Simultaneous synthesis and sintering of TiC/Al₂O₃ composite via self propagating synthesis with direct consolidation technique.

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Abstract

This manuscript presents the results of a study on the use of self propagating high temperature synthesis technique combined with direct consolidation for simultaneous synthesis and densification of TiC/Al₂O₃ composite. The effects of both the mixing procedure of the starting reactant powders, and the addition of different mole fraction of alumina were investigated. The samples were characterized chemically, physically, and morphologically and their mechanical properties measured. It was found that changing the mixing procedure of the starting powders reduced the porosity of the combustion product from 24 to 7 vol%. On the other hand, the addition of 1.0 mol alumina was found to increase the porosity to 13 vol% and hence lower the mechanical properties. However, the addition of alumina resulted in samples with much better geometric definition and therefore this approach should be further investigated with optimized processing conditions.

Keywords: TiC/Al₂O₃ composite; Self propagation high temperature synthesis; Microhardness; Fracture toughness.

1. Introduction

Design and development of advanced materials for high performance applications and deployment of these materials in engineered systems is an important technological challenge.

Refractory ceramic materials (like Al₂O₃ and TiC based ceramics) are candidates for demanding applications, like wear, due to their good mechanical properties and stability at very high temperature [1]. The properties of ceramics can be further improved by making ceramic-ceramic composites. These composite materials possess unique properties designed for specific applications. For example when TiC is combined with Al₂O₃, the composite has better oxidation resistance, and possesses higher fracture strength and fractural toughness than TiC alone without significant decrease in hardness [2]. The improvement in the wear resistance of this composite makes it an attractive material for cutting tools' applications. In addition, the low electrical resistivity of these materials makes it a suitable candidate for electrical discharge machining [3]. As a result, TiC/Al₂O₃ composites have been developed [4]. They have been used as cutting tool in the past decade because of their chemical stability, high strength and hardness, and especially excellent wear resistance [2-4]. In these composites TiC can prevent crack propagation [5], and therefore improve the fracture roughness of Al₂O₃ based ceramics.

However, one of the most important challenges for composite ceramics is their processing. It is well known that in order to get high levels of mechanical performance, the density of the component should be high. The most versatile approach to densify ceramics is pressureless sintering [6]. However, the pressureless sintering of composites is a challenge and results in low

sintered densities [7,8]. As a result, the current methods for manufacturing dense TiC/Al₂O₃ composite include procedures like liquid phase sintering, hot pressing or hot isostatic pressing of a mixture of pure TiC and Al₂O₃. These approaches have many disadvantages. Typically, long processing times are required leading to reaction between TiC and Al₂O₃ which in turn compromises the properties [9]. Liquid phase sintering also leads to **composites** with lower mechanical properties. Finally, techniques like hot pressing and hot isostatic pressing require expensive equipment and consume significant energy due to the requirement of maintaining high **temperature and pressure** for long time. In order to overcome some of these drawbacks, in-situ **reactive processing** with densification represents a potentially attractive approach for producing high density TiC/Al₂O₃ composite. Three different routes can be utilized for reactive processing; combustion synthesis (self propagation high temperature synthesis (SHS)), solid-state displacement reaction and reactive infiltration [3]. Ceramic materials prepared by combustion synthesis (SHS) are reported to be purer and require less energy to be produced than by conventional methods [10]. As a result, it is recognized as one of the emerging **approaches** to synthesize refractory ceramic materials [11-14]. The principal of this technique is that initial reagents, when heated to **suitable temperatures**, spontaneously transform into product due to the exothermic heat of reaction. The combustion synthesis of TiC/Al₂O₃ composite **can be** described as follows: [15]

$$\Delta H_{f,298.15\text{K}}^{\circ} = -1076.6 \text{ kJ/mol}$$

$$\Delta G_{f,298.15\text{K}}^{\circ} = -1038.9 \text{ kJ/mol}$$

The major advantages of this technique are the very high reaction rate and the elimination of the need for high temperature **furnaces that are required** for conventional consolidation processes. However, an inherent limitation of combustion synthesis arises from the high retained porosity in

