

Mechanical properties of $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites prepared by directed oxidation of an aluminum alloy

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Abstract

Silicon carbide particulate reinforced alumina matrix composites were fabricated using Directed Metal OXidation (DIMOX) process. Continuous oxidation of an Al–Si–Mg–Zn alloy with appropriate dopants along with a preform of silicon carbide has led to the formation of alumina matrix surrounding silicon carbide particulates. $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites fabricated by the DIMOX process, possess enhanced mechanical properties such as flexural strength, fracture toughness and wear resistance, all at an affordable cost of fabrication. $\text{SiC}_p/\text{Al}_2\text{O}_3$ matrix composites were investigated for mechanical properties such as flexural strength, fracture toughness and hardness; the composite specimens were evaluated using standard procedures recommended by the ASTM. The $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites with SiC volume fractions from 0.35 to 0.43 were found to possess average bend strength in range 158–230 MPa and fracture toughness was found to be in range of 5.61–4.01 $\text{MPa}\sqrt{\text{m}}$. The specimen fractured under three-point loading as observed under scanning electron microscope was found to fail in brittle manner being the dominant mode. Further the composites were found to possess lower levels of porosity, among those prepared by DIMOX process.

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1. Introduction

Ceramics have excellent strength-to-weight ratio when compared to advanced metals and alloys. These attractive properties can also be maintained to extremely high temperature, which make them a sole choice for high temperature applications. A variety of structural applications are possible for ceramic materials ranging from high temperature gas turbines and adiabatic diesel engines to cutting tools and other wear-resistant parts. In each of the said applications, beneficial properties of ceramics such as high stiffness, strength and hardness, low density, and good resistance to corrosion, oxidation, and wear at high temperatures, have been explored. With the ever-increasing performance requirements of engineering materials,

the properties of monolithic materials are pushed to their limits. Monolithic ceramics possess high strength but lack the fracture toughness, required in many applications, such as components in jet engines. Ceramic materials have properties that make them ideal candidates for many elevated temperature applications such as heat exchangers and turbine engines components. Due to the refractory nature of ceramics, they are, at times, the only choice for a material that can potentially satisfy the most demanding requirements particularly at high temperatures. In addition to high melting or decomposition temperatures, many ceramics possess other attractive features such as low density, high temperature strength, high hardness and resistance to creep deformation, thermochemical stability and lack of reactivity in contact with other materials and various atmospheres, and last, but not least, high wear resistance.

However, the most important disadvantage of ceramic materials is their relatively low fracture toughness, which is the resistance to the propagation of a fine crack [1]; the lack of toughness renders them sensitive to sudden catastrophic failure

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in response to accidental overloading, contact damage, or rapid temperature changes. In order to overcome the limitation on fracture toughness, much emphasis was laid on the processing methods involved in the ceramic composite production. Many methods and their variants have been suggested as means to enhance fracture toughness in ceramic materials [2,3]. These include control of grain size, porosity, or other microstructural features [4,5], and transformation toughening in tetragonal ZrO_2 -based ceramics [6], incorporation of metallic and/or ceramic fibers and or whiskers [7,8]; fracture toughness of ceramic materials can be improved by the incorporation of second phases containing metallic reinforcement that possess high magnitudes of toughness. Addition of second phase particles, both ductile and brittle, not only affects the microstructural features but also mechanical properties [9]. Second-phase particulates can play a number of roles. They can deflect cracks out of their paths, cause them to bow between obstacles, cause them to bifurcate, or cause the nucleation of additional microcracks ahead of the primary crack [10].

Among various methods for the preparation of ceramic matrix composites, the directed metal oxidation process developed by the Lanxide Corporation has attracted considerable interest. The process has the potential to form a three dimensional network of Al alloy which is the source for the formation of Al_2O_3 matrix. The process involves oxidation of molten Al alloy into a preform, accompanied by wicking action in a cyclic manner. Appropriately tailored processing schedule for directed metal oxidation process, ensures a three dimensional network of Al alloy ranging between 5 and 30 vol.%. Some of the composite systems developed using the Lanxide process are $\text{Al}_2\text{O}_3/\text{Al}$, AlN/Al , ZrN/Zr , and $\text{ZrB}_2/\text{ZrC}_x/\text{Zr}$ composites [11–13]. Among the most vigorously studied systems has been the $\text{Al}_2\text{O}_3/\text{Al}$ system for the formation of $\text{SiC}/\text{Al}_2\text{O}_3$ –Al ceramic composite. The process enables fabrication of composites with uniform microstructure through continuous wicking of molten Al alloy towards the oxidation front. Specific dopants (e.g., Mg combined with Group IV elements such as Si) provided in the composite system prior to oxidation, were found to affect the growth conditions [13].

Many works have been reported on enhancement of strength and toughness of ceramic composites. However, any work in this context was found to be focused at specific volume fraction of SiC that were relevant to industrial applications. Further, a systematic effort to understand the practical role of SiC particulate volume fraction on these mechanical properties is absent. In the present study, it is aimed to understand the effect of SiC particulates volume fraction on the mechanical properties, namely flexural strength, fracture toughness, compressive strength and hardness of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites.

