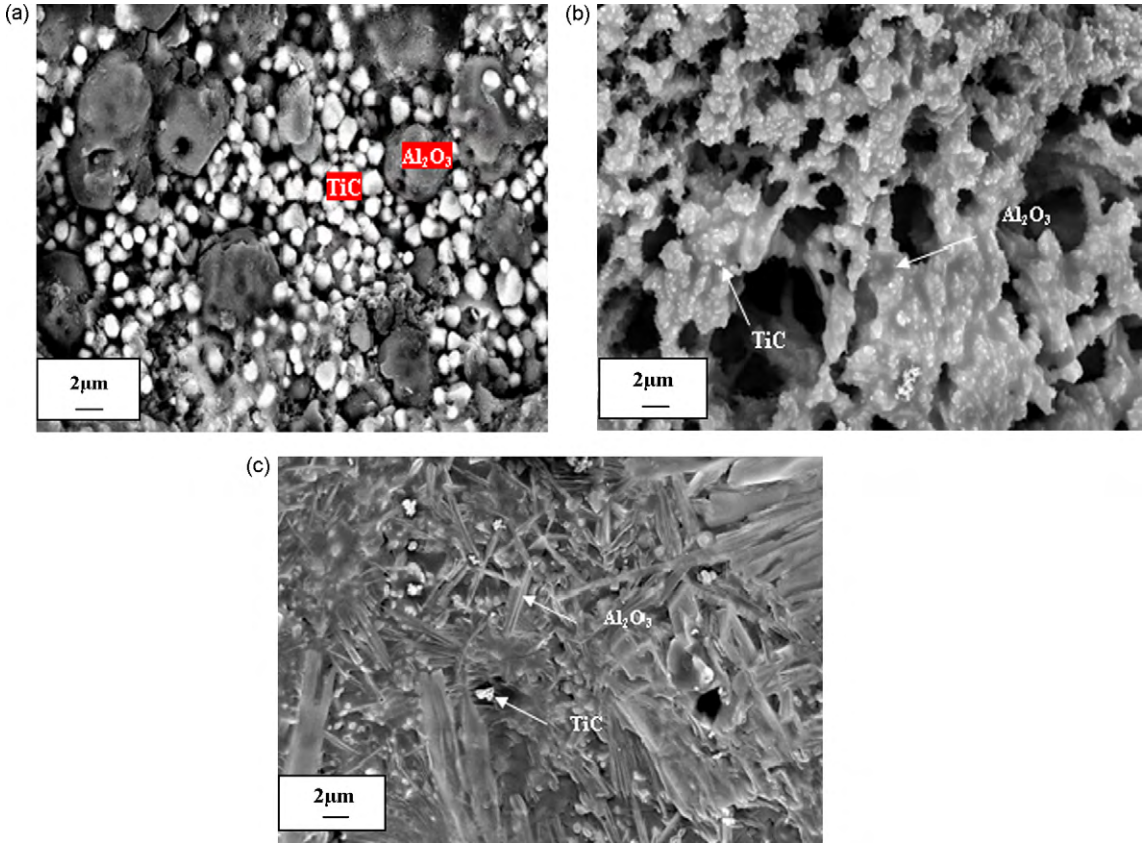

Fig. 2. SEM micrographs of sample 6: (a) micron sample and (b and c) nano-sample.

tion temperature on above parameters for both nano- and micron samples.

3.2. Calculation of crystallite size and related S/N ratios

Crystallite sizes of Al_2O_3 and TiC phases in all micron and nano-samples were calculated using XRD patterns and Sherrer equation (Eq. (2)) [11]:

$$D = \frac{0.9\lambda}{B_{1/2}\,\mathrm{Cos}\,\theta} \tag{2}$$

where D is the crystallite size (nm); λ is the wave length (0.15418 nm); $B_{1/2}$ is the FWHM (Rad); θ is the half of brrag.