the synthesized materials. This high porosity is due to the initial voids present in the green compact, the expansion of the compact due to volume change as reactants convert to products, the violent expulsion of gases during the chemical reaction, and limited shrinkage due to rapid cooling of the compact through the ductile-brittle transition temperature (DBTT), (e.g., 800 °C-1200 °C in the case of TiC ceramics) [12]. **As a result, the SHS products cannot be used directly as structural materials because they are generally quite porous.** However, this limitation can be overcome by combining the SHS reaction with a densification step.

Pressureless sintering during combustion synthesis of solid state materials is not sufficient to produce high density products, since the time available at the extremely high SHS temperatures is short. The expulsion of volatile impurities can also lead to entrapped gases and swelling of the product during pressure-less sintering [11]. Coupled with the SHS process, three approaches have been used to form high density materials: (a) simultaneous combustion synthesis and pressure-less sintering of the product, (b) the application of pressure during or shortly after the passage of combustion wave through the reactant green compact, and (c) use of liquid phase in the combustion process [11]. A variety of uniaxial, isostatic, and dynamic pressure application approaches have been utilized in attempts to produce high-density products of SHS reaction synthesis [16-36]. Dynamic densification methods provide the opportunity for pressure application immediately (within seconds) following the SHS reaction and before the products cool below DBTT. In the self propagation high temperature synthesis with dynamic consolidation (SHS/DC) technique, hot, porous, ceramic bodies formed during the SHS reactions are consolidated to high density by the application of stresses using **external load** or by the action of a pressure wave generated from the detonation of a high explosive and/or the impact of an

explosively driven flyer plate. The SHS/DC technique has the merit of being less dependent on expensive facilities and higher in throughput than other techniques [37].

This manuscript is focused on the influence of critical process parameters in the reaction of TiO_2 , C, Al and the consolidation of the resultant TiC/Alumina composite. The parameters that have been investigated are: (a) the mixing procedure the starting reactant precursors; and (b) alumina dilutions. The effect of three mixing procedures of the starting reactant precursors and two different alumina mole fraction on the chemical, physical, and morphological characteristics and the mechanical properties of the synthesized samples has been investigated.

2. Experimental

2.1. Materials

Titanium oxide (TiO_2) powder < 1 μm and 98 % purity supplied by Nice Chemical Pvt. Ltd. India, graphite (C) powder < 5 μm and 98 % purity supplied by Post Apple Scientific, Inc. USA, aluminum metal (Al) powder < 5 μm and 99.5 % purity supplied by GFS Chemicals, Inc. USA and alumina (Al_2O_3) powder < 5 μm and 99.5 % purity supplied by Aloca Co. USA were the starting reactant used in this investigation.

2.2. Procedures

The starting reactant powders were weighed in the required molar ratio according to the following reaction equation (1):

The weighed powders were dry mixed in agate mortar for 15 min then formed into cylindrical pellets (2 cm diameter and 2 cm length) using uniaxial hydraulic press at 78 MPa without binder,

(Model KPD-50E Mega Melchor Gabilondo, Spain). The pressed pellet was transferred to an in-house fabricated combustion reactor. The reactor was evacuated to 10^{-3} bar and the temperature of the green compact was increased using a resistance coil around the compact till it reached the ignition temperature. When the reaction was triggered (the temperature data logger recorded a sudden jump in the temperature), the required load was immediately applied (using motorized hydraulic press). After cooling down, the sample was removed from the reactor and prepared for analysis and testing.

