

## Al<sub>2</sub>O<sub>3</sub>-SiC Composites from Natural Mineral Precursors

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In-situ synthesis of ceramic-ceramic composite powder containing mainly  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and  $\beta$ -SiC has been accomplished using natural alumina silicate(s) and carbon as precursor mixtures. For this purpose, kaolinite, kyanite and pyrophyllite have been tested. The size and morphology of the carbide phase can be easily controlled using carbon particles of specific size and morphology. The role of the impurities in the final composite powder is under intensive investigation. Although the alkalis and magnesia are mostly eliminated during the synthesis reaction, the role of CaO and Fe<sub>2</sub>O<sub>3</sub> is not clear. The kinetic study of the carbothermal reduction process during the formation of the alumina-silicon carbide composite powder has been carried out. This was done to determine the basic reaction mechanisms which govern the overall reduction reaction. Thus far, thermogravimetric analysis experiments have shown the reaction to be of the first order with an activation energy of ~250-300 kJ/mole.

### 1.0 Introduction

The development of advanced ceramic composites also referred to as CMC's (Ceramic matrix ceramic composites) relies on the use of high purity powders with fine and well-controlled morphologies. It is also imperative in manufacturing composite materials that the reinforcing phase be uniformly distributed throughout the product. Thus, a range of techniques to disperse the second phase (such as whiskers) in a powder matrix, without damaging them, have been developed. These include colloidal processing<sup>1</sup>, slurry mixing under controlled pH condition with ultrasonic dispersion<sup>2-5</sup>, followed by filtration and centrifugation etc. Non-powder processing methods for developing ceramic matrix composites via infiltration of the matrix into reinforcement preforms have been tried by several workers<sup>6-7</sup>.

During the last 10-15 years considerable studies have been carried out on Al<sub>2</sub>O<sub>3</sub> - SiC whisker composites, because of its high fracture toughness and strength<sup>8-9</sup>. Very recent studies<sup>10</sup> on dynamic fatigue characteristics of Al<sub>2</sub>O<sub>3</sub> - 25% SiC whisker composite showed that this material has a high resistance to slow crack growth. Greenleaf Corporation of U.S.A.<sup>11</sup> is currently marketing cutting tools made from Al<sub>2</sub>O<sub>3</sub> - SiC whisker composites because of its superior performance. However, in spite of extensive efforts in developing these ceramic composite materials, commercial application of these products have not been significant. This is primarily for two reasons:(i) complexity of manufacturing very homogeneous mixed powders and the cost associated with it and (ii) difficulties encountered in fully densifying these powders. Currently, the techniques available to fully densify Al<sub>2</sub>O<sub>3</sub> - SiC

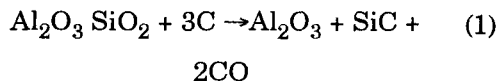
whisker composites are pressure sintering and hot-isostatic pressing.

In order to significantly reduce the cost of production CMC powders, attempts have been made to synthesize  $\text{Al}_2\text{O}_3$  - SiC based composite powders from natural mineral precursors. Additional studies were also made using an alumina-silica freeze-dried gel powder to synthesize such composite powders. Some preliminary results of these studies have already been reported<sup>12,13</sup>.

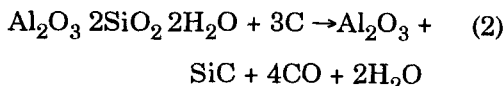
An extensive literature survey showed three publications which are relevant to this study. Bechtold and Cutler<sup>14</sup> carried out carbothermal reduction of kaolinite with carbon, with the objective of extracting  $\text{Al}_2\text{O}_3$  and separating SiC. Bentsen et. al.<sup>15</sup> studied the thermodynamics of SiC formation from mullite. Kimura et. al.<sup>16</sup> tried to produce  $\text{Al}_2\text{O}_3$  and SiC composite powder from a hydrous mica (sericite), which is an alkaline alumino-silicate.

Reactions involved in the carbothermal reduction of natural minerals and synthetic alumino-silicate gel for synthesizing  $\text{Al}_2\text{O}_3$  - SiC composite powders are as follows:

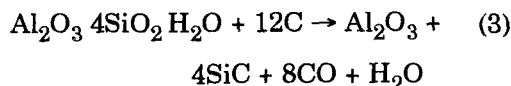
(Kyanite)



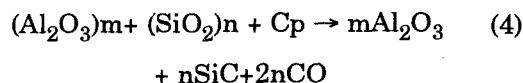
(kaolinite)



(Pyrophyllite)



(Synthetic gel) (where  $p = 3n$ )



The stoichiometric reactions as given in equation (1) to (3) will produce composites containing the silicon carbide phase varying from 28 wt.% to 61 wt.%. However, any composition with an intermediate concentration of SiC can be easily produced from a mixture of different natural minerals or by adding excess alumina or silica. It should be realized that synthetic alumino-silicate gel has the freedom to make any desired composite containing  $\text{Al}_2\text{O}_3$  and SiC as the major phases.