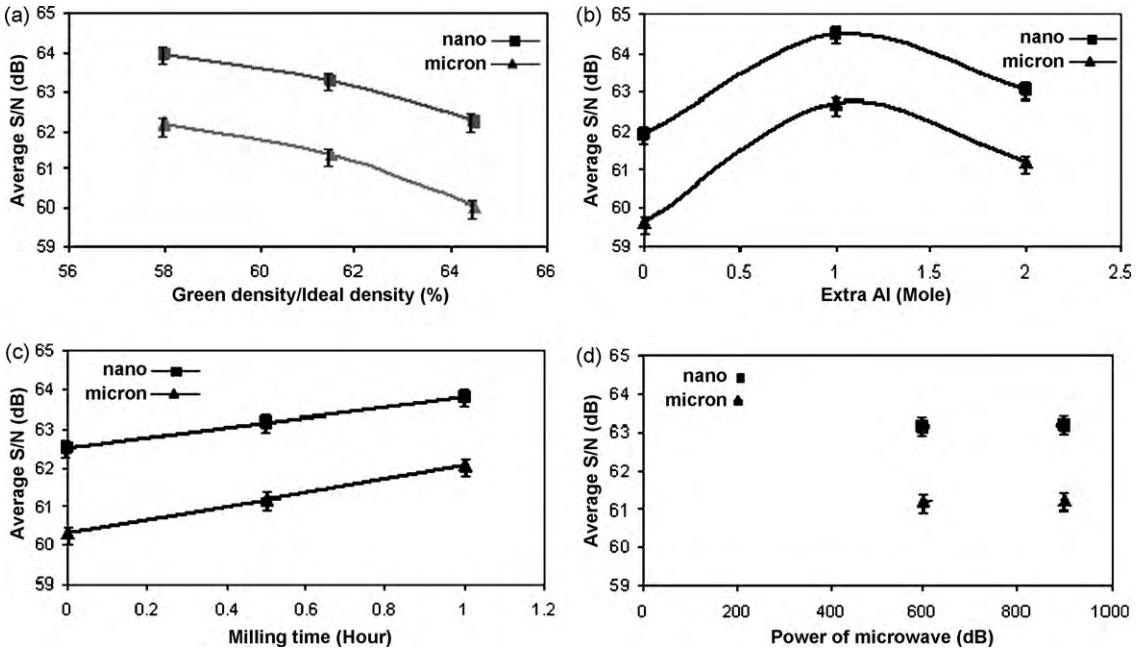

Fig. 3. Effect of green density, extra Al, milling time and microwave power on combustion temperature.

Table 4
Taguchi orthogonal array $L_9(3^4)$ and the experimental measured values and S/N ratio for TiC and Al_2O_3 crystallite sizes when micron TiO_2 was used in raw materials (micron samples).

Exp. no.	Green density (%)	Extra Al (mole)	Milling time (h)	Power (W)	TiC crystallite size (nm)	S/N	Al_2O_3 crystallite size (nm)	S/N
1	100	0	0	600	46.6	−33.37	208.8	−46.39
2	100	1	0.5	900	75.4	−37.55	140.4	−42.95
3	100	2	1	600	70.4	−36.95	64.9	−36.24
4	150	0	0.5	600	69.7	−36.86	194.6	−45.78
5	150	1	1	600	87.9	−38.88	85.6	−38.65
6	150	2	0	900	51.9	−34.30	68.5	−36.71
7	200	0	1	900	68.8	−36.75	155.7	−43.84
8	200	1	0	600	49.5	−33.89	147.6	−43.38
9	200	2	0.5	600	64	−36.12	63.8	−36.10

Table 5
Taguchi orthogonal array $L_9(3^4)$ and the experimental measured values and S/N ratio for TiC and Al_2O_3 crystallite size when nano-TiO_2 was used in raw materials (nano-samples).

Exp. no.	Green density (%)	Extra Al (mole)	Milling time (h)	Power (W)	TiC crystallite size (nm)	S/N	Al_2O_3 crystallite size (nm)	S/N
1	100	0	0	600	38.2	−31.64	56.9	−35.11
2	100	1	0.5	900	54.7	−34.76	74.0	−37.39
3	100	2	1	600	51.9	−34.30	57.2	−35.15
4	150	0	0.5	600	51.3	−34.20	66.4	−36.44
5	150	1	1	600	68.7	−36.74	71.8	−37.12
6	150	2	0	900	48.4	−33.70	77.1	−37.74
7	200	0	1	900	58.1	−35.29	54.4	−34.71
8	200	1	0	600	46.5	−33.36	99.5	−39.96
9	200	2	0.5	600	64	−36.12	59.5	−35.49