Three different mixing procedures were used to mix the reactant powders prior to compaction. In all mixing procedures stoichiometric amounts of graphite, aluminum and titanium oxide according to reaction equation (Eq. 1) were used. In the first mixing procedure, all of the weighed materials were mixed together in the agate mortar. This procedure is designated as M1. In the second procedure (labeled M2), the graphite powder was first mixed with the titanium oxide powder and then the aluminum was added to this mixture and thoroughly mixed. In the last mixing procedure (M3) the aluminum powder was mixed with the titanium oxide powder and then the graphite was added to the mixture and thoroughly mixed. The effect of additions of alumina (1.0 and 2.0 moles) on the chemical, physical and mechanical properties of the product was also studied. The sample with 1.0 mole alumina addition was assigned as A1 and that with 2 moles alumina as A2. All the compacts were subjected to the SHS reaction with dynamic densification under identical conditions. The sample was preheated at 400°C for 30 minutes. Once the reaction begins (without delay) a compressive stress of 200 MPa was applied for two minutes. The produced samples were machined using a high-speed diamond saw and divided into two halves for characterization.

2.3. Characterization

The phases of the combustion products in the processed samples were identified by X-ray diffraction analysis using X-ray diffractometer (D8 Advanced Bruker AXS, GmbH, Karlsruhe, Germany). Microstructure of specimens was investigated using scanning electron microscope (SEM, Model JSM-5410, JEOL, Tokyo, Japan) equipped with electron dispersive spectroscopy (EDS) and on a field emission SEM (Model JSM-7000F, JEOL, Tokyo, Japan). SEM investigations were conducted on polished sections coated with gold to ensure good electrical conductivity of the entire sample. The sample porosity was measured using Archimedes method. For detailed SEM analysis and micro-hardness measurements a part of the sample was mounted in 32 mm phenolic hot mounting resin such that the largest flat surface of each specimen was exposed. The mounted specimens were ground using a diamond-impregnated pad and polished to a 1 μm finish using diamond suspensions. Vickers micro-hardness analysis was performed on each sample in accordance with the ASTM E384 using commercial micro-hardness testing system (AMH 43 Automatic Micro/Macro-indentation Hardness Testing System, LECO Corporation, Michigan, U.S.). A total of five indentations were performed on each specimen using 1 kg indentation load. The dwell time for each indentation was 13 seconds. The micro-hardness testing system used for this investigation is fully automated and makes use of a 50x microscope to automatically measure indentation dimensions and calculate the micro-hardness. After micro-hardness testing, the microstructures of the specimens were analyzed on a field emission SEM with an accelerating voltage of 15 kV and 10 mm working distance. Secondary electron images were used to assess the level and nature of porosity while backscatter electron images were used to reveal the different phases present in the samples. The chemical constituents of the phases seen under backscatter were determined using EDS. In addition to the micro-

structural and compositional analysis, SEM was also used to analyze the nature of each indent. This analysis was performed in order to assess the presence and extent of micro-cracking extending from the corners of the indentation. Indents that had no micro-cracking were used to calculate the micro-hardness of the specimens while indents accompanied by micro-cracking were used to estimate the fracture toughness.

The fracture toughness, K_{Ic} , of the composite specimens was calculated using Vickers indentation fracture technique described by Fukuhara, et al and used broadly to estimate the fracture toughness of similar ceramic and ceramic composite materials [38]. In this study, this technique was applied to the micro-indentations in which radial cracks were induced. From the Vickers indents, the micro-hardness, H , the indentation diagonal length, $2a$, and micro-crack length, $2c$, are measured. These values are then substituted into equation 2 to yield fracture toughness in units of $\text{MPa.m}^{1/2}$

$$K_{Ic} = \frac{0.203Ha^{1/2}}{(c/a)^{3/2}} \quad (2)$$

The values reported for hardness and toughness are averages of a minimum of five measurements on each sample.

3. Results and Discussion

3.1. Calculation of the maximum possible temperature during combustion synthesis of $\text{TiC/Al}_2\text{O}_3$

Thermodynamic calculations were done to check whether the combustion reaction can proceed in a self-sustaining mode or not. These calculations also give an estimate of the maximum temperature that is reached during combustion synthesis (using adiabatic boundary conditions).

This maximum temperature can be used to postulate if any of the phases have the potential of melting during SHS process.