2. Experimental details

2.1. Fabrication of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composite

In the present work, $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites with different volume fractions were prepared by directed metal oxidation process. This was comprised of two steps namely preparation of

Table 1

Details of volume fractions of composites studied.

| Label | Grit size | Average particle size (μm) | Tapped packing density (kg/m^3) | Volume fraction |
|-------|-----------|---|---|-----------------|
| B1 | #100 | 125 | 1118 | 0.35 |
| B2 | #120 | 106 | 1286 | 0.40 |
| B3 | #220 | 53 | 1382 | 0.43 |

SiC preforms with different volume fractions and appropriate heat treatment schedule to aid formation of Al_2O_3 matrix. The volume fraction of SiC was varied by using SiC particulates of different grit sizes namely #100, #120 and #220 procured from Carborundum Universal, India. The corresponding particle size, tapped packing density and volume fraction, as calculated using density of α -SiC ($3197 \text{ kg}/\text{m}^3$) are detailed in Table 1. SiC powders were subjected to heat treatment in air at 1100°C for 4 h to develop an adherent coating of SiO_2 on the surface of SiC particles. This treatment is known develop a thickness of $\sim 120 \text{ nm}$ of SiO_2 [14]. This is an important step in the processing of composite materials involving Al-alloys. Molten Al-alloy reacts with SiC to form Al_4C_3 causing deterioration of SiC phase. Further, the reaction product when comes in contact with moisture results in the formation of $\text{Al}(\text{OH})_3$ and methane gas [14]. Pre-oxidation of SiC would help protect SiC from degradation, by forming and adherent layer of SiO_2 . Subsequently, loose powder preforms (measuring $70 \times 70 \times 20$, in mm) of oxidized SiC, with corresponding tapped packing densities, were contained in refractory crucibles.

Al alloy ($\text{Al}-9\text{Zn}-8.5\text{Si}-1.5\text{Mg}$) blocks were used as source for formation of Al_2O_3 . The Al alloy block was ground and polished to good surface finish and cleaned with acetone. Gypsum was coated on five sides of the block to prevent growth into sides and the top; the bottom side of the Al block was coated with a mixture of Bi_2O_3 and SnO_2 to serve as growth promoter compound. A schematic of the experimental set-up is shown in Fig. 1. The loose powder preforms were rested on bed of Zircon sand in order to ensure adequate supply of oxygen at surface of Al-alloy that is in contact with perform. The samples were heated to a temperature in the range of 950 – 980°C in an atmosphere of oxygen for a dwell time of 65 h followed by furnace cooling to room temperature.

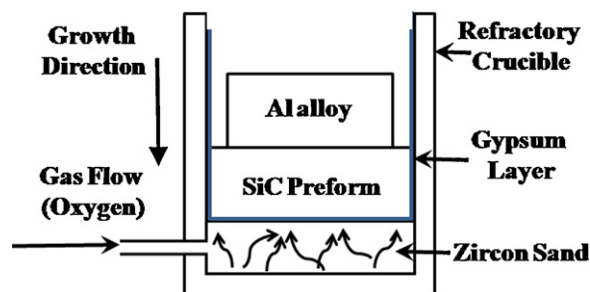


Fig. 1. Schematic view of set-up used in DIMOX process for fabrication of $\text{SiC}_p/\text{Al}_2\text{O}_3$.

2.2. Characterization of SiC_p/Al₂O₃ composites

2.2.1. Microstructure

Detailed characterization of microstructure formed by continuous oxidation process is a vital part in the understanding of mechanical behavior of SiC_p/Al₂O₃ ceramic matrix composites. Small sections from composites having different volume fractions of SiC were sectioned using a low speed cutter employed with a thin diamond blade. The cut sections were polished according to standard metallographic methods using diamond abrasives down to a size of 1/4 μm. Prior to microstructural observations, a thin layer of gold was coated on the specimens by using a RF sputtering unit. The polished sections were examined under SEM in order to study distribution of phases and associated porosity. ESEM from FEI (Model: Quanta 400) with a FEG was used in SE mode to acquire high quality microstructures at an acceleration voltage of 20 kV. Various phases present in the microstructure were quantified using image analyzer software – Bio-Vis Materials Plus version 1.5, based on point count method.

2.2.2. Mechanical properties

Mechanical properties were measured on test specimens machined from as fabricated blocks of ceramic matrix composites. A high speed diamond saw was used to machine specimens according to prescribed dimensions for different mechanical properties: flexural strength, fracture toughness, compressive strength as per ASTM standards. The surfaces were ground parallel and polished using a diamond disc in order to minimize surface damage. All specimens were tested on a universal testing machine, INSTRON-5500R with a capacity of 100 kN. Load–displacement and stress–strain curves were obtained with help of an in-built software Instron Materials Tester ver.9.0. The testing of specimens was performed at room temperature (23 °C) and constant rate of 0.2 mm/min.

2.2.2.1. Flexural strength. The ceramic composites with dimensions 50 × 8 × 8, in mm and chamfered edges at 45° up to 0.1 ± 0.03 mm were used for the measurement of the flexural strength under 3-point bending load, according to ASTM C1161-94 [15]. A span length of 40 mm was used for all tests. The composites were deflected until rupture occurred in the outer face of the specimen. Three specimens were tested for each volume fraction. The flexural strength (σ) was determined from the following relation, for a rectangular specimen loaded in three-point bend set-up.

$$\sigma = \frac{3FL}{2bd^2} \quad (1)$$

where F is the maximum load to cause fracture, L is the support span, b is the width of the specimen and d is the thickness of the specimen. Results of flexural strength measurements are discussed in comparison with those reported in the literature on Al₂O₃ matrix composites.

2.2.2.2. Fracture toughness. Fracture toughness is ability of material to resist fracture in the presence of a crack. Stress-

intensity factor (K) is a quantitative parameter of fracture toughness determining a maximum value of stress which may be applied to a specimen containing a crack (notch) of a certain length. The test is performed according to ASTM E 399-90 [16]. The composites with dimensions 50 × 8 × 8 in mm were used for the measurement of the fracture toughness by single edge notch beam technique. A notch was machined on one side across the length at the mid-point of the specimen. A span length of 40 mm was used for all tests. The fracture toughness (K_Q) was determined using the following relation.

$$K_Q = \left(\frac{P_Q S}{BW^{3/2}} \right) \cdot f\left(\frac{a}{W}\right) \quad (2)$$

where K_Q is the fracture toughness in MPa√m; P_Q is the load in kN, as determined from ASTM E-399-90; B is the Specimen thickness in cm; S is the span of specimen in cm; W is the Specimen depth (width) in cm; a is the crack length in cm.