Since, often, production a composite with finest crystallites and particles is desirable, thus Eq. (3) that is proper for this purpose [7,8,10] was used for calculating of S/N ratios:

$$\frac{S}{N_N} = -10\,\mathrm{Loh}_{10}\frac{1}{n}\left(\sum y_i^2\right) \quad (3)$$

where y_i is the characteristic property and n is the number of measurements in each experiment. Tables 4 and 5 show TiC and Al_2O_3 crystallite sizes and related S/N ratios for micron and nano-samples respectively. Based on the results appeared in above tables, the presence of nano-TiO_2 in raw materials makes finer crystallites of Al_2O_3 and TiC respect to micron TiO_2. Also it can be seen that TiC crystallite sizes are in nano-range in both micron and nano-samples but Al_2O_3 crystallite sizes only in nano-samples are in nano-range (smaller than 100 nm). Briefly, replacing micron size of TiO_2 by nano-size in raw materials causes to increase the probability of production nano-structure Al_2O_3–TiC composite as well as reduction of Al_2O_3. The reason can be attributed to the nucleation mechanism of TiC from Ti and C. Benoit et al. [12] offered two mechanisms for nucleation of TiC from Ti and C during combustion synthesis. In first mechanism, TiC nucleates from interface of solid titanium and molten titanium that saturated from carbon. In second, TiC nucleation occurs in interface of molten titanium and solid carbon. It is clear that crystallite size of TiC depend on particles size of solid titanium and solid carbon in first and second mechanisms respectively. Fig. 4 shows a TiC particle that surrounded by carbon particle. This means that TiC particles nucleated similar to what proposed by second mechanism, i.e. from surface of carbon particles. Since carbon particles size in both nano- and micron samples are fix, thus TiC particles size do not show a salient variation.

3.3. Optimal parametric setting

For prospecting of the best level from each parameter, graph analysis was used. The mean S/N ratios for each level of the parameters are summarized in Tables 6–9 for TiC and Al_2O_3 crystallite sizes. The highest values have the maximum impact on the final results while the lowest values show least impact [13].

Table 6
Average S/N ratio for each level of the parameters for TiC crystallite size when micron TiO_2 was used in raw materials.

Factor	Average S/N ratio		
	Level 1	Level 2	Level 3
Green density (% ideal density)	−35.96	−36.68	−35.59
Extra Al (mole)	−35.66	−36.77	−35.79
Ball milling time (h)	−33.85	−36.84	−37.53
Power of microwave (W)	−36.01	−36.20	–

Table 7
Average S/N ratio for each level of the parameters for Al_2O_3 crystallite size when micron TiO_2 was used in raw materials.

Factor	Average S/N ratio		
	Level 1	Level 2	Level 3
Green density (% ideal density)	−41.86	−40.38	−41.10
Extra Al (mole)	−45.34	−41.66	−36.35
Ball milling time (h)	−42.16	−41.61	−39.58
Power of microwave (W)	−41.09	−41.17	–

Table 8
Average S/N ratio for each level of the parameters for TiC crystallite size when nano-TiO_2 was used in raw materials.

Factor	Average S/N ratio		
	Level 1	Level 2	Level 3
Green density (% ideal density)	−33.57	−34.88	−34.92
Extra Al (mole)	−33.71	−34.95	−34.71
Ball milling time (h)	−32.90	−35.03	−35.44
Power of microwave (W)	−34.39	−34.58	-

Table 9
Average S/N ratio for each level of the parameters for Al_2O_3 crystallite size when nano-TiO_2 was used in raw materials.

Factor	Average S/N ratio		
	Level 1	Level 2	Level 3
Green density (% ideal density)	−35.88	−37.10	−36.72
Extra Al (mole)	−35.42	−38.16	−36.13
Ball milling time (h)	−37.60	−36.44	−35.66
Power of microwave (W)	−36.54	−36.61	–

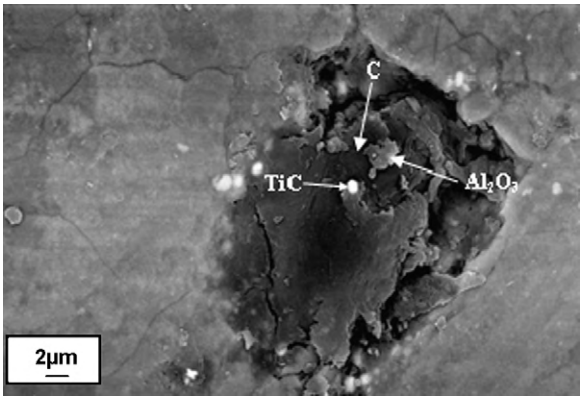

Fig. 4. TiC particle surrounded by retained carbon.