The adiabatic temperature of a combustion reaction T_{ad} (the maximum achievable combustion temperature) can be calculated for any initial temperature T_o from the following general equation [39]:

$$-\Delta H_{r, T_o} = \int_{T_o}^{T_{ad}} C_p(\text{product}) dT + \mu \Delta H_{f, Al_2O_3} \quad (3)$$

Where, C_p in its general form is represented as [39]:

$$C_p = A + B \cdot 10^{-3} T + C \cdot 10^5 T^{-2} + D \cdot 10^8 T^{-3} \quad \text{Jmol}^{-1}\text{K}^{-1} \quad (4)$$

A, B, C and D are the heat capacity constants of the products which are specific and unique for each material and it could be calculated according to the equation (1).

In case of TiC/Al_2O_3 the values of these constant are as follows: $A = 126.441$, $B = 9.498$, $C = -38.409$, $D = 1.887$ [39]. " μ " is the fraction of molten Al_2O_3 and $\Delta H_{f, Al_2O_3}$ is the enthalpy of fusion of Al_2O_3 and equals to 112.99 KJ/mol [39].

Figure 1 represents the calculated adiabatic temperature of the combustion synthesis of TiC/Al_2O_3 from elemental TiO_2 , Al and C at different initial temperatures. It can be seen that the adiabatic temperature of the reaction at 298K is 2403K. Also, the adiabatic temperature of the reaction increases linearly with increasing initial temperature. The linear relation between T_{ad} and T_o is well documented in the literature [40]. These calculations indicate that the value of T_{ad} is higher than that of the empirical value 1800K [41] which means that the reaction between TiO_2 , Al and C can proceed in a self-sustaining manner. Also it is higher than the melting point of Al_2O_3 (2323K) which means that under the assumptions of this calculation (adiabatic boundary conditions), the alumina phase will be in the molten state during synthesis. However,

the adiabatic temperature is much lower than the melting point of TiC (3100K) which means that TiC will be in the solid state.

3.2. Preliminary combustion synthesis of TiC/Al₂O₃ composite

The first set of experiments was conducted under argon atmosphere without applying external load. The goal of these experiments was identification of the process parameters that result in the desired phases. Mixing procedure M3 was used for thoroughly mixing the reactant powders. Thermal explosion mode was used in which the temperature of the green compact is increased continuously until the ignition temperature (910°C) is reached.

The XRD pattern of the combustion product, Fig. 2, reveals the existence of very sharp lines corresponding to the diffraction pattern of titanium carbide and aluminum oxide. No other peaks are detected, suggesting that the reaction between the reactants, TiO₂, Al and C using their stoichiometric values, has a high degree of conversion to the products under the conditions used.

It is worth noting that the sample was scaly and fragmented. The measured porosity revealed that the sample is highly porous (~ 48 % porosity) and contains many micro, macro and radial cracks as can be seen from the optical micrograph, Fig. 3a. Scanning electron micrograph of TiC/Al₂O₃ composite (shown in Fig. 3b) reveals the existence of two different phases one is dark grey and the other is white. Energy dispersive spectroscopy (EDS) of the dark grey phase confirmed that this phase corresponds to Al₂O₃ (Fig. 3c) and the white phase corresponds to titanium carbide (Fig. 3d). It can be seen that the grains of the dark grey phase are sintered together into large grains and some of alumina precipitates are also present in the TiC phase. This is in accordance with the thermodynamic calculations which suggested that alumina will melt during synthesis.

From the above results and literature, it was concluded that the combustion reaction in the TiO_2 –Al–C system was composed of the aluminothermic reduction of TiO_2 by molten aluminum leading to the formation of alumina and molten Ti. The molten Ti then reacts with carbon to produce TiC. These reactions occurred in series of steps leading to the production of the TiC/ Al_2O_3 composite. This conclusion is in good agreement with the study of Cho et al [42]. They reported that in such system the aluminothermic reduction was initially activated by the wet spreading of the Al melt into the interspaces between the TiO_2 particles, formation of the intermediate phases of oxygen-deficient titanium oxides and titanium aluminides, and finally the formation of Al_2O_3 grains and Ti-rich liquid phase. The Al_2O_3 grains nucleated and grew in the Al melt leading to a liquid that progressively becomes Ti-rich. In the later stages and at higher temperature, the Ti-rich phase reacted with the C particles to produce TiC grains in the Ti-rich liquid phase.