Five typical compositions are shown in Table I. Any loss of  $\text{SiO}_2$  as SiO during the reaction was not taken into account. Quantitative x-ray diffraction analyses, using an internal standard, showed that for reaction (1) (also Table I) using kyanite and carbon as precursors some SiO loss was encountered. For example, five repeated runs of kyanite and carbon precursors yielded an average of  $69.84 \pm 1.02$  wt.% of  $\text{Al}_2\text{O}_3$  and  $26.6 \pm 0.60$  wt.% SiC, in place of theoretical values of 71.8 and 28.2 wt.% respectively. The natural mineral kyanite also contains  $\text{Fe}_2\text{O}_3$ , CaO, MgO,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$  etc., which accounts for the balance of the 100 wt.%. It was noted that cold pelletization of precursor mixtures reduced SiO loss from the system, as compared to the conditions when loose powder was used.

Table I. Selected Compositions for Composite Powders.

Starting Powder	Starting Composition	Final Composition
a) kyanite + carbon	82 wt.% kyanite 18 wt.% carbon	72 wt.% Al <sub>2</sub> O <sub>3</sub> + 28 wt.% SiC
b) pyrophyllite + carbon	72 wt.% pyrophyllite 28 wt.% carbon	39 wt.% Al <sub>2</sub> O <sub>3</sub> + 61 wt.% SiC
c) kaolinite + carbon	78 wt.% kaolinite 22 wt.% carbon	56 wt.% Al <sub>2</sub> O <sub>3</sub> + 44 wt.% SiC
d) kyanite + kaolinite + carbon	31 wt.% kyanite 49 wt.% kaolinite 20 wt.% carbon	63 wt.% Al <sub>2</sub> O <sub>3</sub> + 37 wt.% SiC
e) pyrophyllite + kaolinite + carbon	43 wt.% pyrophyllite 31 wt.% kaolinite 26 wt.% carbon	46 wt.% Al <sub>2</sub> O <sub>3</sub> + 54 wt.% SiC

## 2.0 Experimental Procedures

### 2.1 Raw Materials

Although a large number of natural minerals have been tested in this project, the primary activities were confined to three main minerals: (i) kaolinite from Georgia (USA), (ii) kyanite (Virginia, USA) and pyrophyllite (North Carolina, USA). Commercial lamp black was the main source of carbon. Two types of experiments were performed in this program. Initially extensive studies using a variety of minerals were made to follow the reaction sequence during the whole carbothermal reduction process leading to the formation of Al<sub>2</sub>O<sub>3</sub> and SiC. Currently, experimental tests are being made to follow the kinetics of the reaction process using the thermo-gravimetric method. This is possible as all reactions (1) to (4) for the formation of SiC from SiO<sub>2</sub> involve weight loss.

### 2.2 Firing

The details were reported previously<sup>12</sup> for the first type of experiments. This essentially involved heating the precursor mixtures [mineral(s) and carbon black] in an inductively heated graphite crucible (shielded by Ar) in the temperature range 1550-1750°C. For the second type of experiment i.e. kinetic studies by the weight loss measurements, a vertical alumina tube furnace was used.

The reaction chamber consists of an alumina tube (length - 114cm, I.D. - 3.9 cm, O.D. - 4.5 cm) within a Super Kanthal Furnace. Samples contained in alumina crucibles are raised slowly to the "hot" zone in the furnace and then suspended beneath a Mettler Analytical Balance for weight measurements. The operating temperature range of the furnace is between 1,000°C and 1,600°C measured at the hot zone. The temperature is controlled by a Pt-Pt10%Rh thermocouple

connected to a furnace controller. Separate streams of argon, carbon dioxide or carbon monoxide after passing through a purification train, are metered, mixed and injected into the bottom of the reaction tube. Data from the Mettler balance is passed in digital form via an RS-232 interface to an IBM PC where it is stored in data files. The data are then plotted as normalized weight versus time.