3.3.1. Effect of green density on crystallite size

As seen in Fig. 5(a) in micron samples 64.5% green density has the highest impact and should be the best choice among the three levels of green density on TiC crystallite size when micron TiO_2 is used as raw material and 58% and 61.5% green densities are stand on next places respectively. In nano-samples for having finest TiC crystallite size, 58%, 61.5% and 64.5% green densities are proper choices respectively. Fig. 5(b) shows that in micron samples 61.5%, 64.5% and 58% green densities induce to formation of fine Al_2O_3 crystallites respectively. Fig. 5(b) also shows that Al_2O_3 crystallites in nano-samples depends on green density and the minimum size belongs to 58% green density and 64.5%, 61.5% stand in second and third places respectively.

3.3.2. Effect of extra aluminum on crystallite size

The effect of extra Al on crystallite sizes of TiC and Al_2O_3 is shown in Fig. 6. It can be seen that 1 mole extra aluminum causes to form the biggest TiC crystallites in both groups of samples. 2 mole extra aluminum forms mediocrity crystallites and samples without extra aluminum have finest TiC crystallites among these three levels of extra aluminum in both groups of samples. This can explain

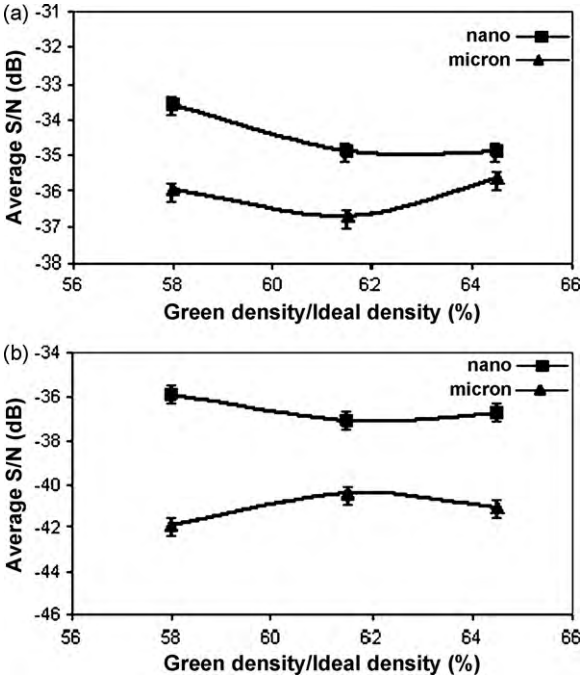

Fig. 5. Effect of green density on (a) TiC and (b) Al_2O_3 crystallite size.

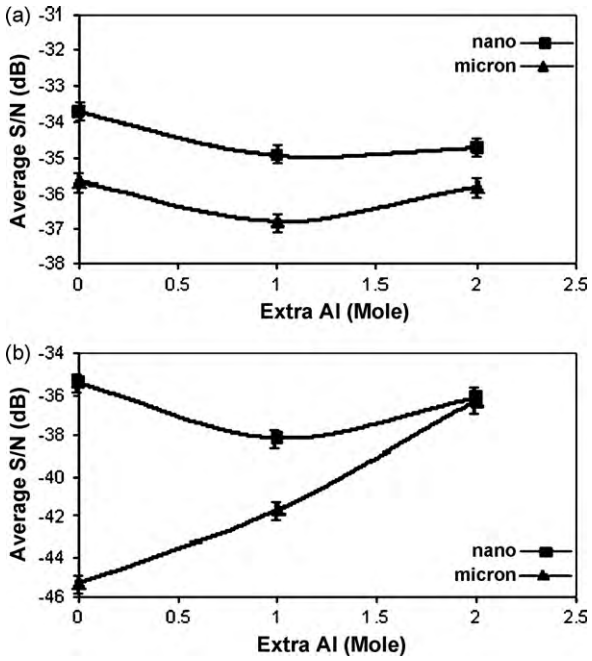

Fig. 6. Effect of extra Al on (a) TiC and (b) Al_2O_3 crystallite size.