3.3. Investigation of the SHS/DC process

3.3.1. Effect of the mixing procedure

The XRD patterns of the three compacts (produced from the three different mixing procedures) are shown in Fig. 4. The patterns clearly show that for M1 compact, the reaction is not complete. In addition to the target phases (TiC and Al_2O_3) a small quantity of graphite is also present. There are no peaks corresponding to TiO_2 and Al (which should be present due to the incomplete reaction. This implies that the amount of these unreacted phases is rather small (less than 1-2%) [43]. For samples made using the M2 and the M3 mixing process, the only phases seen in XRD are TiC and Al_2O_3 indicating that the reaction is complete for these two samples.

The porosity of the three compacts was measured and is tabulated in Table 1. It was found that M1 compact shows the highest porosity while the M3 compact is the lowest porosity sample. The high porosity of the M1 sample could be due to the fact that the reaction is not complete for this mixing procedure. The shape of the produced samples with different mixing procedures is shown in Fig. 5. It was found that the samples produced using mixing procedures M1 and M2 were fragmented during machining. While sample produced with mixing procedure M3 remained intact. Most likely, this is due to the lower density of samples made using M1 and M2 mixing procedure.

The high density of M3 sample is attributed to the fact that the combustion wave velocity and the resulting increase in the temperature depends on the TiO_2/Al contact area [44]. As discussed earlier in this manuscript, the reaction in this system proceeds via first aluminothermic reduction of TiO_2 leading to formation of Ti metal, which subsequently reacts with C leading to the formation of TiC. For this set of reactions to occur rapidly, it is important to have high number of TiO_2/Al as well as TiO_2/C contacts. Increasing the contact area between TiO_2 and Al particles facilitates and accelerates the aluminothermic reduction of TiO_2 . Accordingly the sample reaches a maximum temperature in a short time after the combustion begins. With the fact that the external load is applied after the reaction begins, in this system the compaction is applied at a higher temperature. For this reason the compact produced with the M3 mixing procedure shows the highest density. For the M1 compact the mixing of all the reactants together resulted in arbitrarily random number of contacts between different reactants. As a result, the reaction is not complete and the density is lower. Based on this hypothesis, the density of the M2 compacts should have the lowest density because in this case, graphite is first mixed with TiO_2 . This should result in the lowest probability of Al and TiO_2 contact. In addition, carbon would isolate

Al and TiO_2 particles, and replace effective TiO_2/Al interface by ineffective Al/C ones [43]. However, this compact was denser than one produced with M1 procedure. A plausible explanation for this observation is that at the combustion temperature, Al is molten and easily able to migrate leading to an important contribution from the TiO_2/C interfaces [45]. In case of M2 sample the complete reaction between the starting reactants (as dictated from the XRD pattern (Fig. 4) means that there is enough contact area between TiO_2 and molten Al for sustaining the reaction. The density observations from the Archimedes method were confirmed by SEM observations (Figure 6). This figure clearly shows that the number and size of pores is lowest for the M3 and highest for the M1 mixing procedure. Only two phases appear in all the micrographs: white color corresponding to TiC and dark grey corresponding to Al_2O_3 .

It has been documented out that fracture toughness and hardness are the most important mechanical properties for the wear resistance of brittle materials ceramics [46-48]. Therefore, both hardness and fracture toughness of the sample produced with M3 mixing procedure were measured. The sample was found to possess 6.35 GPa hardness and $3.5 \text{ MPa}\cdot\text{m}^{1/2}$ fracture toughness (Table 2 to be discussed later).