### 3.0 Results

#### 3.1 Intermediate Phase

In our previous paper<sup>12</sup> we have already reported that the intermediate phase- mullite was formed when kaolinite was heated along with carbon. It has been observed that all alumino-silicates, whether natural minerals or synthetic gel, formed mullite in the temperature range 1200-1450°C. Both needle-like mullite crystals and hard agglomerates of mullite (may be partially sintered) were formed. The latter type was more frequently encountered with impure minerals. An example of the hard agglomerated mullite particles is shown in Figure 1. EDS analyses of this particle showed that two major elements are Al and Si. X-ray diffraction analyses of this powder showed that this specimen was only composed of mullite and cristobalite (up to the limit of detectability of the x-ray technique).

During the intermediate stage of transformation, it was also observed that all cold pressed pellets were well sintered into a hard lump as shown in Figure 2, requiring a mortar and pestle to break up. The extent of sintering depended on the nature and amount of impurities present in the natural minerals, as expected.

#### 3.2 Final Phases

During the final transformation of mullite into  $Al_2O_3$  and SiC, not only did the sintered specimens break up but also the hard agglomerated particles



Figure 1. Hard agglomerate of mullite.

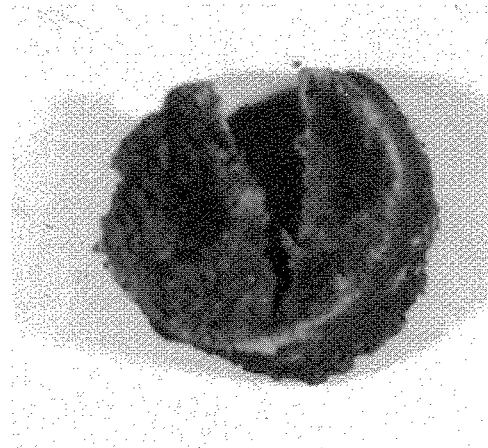


Figure 2. Sintered mullite +  $SiO_2$  + C pellet.

disintegrated during the reduction reaction (by CO), which resulted in the formation of  $Al_2O_3$  and SiC from mullite and silica. An example of the disintegrated particles is shown in Figure

3. Figure 4 shows a magnified view of the surface of the particle shown in Figure 3. It is quite apparent that the large particle in Figure 3 is really composed of a large number of very small (micrometer size) particles.

As the  $\text{SiO}_2$  phase was formed from the dissociation of the natural minerals (kaolinite, kyanite or pyrophyllite), this phase is very uniformly distributed in the whole system. It is expected that the  $\text{SiC}$  phase should similarly be homogeneously distributed in the system.

EDS analyses of these particles confirm that  $\text{Al}_2\text{O}_3$  and  $\text{SiC}$  phases are uniformly distributed in the whole system. X-ray diffraction analyses showed unequivocally that the final major phases are  $\alpha\text{-Al}_2\text{O}_3$  and  $\beta\text{-SiC}$ . These x-ray

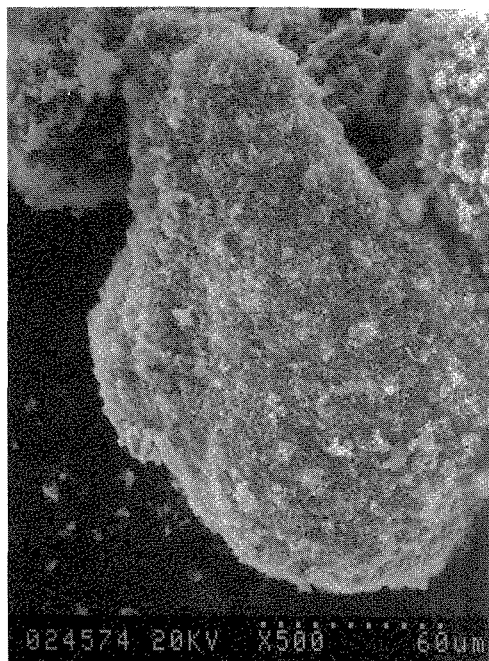


Figure 3. Disintegrated particles containing only  $\text{Al}_2\text{O}_3$  and  $\text{SiC}$ .