The crack length, ' a ' was aimed such that $a/W = 0.5$. However, $f(a/W)$ was computed based on the average value obtained from measurements at the center and edges of the notch subsequent to fracture. The average of these three measurements is used as the crack length to calculate K_Q . Three samples were tested for each volume fraction and the average value was considered for subsequent purposes.

2.2.2.3. Compressive strength. The composites with different SiC volume fractions were tested for uniaxial compressive strength. Specimens with dimensions measuring 6 mm in diameter and 9 mm in length were used for the measurement of the compressive strength according to ASTM standard test method D3410M-95 [17]. The maximum stress and corresponding maximum strain are calculated for every load value. Three samples were tested for each volume fraction and the average value was considered. The data for compressive strength was compared with experimental results available in the literature.

2.2.2.4. Hardness. The hardness of the ceramic matrix composites was evaluated over the Vickers Scale as per ASTM standard E92 [18] using a Vickers hardness tester (model: 270 VRSD, system AFFRI). Tests were conducted at an applied load of 30 kg for a dwell time of 10 s. Prior to indentation, the test specimens were ground flat and polished to a good surface finish down to 1/4 μm. The Vickers hardness number VHN is given by

$$VHN = \left(\frac{2F}{d^2} \right) \cdot \sin\left(\frac{136^\circ}{2}\right) = 1.854 \times \frac{F}{d^2} \quad (3)$$

where ' d ' is the mean diagonal [$d = (d_1 + d_2)/2$] in mm and ' F ' is load in kg. The data for the Vickers was taken from the average of three measurements. The data for hardness was compared with experimental results available in the literature.

3. Results and discussion

3.1. Fabrication of SiC_p/Al₂O₃ composites

In the present work, it was aimed to study the mechanical properties of Al₂O₃ matrix composites containing different



Fig. 2. $\text{SiC}_p/\text{Al}_2\text{O}_3$ composite by directed metal oxidation process ($70 \times 70 \times 20$, in mm).

volume fractions of SiC. A sample of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composite in its as-prepared condition is shown in Fig. 2. The composites had grown almost to the dimensions of the refractory container used. Moreover, the dimensions of the composite material fabricated by the DIMOX process employed in the present

work are large enough for detailed characterization of various mechanical properties. The growth of composite was found to be complete with a hollow left in the metal reservoir. Thus, it can be said that the processing schedule employed in the present work was found to be successful in the fabrication of bulk $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites. Further, for measurements of flexural strength, fracture toughness and compressive strength, $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites were machined into required dimensions as briefed in an earlier section.

3.2. Characterization of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites

3.2.1. Microstructure

Microstructural studies were carried out in order to understand mechanical behavior of the ceramic composites reinforced formed by directed metal oxidation. Typical microstructures of the composites with different volume fractions of SiC are shown in Fig. 3. The microstructures of the composites indicated a nearly homogenous distribution of SiC in a composite matrix. The matrix microstructure comprised of Al alloy embedded in Al_2O_3 and associated porosity. Essentially, the composite