Although, this sample has reasonable fracture toughness, its micro-hardness is lower compared to the reported values (19.2-22 GPa) [49-51]. The difference between the reported micro-hardness values and the ones for this sample are due to the synthesis technique and the final compact density [52]. It has been reported that weak grain boundaries lead to low values of the measured micro-hardness; weak grain boundaries allow easier displacement of grains during hardness testing [53]. It has been suggested that samples processed by the SHS process have weaker grain boundaries because the SHS/DC process is very rapid; consequently, there may not be sufficient time for the atoms to rearrange to produce strong bonds between grains [37]. In

addition, the rapid cooling of the outer surface compared to the inner core could cause extensive stresses or micro-cracks along the grain boundaries. Finally, the higher porosity in the SHS samples also contributes to lower the micro-hardness of the $\text{Al}_2\text{O}_3/\text{TiC}$ composite produced in this study. Another important observation is that although for the samples made from the M3 process, the density is reasonable, the shape of the pellet is highly distorted (Figure 5). This is an indication of the excessive melting leading to a highly deformable sample. One possible approach to reduce the reaction rate, which may lead to stronger boundaries, is to add alumina as an inert dilutant. This was investigated and the results are reported below.

3.3.2. Effect of Alumina dilution

Two different mole fractions of alumina (1 and 2 moles) were mixed with the starting materials before the SHS/DC reaction, to study the effect of alumina dilution on the properties of the composites. The modified mixing procedure M3 was used. First aluminum was mixed with titanium oxide powder, followed by graphite addition, then finally the extra alumina powder was added and all reaction constituents thoroughly mixed.

The overall reaction in this case will proceed according to equation (5).

The other conditions for SHS/DC reaction were kept identical to the ones for the samples without excess alumina.

The shape of the combusted samples with different alumina mole fraction after machining is shown in Fig. 7. It should be noted that these sample maintain their shape much better than samples without alumina dilution. The sample produced with 1 mole alumina addition (A1) seems to be denser than that produced with 2 moles (A2). Also, A2 sample seems to contain

some white regions which may be an indication of incomplete reaction between the reactants. The total porosity of these samples was measured and tabulated in Table. 1. It is clear that increasing alumina addition leads to an increase in the total porosity of the **processed** composites. Backscattered SEM analysis was performed on the composites to investigate in more details the influence of alumina dilution on the sample microstructure (Fig. 8). It was noticed that there is a significant difference between the two samples. The sample with 2 mole excess alumina has a higher porosity and secondary phases. EDX analysis indicates that for sample with 1 mole fraction only TiC (A) and Al_2O_3 (B) phases together with pores (C) are presents. While for sample with 2 mole fraction **in addition to** Al_2O_3 (A) and TiC (C) a significant amounts of TiO_2 (B), Al metal (D) and a large number of pores (E) are present in the sample. This observation confirmed that increasing the alumina dilution leads to a large increase in the porosity and incomplete reaction. To further confirm this observation XRD analysis of these samples was performed (Fig. 9). The patterns revealed that with alumina dilution by 1 mole fraction the composite sample is almost entirely composed of TiC/ Al_2O_3 . While for the 2 mole excess alumina, the main phase is the Al_2O_3 (which is reasonable with excess alumina addition). In addition, a significant amount of unreacted phases (Al metal and TiO_2) are present as well as small amounts of TiC. Thus it is clear that at this level of excess alumina, the reaction mixture is **too** dilute and the temperature never reaches **high enough level to complete the reaction**. Due to the lower temperature, the densification is also not complete leading to significantly higher level of porosity. Due to limited reaction, the amount of liquid phase may also be limited leading to higher levels of residual stresses. **Finally, the** addition of high fraction of alumina resulted in an incomplete reaction because the rate at which the Al liquid phase comes in contact with the TiO_2 particles is impeded by the presence of Al_2O_3 particles [54].

Both micro-hardness and fracture toughness of all the samples are given in Table 2. It can be seen that the addition of alumina leads to lower hardness and fracture toughness. This observation is attributed to both higher porosity and incomplete reactions for samples with excess alumina. Thus addition of excess alumina improves the shape retention of the composite (Figure 7). However, the porosity increases leading to inferior mechanical properties.