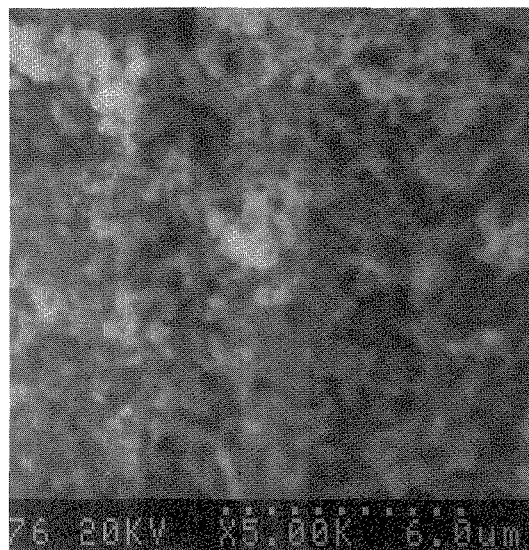


Figure 4. Magnified view of Figure 3.

results are already reported<sup>12,13</sup>. An additional diffraction plot of the final phases formed from a precursor mixture of kyanite-kaolinite-carbon is shown in Figure 5. Particle size analyses were also carried out on the powder, gently crushed in an agate-mortar and pestle or mechanically milled in hexane. These results showed very little difference in the particle size and distribution in these powders, as shown in Figure 6(a) and (b). Moreover, it should be noted that over 95% of the particles are below  $1\ \mu\text{m}$ . It has been observed that similar results are obtained irrespective of the type of natural minerals used.

Similar studies on alumino-silicate gels showed that  $\alpha\text{-Al}_2\text{O}_3$  grains are larger than those formed from natural minerals. One possible explanation may be that concentration of  $\text{Al}_2\text{O}_3$  in the gel was very much higher (molar ratio  $3\text{Al}_2\text{O}_3 : 2\text{SiO}_2$ ) than those present in natural minerals ( $\text{Al}_2\text{O}_3 : 1$  to  $4\ \text{SiO}_2$ ).

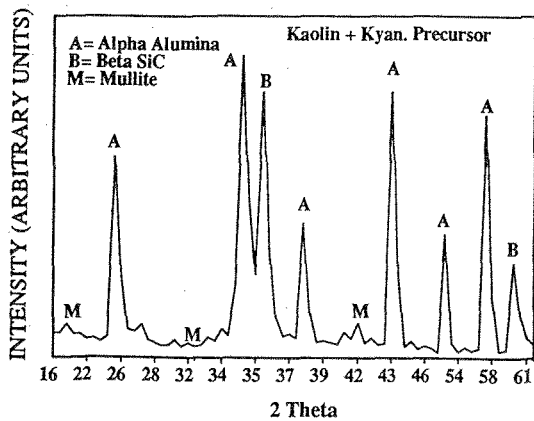
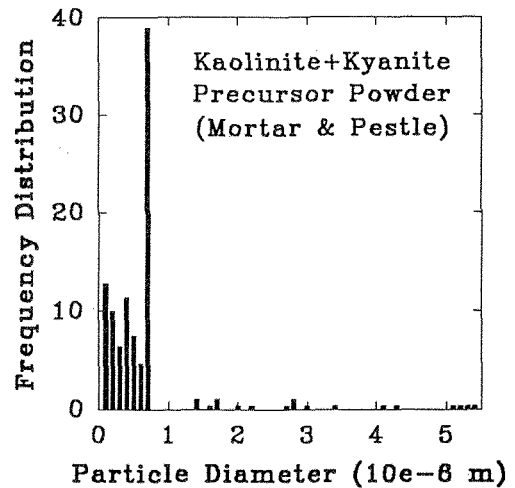


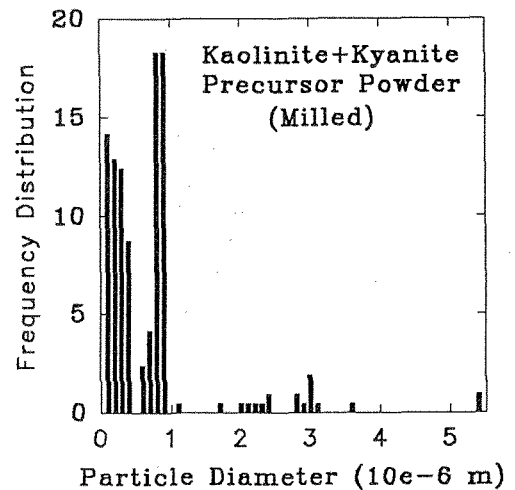
Figure 5. X-ray plot of composite powder from kyanite-kaolin-carbon precursor.