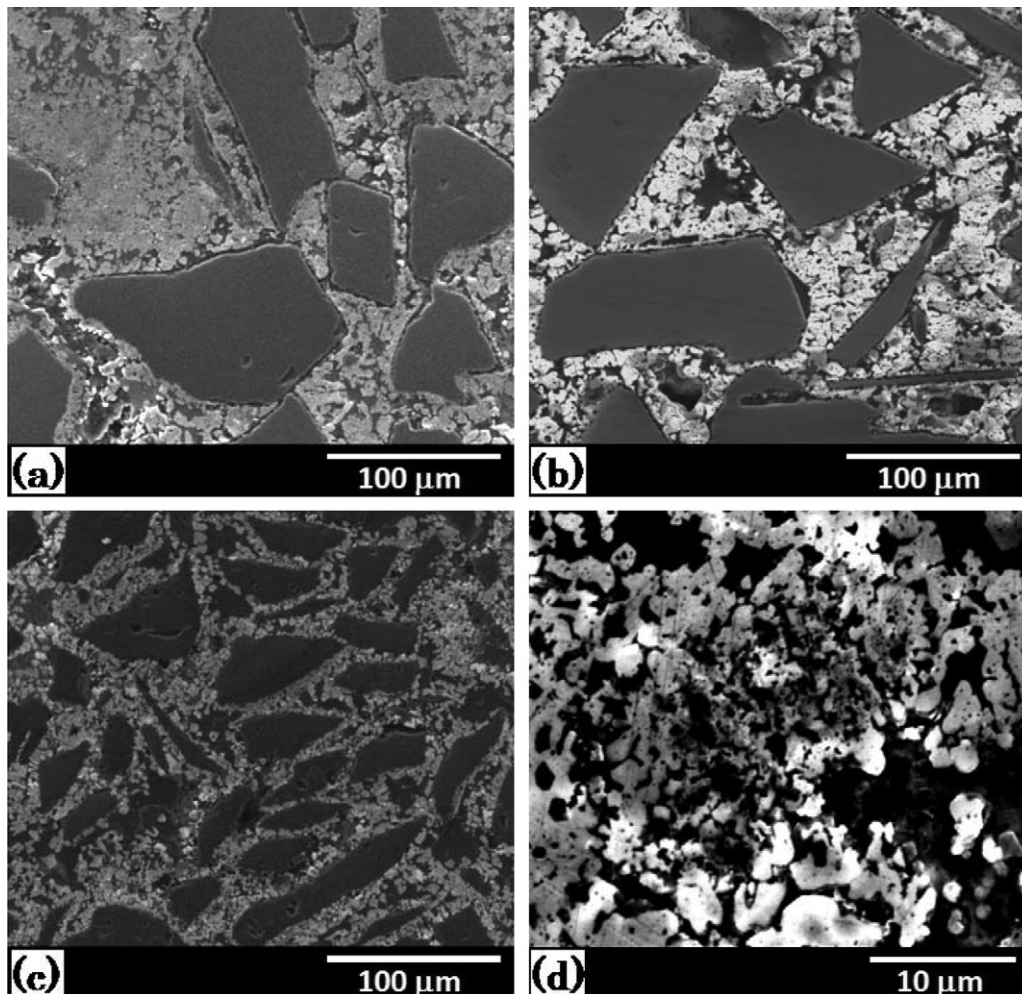


Fig. 3. Typical microstructures of $\text{SiC}_p/\text{Al}_2\text{O}_3$ CMC showing nearly homogenous distribution of SiC particulates at different volume fractions. (a) 0.35, (b) 0.40, (c) 0.43 and (d) Al_2O_3 matrix formed by DIMOX process.

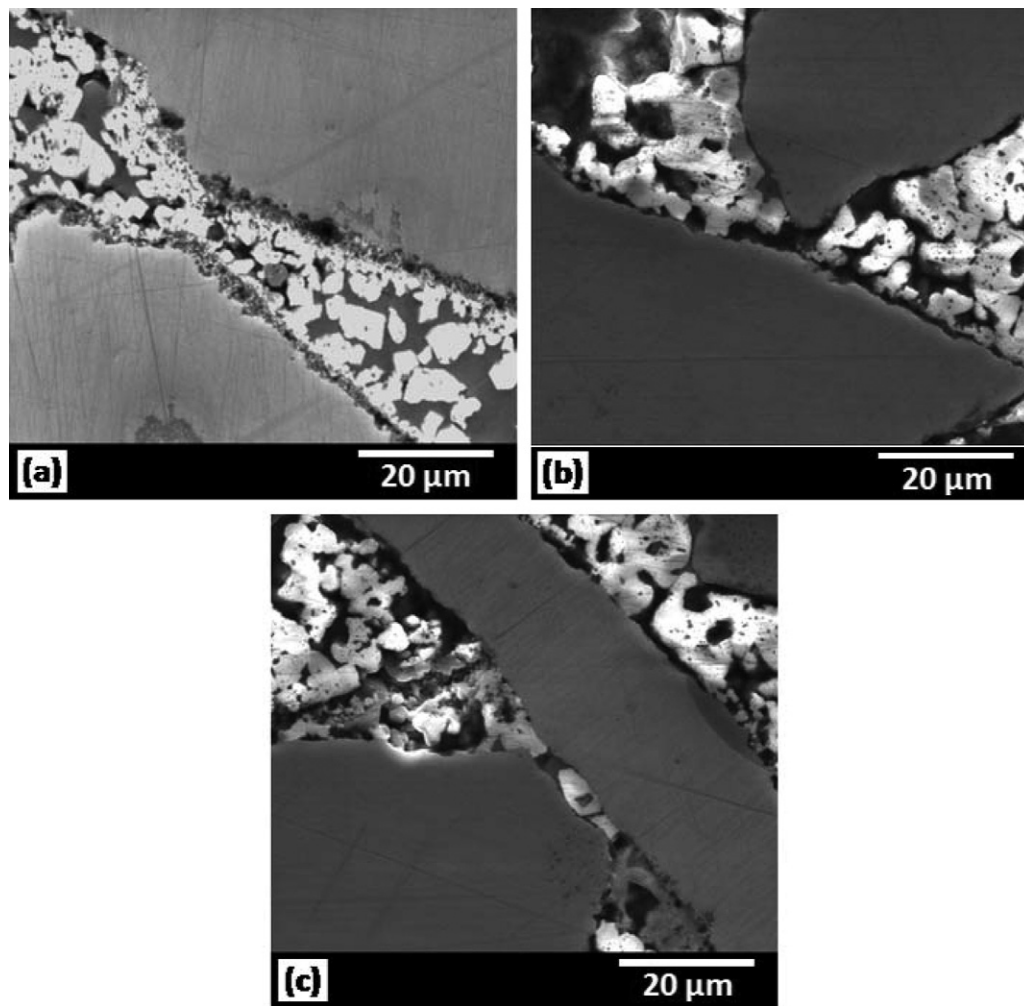


Fig. 4. Microstructures of the composites at high magnification giving an insight into the degree of interfacial bonding between SiC particulate and Al_2O_3 matrix.

microstructure is made up of fine channels of Al alloy and porosity apart from principal phases of SiC particulates and Al_2O_3 as a continuous matrix. Some of the other features that could be noticed from the microstructures are presence of few SiC particulates that are highly acicular and many SiC particulates contained intrinsic porosity. Finer details of microstructures such as bonding at the interface obtained at high magnification are shown in Fig. 4. Mechanical behavior of materials is greatly affected by the presence of porosity that is either formed as a result of processing or a pre-existing feature of raw material. Besides porosity, volume fractions of the

constituent phases affect mechanical properties to a great extent. Thus a realistic measure of constituent phase fraction is highly necessary.

Results of quantitative metallography using image analysis software are shown in Table 2. A good correlation could be observed between SiC volume fraction measured from tapped packing density and quantitative metallographic results. Another observation is that the volume fraction of Al_2O_3 formed by directed metal oxidation process is not affected by SiC volume fraction. This could be understood by the very fact that the process parameters are the same for all the composites studied here. Apart from the principal phases the microstructure consisted of Al alloy and porosity. The volume fraction of Al alloy increased monotonously from 0.0193 to 0.0487 for SiC volume fraction between 0.43 and 0.35, respectively. Finally, the volume fraction of pores did not show any kind of dependence on SiC volume fraction indicating that the average porosity in directed metal oxidation process is $\sim 9.53\%$ by volume. This is in close tolerance with the porosity data obtained from Archimedes principle, 11%. However, the summation of all phases' volume fraction did not tally to unity that may be attributed to small percentage of error involved in sampling and method of measurement.