using Fig. 3(b) that shows the effect of extra aluminum on combustion temperature. As seen the highest combustion temperature is determined in the presence of 1 mole extra aluminum. Since crystallite growth is hardly depend on combustion temperature, thus the biggest TiC crystallites form in the presence of one mole extra aluminum. Similarly from this figure it can be seen that 2 mole extra aluminum makes a medium combustion temperature and leads a medium TiC crystallites and the smallest TiC crystallites forms in the absence of extra aluminum because of lowest combustion temperature. As Fig. 6(b) shows the variations of Al_2O_3 crystallite size in nano-samples is similar to TiC crystallite size that can be verified in Fig. 3(b).

3.3.3. Effect of milling time on crystallite size

Average S/N–milling time graphs for Al_2O_3 and TiC crystallite sizes are shown in Fig. 7. As seen in Fig. 7(a), in both nano- and micron samples finest crystallites of TiC produced in the absence of ball milling. Also coarse crystallites can be seen in samples that were milled for 1 h. The reason of this phenomenon can be explained using pervious work done by Cochepin et al. [14]. It seems the formation of TiC phase during combustion synthesis reaction occurs in some steps. In the first step TiC_x with less carbon content nucleates and its growth takes place with diffusion of carbon in Ti lattice. Milling of raw materials creates some defects in their lattices and finally increases the diffusivity of carbon to Ti lattice. This increment in diffusion coefficient induces to further growth of TiC crystallites. This is why TiC crystallite size increases as milling time increases. Fig. 7(b) shows that Al_2O_3 crystallite size decreases with increasing of milling time. The reasons can be related to decreasing of raw materials size as milling time increases. Another reason can be attributed to the great growth of TiC crystallites that causes the molten Al_2O_3 to disperse among them easily. This is why the Al_2O_3 crystallites are smaller than TiC.

3.3.4. Effect of power of microwave on crystallite size

The effect of power of microwave on TiC and Al_2O_3 crystallite sizes is shown in Fig. 8. As seen in both nano- and micron samples, crystallite sizes of TiC and Al_2O_3 are decreased with reducing power of microwave from 900 to 600 W. This is because reduction

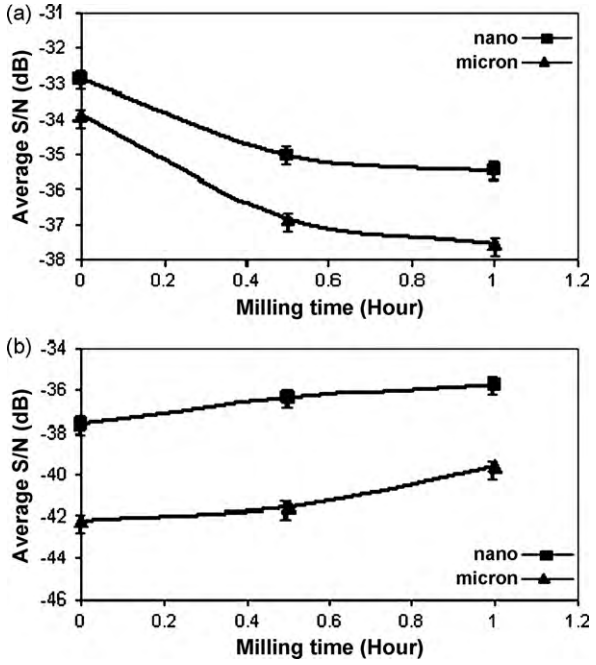

Fig. 7. Effect of milling time on (a) TiC and (b) Al_2O_3 crystallite size.

of power corresponds to the reduction of temperature and means in the current study the smallest crystallite size will be appeared at lowest microwave power, i.e. 600 W.

3.4. Contribution of each parameter on crystallite size

ANOVA method was used to clarify the contribution percent of parameters on crystallite size of alumina and titanium carbide phases. Details of this method were explained in elsewhere [10]. Tables 10 and 11 show that milling time has the most influence on the TiC crystallite size with 84.54% contribution in micron samples and 63.98% contribution in nano-samples. Extra Al and green density are second important factors in micron and nano-samples respectively. The power of microwave has the least influence on the TiC crystallite size in both of micron and nano-samples. While Tables 12 and 13 show that the extra aluminum posses the most influence on the Al_2O_3 crystallite size with 89.49% and 60.04% contribution in micron and nano-samples respectively. Milling time and green density in both group of samples stand on second and third places respectively. The least influence on Al_2O_3 crystallite size belongs to microwave power again.