### 3.3 Morphology

In previous papers we have reported that the size and the shape of the SiC phase can be easily controlled by adding carbon of a specific size and shape in the precursor mixtures. Thus, if a portion of the total carbon, required for stoichiometric carbothermal reduction of minerals or synthetic alumino-silicate gel, includes graphite fibers, spheres or hexagonal flakes, the final SiC phase is formed in the shape of the graphite particles added. An example of SiC fibers formed during the in-situ synthesis of  $\text{Al}_2\text{O}_3$  and SiC composite from a kaolinite-carbon precursor is shown in Figure 7. However, it has been noted in a large number of cases, where not very pure minerals were used in which (>3 wt.% of oxides other than  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  are present, specially >1 wt.%  $\text{Fe}_2\text{O}_3$ ), string-like and droplet type of silicon-rich phases were formed. Further analyses of these string-like particles were identified as both SiC and FeSi alloys. A typical example of a string with a spherical bead of the tip of FeSi alloy is shown in Figure 8.



(a)



(b)

Figure 6. Particle size distribution of composite powders. (a) crushed in a mortar and pestle, and (b) mechanically milled in hexane.

### 3.4 Impurities in Natural Minerals

All natural minerals contain impurities, such as  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$  etc. If the  $\text{Al}_2\text{O}_3$  and SiC

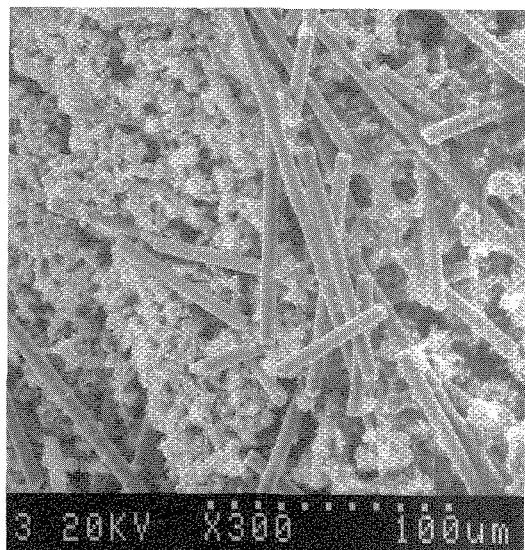


Figure 7. SiC fibers from carbon fiber precursor.

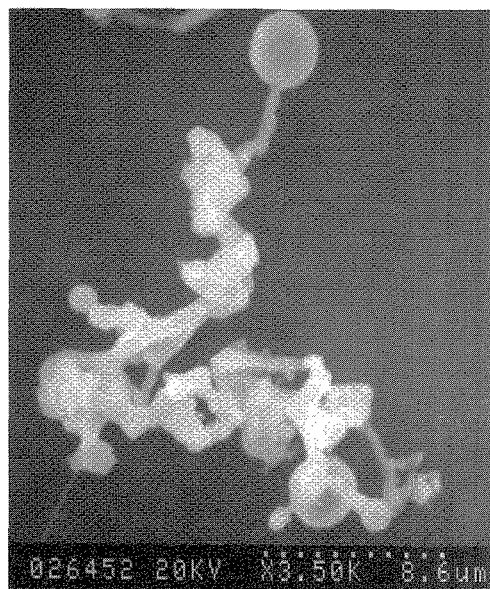
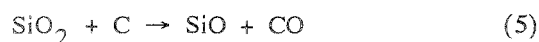


Figure 8. String-like particles identified as both SiC and FeSi alloy.

composite powders synthesized from natural aluminosilicates are ever considered for high-tech applications, it is important to know their effects on composite properties, such as the sintering behavior, strength, fracture behavior and high temperature properties. It has been reported previously<sup>12,13</sup> that three oxides ( $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$  and  $\text{MgO}$ ) which are considered to be harmful are significantly reduced or almost eliminated from the system during the carbothermal reduction of aluminosilicates. Further evidence of this reduction is shown in Table II. Titanium dioxide is converted into TiC. Two other oxide impurities are CaO and  $\text{Fe}_2\text{O}_3$ . The carbothermal reactions which produce the  $\text{Al}_2\text{O}_3$  and SiC composites involve mass loss, as given in equation (1) to (4). For example, the kaolinite-carbon reaction (equation 2) results in a minimum mass loss of 37 wt.%. In practice, the mass loss is larger, as some SiO is also lost from the system because the formation of SiC from  $\text{SiO}_2$  involves at least the following reactions.



Thus, it is expected that the relative proportion of both CaO and  $\text{Fe}_2\text{O}_3$  would increase in the composites. This can be evidenced from the chemical analyses shown in Table II. Composite powders were reoxidized at  $\sim 1200^\circ\text{C}$  to convert SiC and TiC into  $\text{SiO}_2$  and  $\text{TiO}_2$  for chemical analysis. In some instances, the concentration of CaO or  $\text{Fe}_2\text{O}_3$  are found to be lower in the composites than present in the original raw materials. It should be noted that the data in Table II for the raw

Table II. Chemical Analysis of Natural Minerals and Composite Powders.