Table 2
Results of quantitative metallography showing volume fractions of SiC, Al_2O_3 , Al alloy and porosity.

| Label | Volume fraction of SiC | Volume fraction by image analyses | | | |
|-------|------------------------|-----------------------------------|-------------------------|----------|----------|
| | | SiC | Al_2O_3 | Al alloy | Porosity |
| B1 | 0.35 | 0.3607 | 0.4451 | 0.0487 | 0.1043 |
| B2 | 0.40 | 0.4125 | 0.4296 | 0.0284 | 0.0874 |
| B3 | 0.43 | 0.4358 | 0.4376 | 0.0193 | 0.0943 |

The composites were further examined at high magnification to obtain an insight into the interfacial bonding between the particulate and the matrix formed by directed oxidation of Al–Si–Mg–Zn alloy. The microstructures showed that the contiguity in the interfacial bond is limited and this improved with SiC volume fraction. However, a precise measure of contiguity in the interfacial bond is a complex task as it involves measurement of surface area that is in contact with the matrix phase.

3.2.2. Mechanical properties

The motive of this work was to present successful fabrication of SiC reinforced Al_2O_3 CMC formed by directed metal oxidation process and followed by preliminary evaluation of mechanical properties of prior importance, namely – flexural strength, fracture toughness, compressive strength and hardness. Thereby have a relative measure of the potential of various processing parameters reflected in terms of overall mechanical properties. In this study, the range of particle size is between $\sim 125\ \mu\text{m}$ and $\sim 53\ \mu\text{m}$ which is very narrow. It may be noted that properties may vary with respect to particle's size at a very different length scales, e.g. micro to nano scale. However, in the present case as the range of particle size is quite narrow, the expected variation in the respective physical properties will be negligible. Within a small percentage of error, changes in mechanical properties are attributed to differences in SiC volume fraction.

3.2.2.1. Flexural strength. $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites were evaluated for flexural strength at room temperature. Load vs. displacement curves for SiC particulate reinforced Al_2O_3 matrix composites with volume fraction, V_f : 0.35–0.43 are shown in Fig. 5. Details of flexural strength of $\text{SiC}_p/\text{Al}_2\text{O}_3$ are given in Table 3. Fig. 6 shows the gradual increase in flexural strength of the Al_2O_3 matrix composites as a function of SiC volume fraction. The flexural strength of SiC particulate reinforced Al_2O_3 matrix composites fabricated in the present work was found to be in the range of 158–231 MPa for SiC volume fractions in the range of 0.35–0.43, respectively. The

Table 3

Mechanical properties of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites.

| Label | Volume fraction | Flexural strength (MPa) | Fracture toughness ($\text{MPa}\sqrt{\text{m}}$) | Vickers hardness | |
|-------|-----------------|-------------------------|--|------------------|-------|
| | | | | HV | GPa |
| B1 | 0.35 | 157.65 | 5.61 | 884.85 | 8.84 |
| B2 | 0.40 | 161.00 | 4.64 | 785.00 | 8.25 |
| B3 | 0.43 | 230.60 | 4.01 | 903.00 | 8.92 |
| B4 | 0.48 | – | – | 1296.78 | 12.85 |

strength levels observed were not appreciably high but are adequate for a number of applications. The highest flexural strength was found to be 231 MPa at V_f : 0.43.

An increase in the flexural strength can be observed in SiC particulate reinforced Al_2O_3 matrix composites for SiC volume fraction in the range of 0.35–0.43. Murthy and Deepak reported microstructure and mechanical properties of $\text{SiC}_p/\text{Al}_2\text{O}_3/\text{Al}$ composites fabricated using directed metal infiltration process. The above mentioned work reported a flexural strength on an average of 145 MPa, with a maximum of 196 MPa [19].

Another work by the inventors of DIMOX process viz: Newkirk et al. report formation of ceramic matrix composites of #500 $\text{SiC}/\text{Al}_2\text{O}_3/\text{Al}$ composites, which resulted in the flexural strength of 350 MPa, for growth temperatures between 900 and 1000 °C [12]. Subsequently, Pickard et al. report mechanical properties of ceramic matrix composites fabricated using DIMOX process. The above mentioned work reported that the flexural strength increases as SiC particulate size decreases [20]. However, in the present work it was observed that directed metal oxidation process in a lower narrower temperature range of 950–980 °C and duration of 65 h was sufficient to obtain completely infiltrated bulk $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites with the following features. A maximum flexural strength of 231 MPa, at SiC volume fraction of 0.43 was obtained. This clearly shows that it is possible to further increase the flexural strength by increasing the metal content, simultaneously employing a finer reinforcement thereby reducing the porosity levels of the composites. However, the

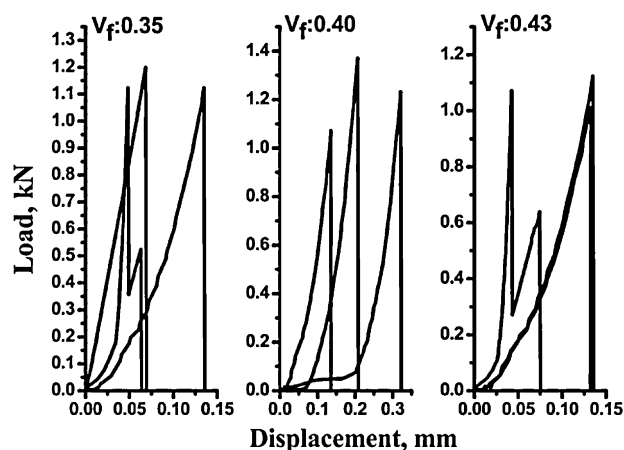


Fig. 5. Load vs. displacement curves from three point bend test for evaluation of flexural strength of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites.