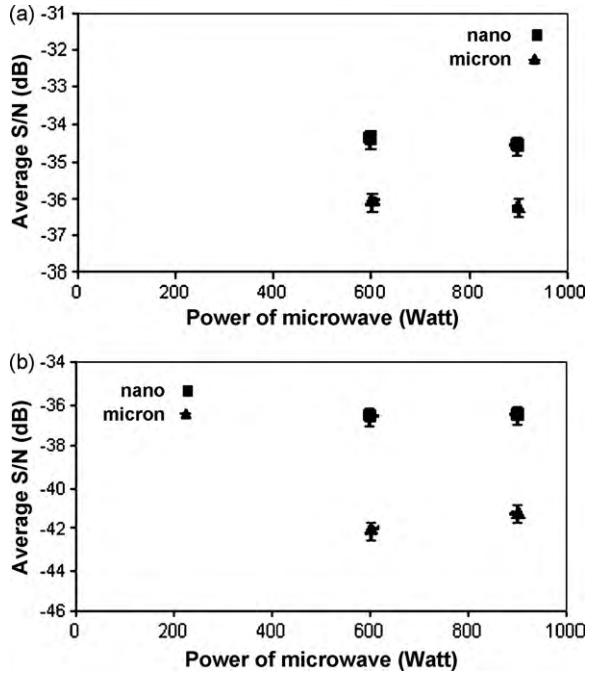

Fig. 8. Effect of power of microwave on (a) TiC and (b) Al_2O_3 crystallite size.

Table 10
ANOVA for S/N ratio of the TiC crystallite size in micron samples.

Factor	DF	SS	MS	ρ (%)
Green density	2	1.84	0.92	6.79
Excess Al	2	2.21	1.10	8.14
Milling time	2	22.96	11.48	84.54
Microwave power	1	0.073	0.073	0.53
Total	8	–	13.57	100

Table 11
ANOVA for S/N ratio of the TiC crystallite size in nano-samples.

Factor	DF	SS	MS	ρ (%)
Green density	2	3.54	1.77	20.30
Excess Al	2	2.59	1.30	14.88
Milling time	2	11.16	5.58	63.98
Microwave power	1	0.073	0.073	0.84
Total	8	–	8.72	100

Table 12
ANOVA for S/N ratio of the Al_2O_3 crystallite size in micron samples.

Factor	DF	SS	MS	ρ (%)
Green density	2	3.29	1.64	2.40
Excess Al	2	122.56	61.28	89.49
Milling time	2	11.08	5.54	8.09
Microwave power	1	0.01	0.01	0.02
Total	8	–	68.47	100

Table 13
ANOVA for S/N ratio of the Al_2O_3 crystallite size in nano-samples.

Factor	DF	SS	MS	ρ (%)
Green density	2	2.34	1.17	11.57
Excess Al	2	12.13	6.07	60.04
Milling time	2	5.72	2.86	28.29
Microwave power	1	0.01	0.01	0.10
Total	8	–	10.11	100

4. Conclusion

In the current study Al_2O_3/TiC composite has been produced using microwave combustion synthesis method. For this purpose Al, C, both nano- and micron TiO_2 was used as raw materials. The results are remarked as below.

(i) Crystallite size of Al_2O_3 and TiC depends on extra aluminum, power of microwave, milling time and green density as well as particle size of TiO_2.
(ii) The presence of nano-TiO_2 in raw materials causes to make finer crystallites of Al_2O_3 and TiC respect to micron TiO_2.
(iii) The shape of Al_2O_3 phase in micron samples is as a particle and in nano-samples is looks like a whiskers.
(iv) 1 mole extra aluminum causes to form the biggest TiC crystallites in both groups of samples.

(v) Milling time had the most influence on the TiC crystallite size in both groups of samples.
(vi) Extra aluminum posed the most influence on the Al_2O_3 crystallite size in both groups of nano- and micron samples.
(vii) Power of microwave had the least influence on the TiC and Al_2O_3 crystallite size in both of micron and nano-samples.

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