	Kaolinite (Georgia) <sup>♦</sup>		Kaolinite (Lang Bay, Canada) <sup>♦♦</sup>	
	Dehydrated	Composite	Dehydrated	Composite
Al <sub>2</sub> O <sub>3</sub>	44.60	56.45	41.04	39.75
SiO <sub>2</sub>	52.80	40.95	52.60	55.10
CaO	0.95	0.10	0.70	0.35
MgO	0.20	0.18	0.65	0.30
Fe <sub>2</sub> O <sub>3</sub>	0.40	0.80	2.80	3.70
K <sub>2</sub> O	0.10	0.05	0.58	0.12
Na <sub>2</sub> O	0.30	0.20	0.46	0.04
TiO <sub>2</sub>	1.60	1.13	1.25	1.10
P <sub>2</sub> O <sub>5</sub>	---	0.02	0.11	0.05

Chemical analyses of raw powder were provided by the suppliers.

♦NAAM Corporation, Houston, Texas, U.S.A.

♦♦ Langbay Resources Ltd., B.C., Canada

materials are an average of a large number of batches and these are supplied by the companies noted.

It has not been possible to-date to identify the location of CaO in the composites, as the concentration is too low to be detectable by the x-ray diffraction technique. It is possible that CaO may react with Al<sub>2</sub>O<sub>3</sub> and has gone into solid solution with Al<sub>2</sub>O<sub>3</sub> or formed a Calcium aluminate compound. In the case of Fe<sub>2</sub>O<sub>3</sub>, with large concentration (>2 wt.%), there is definite evidence of formation of iron silicates, which then was reduced to FeSi alloys, as shown in Figure 8. Further studies are being done to estimate the change in lattice parameters of both  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> and  $\beta$ -SiC, because of the presence of CaO and Fe<sub>2</sub>O<sub>3</sub> in the system. On the other hand, addition of less than 1% iron oxide in a pure kaolinite showed a

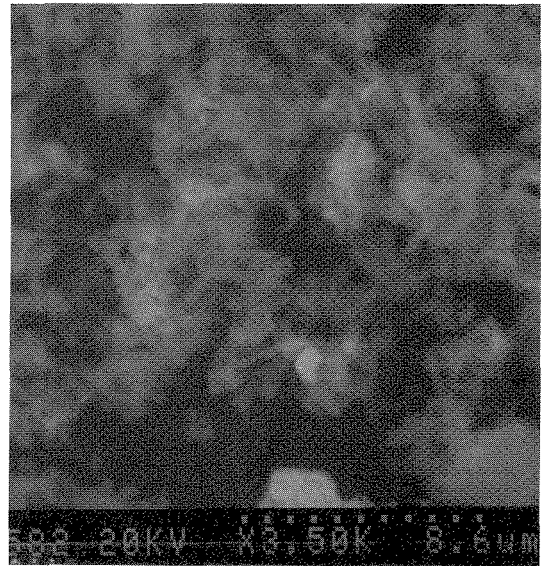


Figure 9. SiC whiskers formed in kaolinite with ~0.5 wt.% Fe<sub>2</sub>O<sub>3</sub> addition.



tendency to form SiC whiskers in the system as shown in Figure 9.

It is important to note that the presence of impurities in the precursor minerals such as CaO and Fe<sub>2</sub>O<sub>3</sub> (up to the concentration tested so far i.e. 2-3 wt.%) has no significant bearing on the sintering behavior of these powders. This will be discussed later on.

### 3.5 Kinetics of Formation of Al<sub>2</sub>O<sub>3</sub> and SiC from Kaolinite

The kinetics of formation of Al<sub>2</sub>O<sub>3</sub> and SiC from kaolinite-carbon precursor mixtures (Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>·2H<sub>2</sub>O + C) were studied from the weight loss measurements in a thermo-gravimetric equipment described earlier. A previous kinetic study was carried out by Lee and Cutler<sup>17</sup> on powder and pelletized forms in Ar and N<sub>2</sub>. Our studies involved only powder specimens, suspended in an alumina crucible in gaseous environment which was primarily Ar or CO. Different variables affecting the formation of SiC from SiO<sub>2</sub>, (present in kaolinite), were studied such as, temperature, time, sample size (with kyanite), gas environment, etc. Details of these studies will be published elsewhere. Some results on the weight loss behavior at different temperatures as a function of time are shown in Figure 10. Semi-logarithmic plots of the data shown in Figure 10, show linearity up to 50 to 55% of weight loss, indicating a first order reaction up to this stage. An Arrhenius plot of the data yielded an activation energy for the reaction as 250-300 kJ/mole, which is very similar to the value reported by Lee and Cutler<sup>17</sup>.