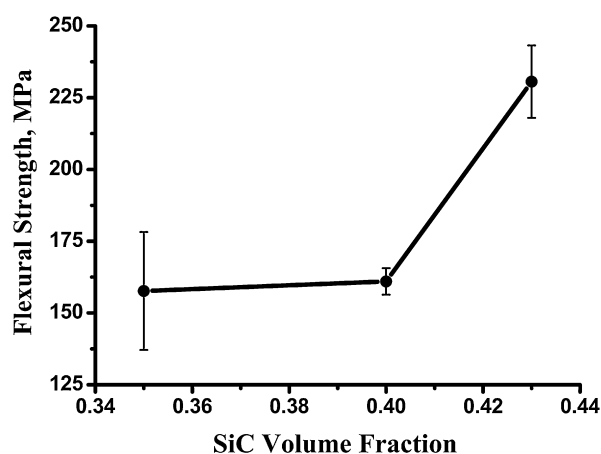


Fig. 6. A plot showing variation in flexural strength of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites with SiC volume fraction.

present flexural strength levels still stand superior when compared to 196 MPa (maximum) reported by Murthy and Deepak, which also reported at higher SiC volume fraction and metal content. However, the flexural strength levels in the present work 231 MPa stand inferior as compared to 340 MPa in the case of Newkirk et al. nevertheless, it is to be noted that these two differ in the size of SiC particulate used for reinforcement. The difference in strength is in agreement with observation of Pickard et al. where it was found that strength increases with decreasing particle size.

3.2.2.2. Fracture toughness. This is one of the most important mechanical properties that are helpful in monitoring the health of a structural material. It is a measure of stress that would initiate the propagation of a pre-existing crack. Fracture toughness of SiC particulate reinforced Al_2O_3 matrix composites fabricated in the present work was determined by the single edge notched beam method under three point bending configuration. Composites with different volume fractions of SiC were evaluated at room temperature. The load vs. displacement curves for SiC particulate reinforced Al_2O_3 matrix composites with volume fraction, V_f : 0.35–0.43 are shown in Fig. 7. Table 3 shows fracture toughness values of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composite and its variation was studied as a function of volume fraction of SiC.

A gradual decline was observed in the fracture toughness of SiC particulate reinforced Al_2O_3 matrix composites with increase in volume fraction of SiC as shown in Fig. 8. The fracture toughness of SiC particulate reinforced Al_2O_3 matrix composites fabricated in the present work was found in the range of 5.61–4.01 $\text{MPa}\sqrt{\text{m}}$ for SiC volume fractions in the range of 0.35 and 0.43, as shown in Table 3. The fractured surfaces were subsequently examined under SEM in order to throw light on the overall nature of fracture. The dominant mode of fracture was found to be brittle in the case of $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites with volume fraction in the range of 0.35 and 0.43. The main reason behind the low levels of fracture toughness was supposed to be the reinforcement particle size.

Materials reinforced with fine particulates often have high values of fracture toughness, owing to high density of

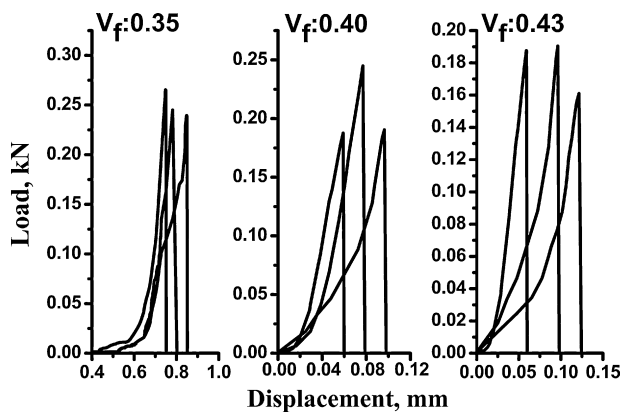


Fig. 7. Plots of load vs. displacement curves for $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites at different volume fractions of SiC, from SENB tests under three point bending load.

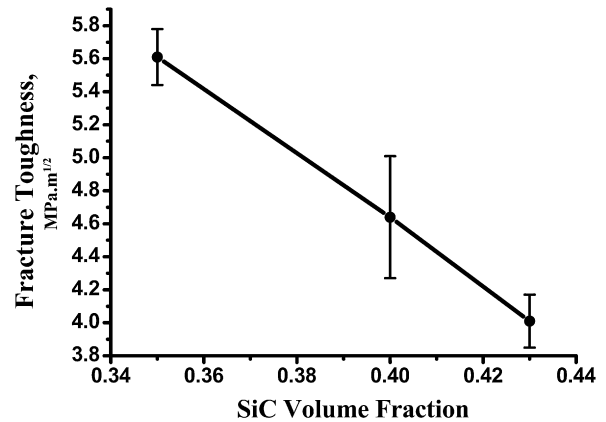


Fig. 8. A plot showing variation in fracture toughness of $\text{SiC}_p/\text{Al}_2\text{O}_3$ CMCs with volume fraction of SiC.

dislocations developed around a finer inclusion than a coarser one. In the case of fracture through the matrix region, it is these dislocations that offer resistance to propagation of an intrinsic/extrinsic crack. In the present work, the size of the reinforcement is quite large. As a result, the overall resistance to crack propagation through matrix region was rather limited. Moreover, the composition of the matrix phase too has an effect on the fracture toughness of $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites. Large size of reinforcement particulates and pores within the reinforcement which would act as stress concentrators could be ascribed to the low levels of fracture toughness obtained in the present work.