4. Conclusion

Titanium carbide/alumina composite were successfully synthesized and sintered simultaneously using self propagation high temperature synthesis combined with direct pressure assisted consolidation. The sequence of mixing the starting reactant powders was found to be important for completion of reaction, the amount of residual porosity and the measured mechanical properties of the composite. The best mixing procedure was M3 in which the aluminum powder was mixed with the titanium oxide powder and then the graphite was added to the mixture and thoroughly mixed. It is postulated that this procedure leads the highest level of contact area between TiO_2 and Al particles which facilitates and accelerates the aluminothermic reduction of TiO_2 (the first step in the total reaction). One mole fraction of alumina addition was found to lead to complete reaction and a significantly better shape retention. However, the density of this sample was lower than that of sample without excess alumina. This resulted in lower hardness and toughness. Increasing the excess alumina content to 2 mole fractions lead to further retardation of densification and incomplete reaction. One possible avenue for future investigation is further optimization of the amount of dilutant and optimizing the processing conditions for samples with dilutant (e.g. higher compressive stresses during consolidation). Another possible

avenue is use of TiC or ductile metals as the dilutant. Both these approaches are under investigation.

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Table 1: The effect of different **mixing procedures** and different alumina mole fraction additions on the open porosity of SHS/DC products

Table 2: The effect of alumina mole fraction addition on the Vickers micro-hardness and fracture toughness of the composite samples

Fig. 1 Dependence of adiabatic temperature of $\text{TiC}/\text{Al}_2\text{O}_3$ on the initial temperature of the reactants

Fig. 2 XRD of $\text{TiC}/\text{Al}_2\text{O}_3$ composite product synthesized in thermal explosion mode (no load)

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Fig. 7 Photographs of composite samples produced with different alumina mole fractions addition after machining: (A1) 1 mole and (A2) 2 mole

Fig. 8 Backscattered SEM micrographs for composite samples produced with the addition of different Al_2O_3 mole fractions: (a) 1 mole and (b) 2 mole

Fig. 9 XRD patterns of composite samples produced with different amount of alumina additions

Fig. 1: Dependence of adiabatic temperature of TiC/Al₂O₃ on the initial temperature of the reactants

Fig. 2: XRD of TiC/Al₂O₃ composite product synthesized in thermal explosion mode (no load)

Fig. 3 Micrographs of SHS produced samples; [a] Optical, [b] SEM, [c] EDX of white grains and [d] EDX of dark gray grains

Fig. 4: XRD patterns of SHS/DC samples produced using different mixing procedures

Fig. 5: Photographs of SHS/DC samples produced using different mixing procedures

Fig. 6: Scanning electron micrographs for composite samples produced with different mixing procedures at both low and high magnifications; (a) and (b) are for MI sample, (c) and (d) are for M2 sample, and (e) and (f) are for M3 sample

**Fig. 7: Photographs of composite samples produced with different alumina mole fractions
addition after machining: (A1) 1 mole and (A2) 2 mole**

Fig. 8 Backscattered SEM micrographs for composite samples produced with the addition of different Al_2O_3 mole fractions: (a) 1 mole and (b) 2 mole

Fig. 9: XRD patterns of composite samples produced with different amount of alumina additions

Table 1: The effect of different mixing procedures and different alumina mole fraction additions on the open porosity of SHS/DC products

Sample type	M1	M2	M3	A1	A2
Open porosity, Vol%	24.20	18.13	7.00	13.07	18.61

Table 2: The effect of different alumina mole fraction additions on the Vickers micro-hardness and fracture toughness of the composite samples

Sample type	Vickers micro-hardness, GPa	Fracture Toughness, MPa.m ^{1/2}
Sample without alumina addition	6.35	3.5
Sample with 1 mole alumina addition	5.00	3.2
Sample with 2 mole alumina addition	1.56	Nd*

*Nd: Not determined due to high level of porosity