It has been noted during these investigations that extraneous effects can affect the rate of formation of SiC from SiO<sub>2</sub> e.g. batch size and presence of Al<sub>2</sub>O<sub>3</sub> in the system. Figure 11 shows the effect of batch size on the reduction behavior of metakaolin at 1450°C. For the smaller

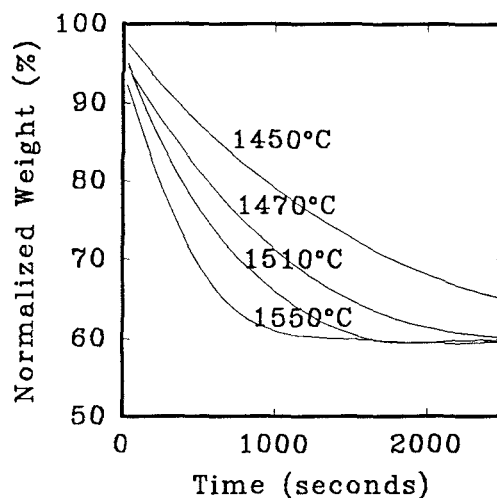
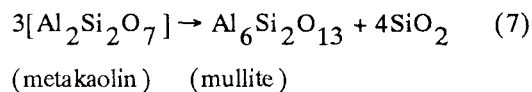


Figure 10. Weight loss behavior at different temp. as a function of time.

batch, the specimen was in a thin layer, which allowed SiO vapor to escape without reacting with carbon, thus, forming much less SiC than the heavier two batches (confirmed by x-ray). Figure 12 shows the weight loss behavior of pure SiO<sub>2</sub>, metakaolin and mullite at two different temperatures 1450 and 1550°C. At 1450°C, the weight loss behavior of SiO<sub>2</sub> and metakaolin is similar, but weight loss of mullite is slower. This difference may be due to the fact that above 1200°C metakaolin will form free SiO<sub>2</sub> and mullite by the following reactions, the reduction behavior of this free SiO<sub>2</sub> is similar to pure SiO<sub>2</sub>.



On the other hand, mullite does not have free SiO<sub>2</sub>, thus dissociation of mullite must take place before SiO<sub>2</sub> in mullite can be reduced.

At 1550°C, significant differences exist between the weight loss behavior of

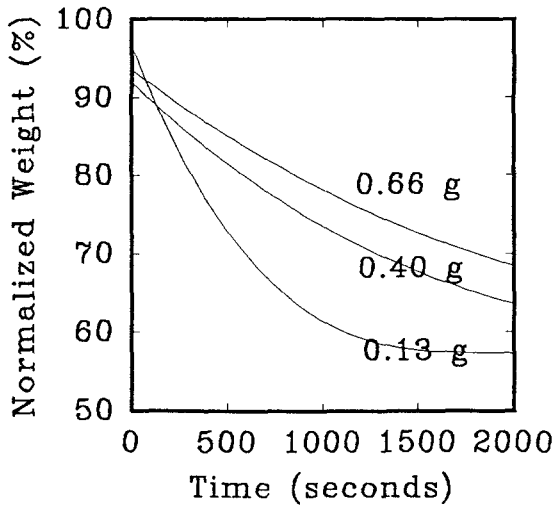
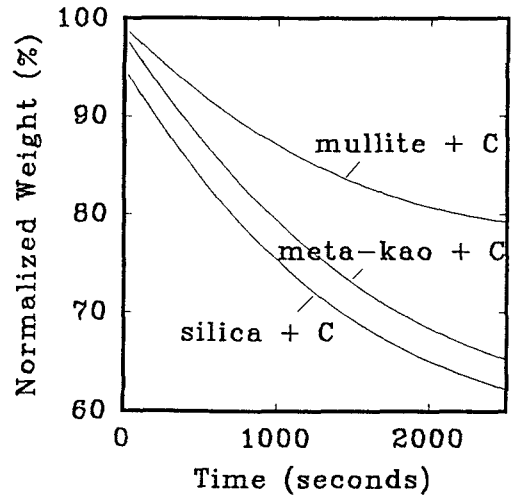


Figure 11. Effect of batch size on the reduction behavior of metakolin at 1450°C.