Akimune et al. reported $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites fabricated by using directed metal oxidation process, with a fracture toughness of $\sim 5.2 \text{ MPa}\sqrt{\text{m}}$ [21]. However, in the present work it was observed that the fracture toughness is $5.61 \text{ MPa}\sqrt{\text{m}}$ which is superior. Subsequently, Murthy and Deepak studied 50–55 vol.% SiC/30–35 vol.% Al_2O_3 /6–8 vol.% Al composites fabricated by DIMOX process, and reported fracture toughness values of $6.2 \text{ MPa}\sqrt{\text{m}}$ [19]. Similar results could be realized in the present work; despite processing in a narrow temperature range, for a lesser volume fraction of SiC and lower metal content.

In another work by the inventors of the DIMOX process, Newkirk et al., reported $\text{SiC}/\text{Al}_2\text{O}_3/\text{Al}$ composites prepared by directed metal oxidation process with a fracture toughness of $7.8 \text{ MPa}\sqrt{\text{m}}$ [12]. However, no details of volume fraction are provided. It was found to be Subsequently, Nagelberg et al. studied fracture toughness of 48 vol.% SiC/ Al_2O_3 /Al composites fabricated using directed metal oxidation process. The fracture toughness measured by four point flexure single notch beam technique was found to be in the range of $7\text{--}8 \text{ MPa}\sqrt{\text{m}}$ [22]. However, in the works cited above the information on reinforcement size and shape is unclear. In the present work, it was observed that for $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites fabricated using DIMOX process, the value of fracture toughness was in the range of $5.61\text{--}4.01 \text{ MPa}\sqrt{\text{m}}$ with SiC volume fraction varying from 0.35 to 0.43. The difference can be attributed to the lower residual metal content and porosity at the interface.

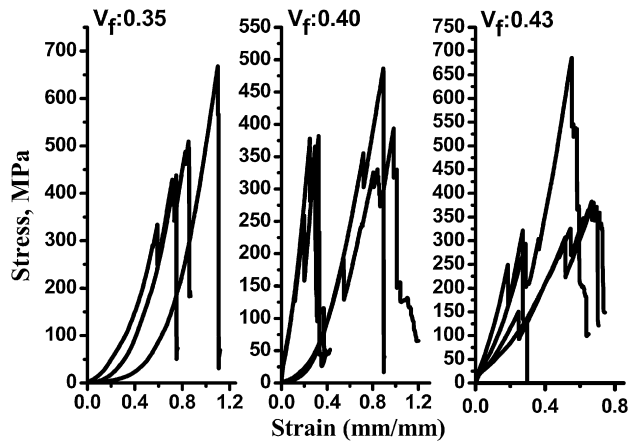


Fig. 9. Plots of stress–strain curves for $\text{SiC}_p/\text{Al}_2\text{O}_3$ CMCs tested under uniaxial compressive load, for different volume fractions of SiC.

3.2.2.3. Compressive strength. Another important mechanical property of ceramic materials meant for structural applications is its compressive strength. The corresponding stress vs. strain curves at different volume fractions of SiC is shown in Fig. 9. The compressive strength of SiC particulate reinforced Al_2O_3 matrix composites was determined and its variation was studied as a function of SiC volume fraction (Fig. 10). The compressive strength and compressive modulus of SiC particulate reinforced Al_2O_3 matrix composites fabricated in the present work was found to be in the range of 382–668 MPa and 6.75–10.45 GPa respectively, for SiC volume fractions in the range of 0.35–0.43 as shown in Table 4.

Murthy and Deepak reported a compressive strength in the range of 1.4–1.8 GPa for $\text{SiC}/\text{Al}_2\text{O}_3/\text{Al}$ composites prepared by directed melt infiltration process [19]. Newkirk et al. reported SiC particulate reinforced Al_2O_3 composites produced by directed metal oxidation process and a compressive strength of 1.95 GPa [12]. However, in the present work on $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites fabricated using DIMOX process, a maximum compressive strength of 668 MPa could only be observed. These low values are supposed to be due the microstructure developed in the matrix phase, the primary reason being the pore network

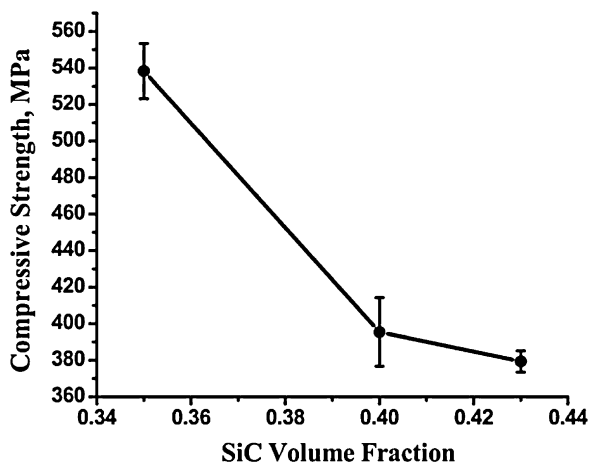


Fig. 10. Plot showing variation in compressive strength of $\text{SiC}_p/\text{Al}_2\text{O}_3$ CMCs as a function of SiC volume fraction.

Table 4

Details of compressive strength of $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composite.

| Label | Volume fraction | Max. stress (MPa) | Max. strain (%) | Modulus (MPa) | Yield load (kN) |
|-------|-----------------|-------------------|-----------------|---------------|-----------------|
| B1 | 0.35 | 668.00 | 4.21 | 10,450 | 1.29 |
| B2 | 0.40 | 486.00 | 4.79 | 8249 | 6.07 |
| B3 | 0.43 | 382.40 | 4.70 | 6752 | 5.24 |

instead of a dense matrix. (Porosity $\sim 11\%$, reported elsewhere). The low strength of the composite stems from its irregular and interconnected pore structure, which provides both local stress concentrations to drive the crack and preferred paths for crack propagation. The interconnected Al present in present composite is much less constrained as to its mode of deformation. Other reasons include limited bonding between the particulate and the matrix phase as can be seen from Fig. 4.