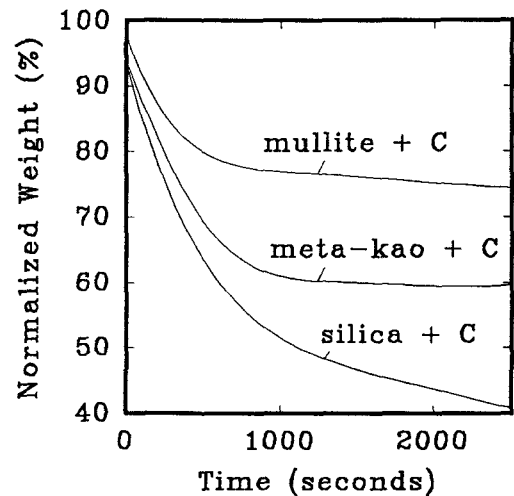
$\text{SiO}_2$ , metakaolin and mullite. At this temperature mullite is not stable in CO environments. In spite of that mullite has the lowest rate of reduction, as compared to silica, which has the fastest rate. It is possible that the rate of reduction is being affected by the presence of  $\text{Al}_2\text{O}_3$ . Mullite has ~72 wt.%  $\text{Al}_2\text{O}_3$  versus 46 wt.% in metakaolin. In the formation of SiC from  $\text{SiO}_2$ , three gaseous species SiO, CO and  $\text{CO}_2$  are involved in the reaction.  $\text{Al}_2\text{O}_3$  may impede the transportation of these gaseous species, which slowed down the reaction rate in mullite and metakaolin.

### 3.6 Densification of Composite Powders

It has not been possible to sinter these composite powders to greater than 90% density (of the theoretical i.e. 10% of porosity), even when sintered at 1850°C for one hour. Heating at this temperature for a longer period resulted in the formation of  $\text{Al}_4\text{C}_3$ . Some of these composite powders had as much as 4-5% impurity oxides, such as  $\text{Fe}_2\text{O}_3$  and CaO. In spite of the impurity content, these



(a)



(b)

Figure 12(a) and (b) Weight loss behavior of pure  $\text{SiO}_2$ , mullite and metakaolin at 1450 and 1550°C.

powders could not be sintered to high density (i.e. <90%). Even additives, which are normally used as sintering agents did not improve significantly the sintered density of these composite powders. The

technique of hot-pressing was used to produce dense pellets, having density greater than 98% (of the theoretical). Hot-pressing was carried out at 1850°C for one hour at a pressure of 20 MPa in a graphite die. Depending upon the nature of starting powder (i.e. ratio of  $\text{Al}_2\text{O}_3$ : SiC, natural mineral used for powder synthesis, type of carbon precursors etc.) some grain growth, specially of  $\text{Al}_2\text{O}_3$ , was encountered. In all cases, the SiC phase was found to be very homogeneously distributed. This is not surprising, as most of the particles are less than 1  $\mu\text{m}$  (see Figure 6) and are very intimately mixed because of in-situ formation of the carbide phases. In some cases, a duplex structure of large white and gray areas were observed as shown in Figure 13 with a substructure within these areas. EDS analyses showed that the white and gray areas are rich in Al and Si respectively, indicating that these are predominately  $\text{Al}_2\text{O}_3$  and SiC. The substructure, which is in nano-size scale, is mainly the SiC phase.

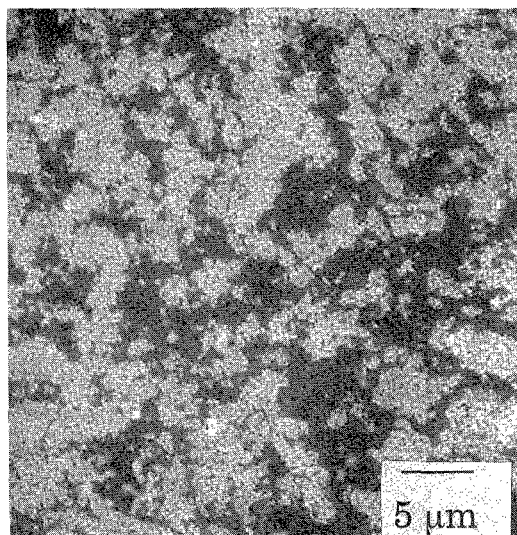


Figure 13. Duplex structure of hot-pressed pellet.

## Conclusion

It has been possible to synthesize  $\text{Al}_2\text{O}_3$ -SiC composite powders using natural mineral precursors such as kaolinite and pyrophyllite. The distribution of the SiC phase is very homogeneous, even up to the nano-size scale in some cases. It has not been possible to sinter these powders to high densities (i.e. <90% of the theoretical). Only hot-pressing could produce high density pellets so far.

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