Ceramic matrix composites reinforced with fine particulates has often high values of compressive strength owing to high density of dislocations developed around a finer inclusion than a coarser inclusion. In the case of fracture through the matrix region, it is these dislocations that offer resistance to propagation of an intrinsic or extrinsic crack. In the present work, the size of the reinforcement is quite large. As a result, the overall resistance to crack propagation through matrix region was rather limited. Moreover, the composition of the matrix phase too has an effect on the compressive strength of SiC particulate reinforced Al_2O_3 matrix composites. As a consequence of processing schedule, the composition of matrix phase has changed substantially. The matrix phase has developed a measurable porosity resulting low levels of compressive strength. Reasons for low compressive strength are large size of reinforcement particulates and pores within the reinforcement which would act as stress concentrators.

3.2.2.4. Hardness. Variation in the Vickers hardness of SiC particulate reinforced Al_2O_3 matrix composites fabricated by directed metal oxidation process was studied as a function of volume fraction of SiC. Hardness of SiC particulate reinforced Al_2O_3 matrix composites increased with volume fraction of SiC. However, the increase in hardness was rather slow up to a volume fraction of 0.43, beyond which a rapid increase in hardness was observed as shown in Fig. 11. The Vickers hardness of SiC particulate reinforced Al_2O_3 matrix composites fabricated in the present work was found to be in the range of 8.84–12.85 GPa for SiC volume fractions in the range of 0.35–0.48 as shown in Table 3.

Akimune et al. studied hardness of SiC particle/ Al_2O_3 composites fabricated by directed metal oxidation process and observed a value of 12.5 GPa [21]. Subsequently, Jayaram et al. reported hardness of $\text{Al}_2\text{O}_3/\text{SiC}$ composites prepared by directed metal oxidation process as a function of SiC particle size (5–60 μm), measured by Vickers indenter with 30 kg load. The hardness of the composites falls in the range of 2.59–7.67 GPa [23]. The hardness values obtained in the present work ranges from 8.24 to 12.85 GPa, which stand superior when compared to 12.5 GPa and 2.59–7.67 GPa, reported by Akimune et al. and Jayaram et al., respectively.

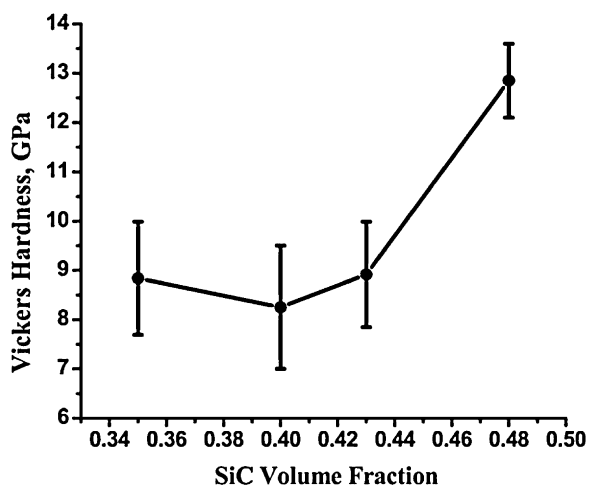


Fig. 11. A plot showing variation in Vickers hardness of $\text{SiC}_p/\text{Al}_2\text{O}_3$ CMCs at different volume fractions of SiC.

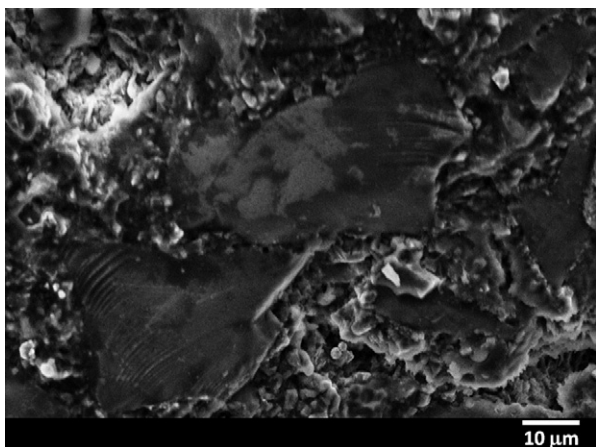


Fig. 12. Microscopic features of $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites fractured under three point bending load.

3.2.2.5. Fractography. Specimens fractured during three point bend tests, performed for the evaluation of flexural strength and fracture toughness of $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites, were examined under SEM for further investigation pertaining to the nature of fracture. Typical fractographs of a few composites are shown in the following Fig. 12. The fracture of $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composite is comprised of brittle modes of fracture. Features of brittle mode of fracture are evident even at low magnifications as can be seen from the figure. Also it can be observed that the reinforcement phase has failed in a brittle manner with cleavage faces across the path of the crack. The matrix phase has failed in a brittle manner.

4. Conclusions

In the present work it was observed that the DIMOX process could be successfully reproduced to fabricate bulk $\text{SiC}_p/\text{Al}_2\text{O}_3$ ceramic matrix composites at a rather low temperature. The composites thus obtained were evaluated for mechanical properties such as flexural strength, fracture toughness, compressive strength and hardness. Appreciable magnitudes of flexural strength, fracture toughness and hardness were

realized for $\text{SiC}_p/\text{Al}_2\text{O}_3$ composites developed in the present work. However, a low compressive strength was a matter of concern which was likely to be a consequence of limited bonding between particle and matrix and an irregular and contiguous pore network.

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