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### Review on Oxide Bonded Porous SiC Ceramics: Processing, Properties and Applications

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#### Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

#### Article Information

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**Review Article** 

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#### ABSTRACT

Recently the use of porous SiC ceramics is increasing tremendously for various industrial applications due to its excellent properties. Various processing techniques are tried to develop porous SiC ceramics. The fabrication of porous SiC ceramics by an oxidation bonding technique offers several advantages like good oxidation resistance because of air sintering of the ceramics, low sintering temperature, cost effectiveness and easy control over the porosity and other properties of the resultant ceramics. This review deals with processing strategies of oxidation bonding technique. The effect of pore former, powder size, sintering temperature and sintering additives on the properties of the oxide bonded porous SiC ceramics are discussed in detail. The available literatures are thoroughly reviewed with emphasis on mechanical properties, thermal shock resistance properties, gas permeability behavior and separation of particulate from off gas. This review has also enlightened future scope on processing of oxide bonded porous SiC and its usage in various applications.

Keywords: Porous SiC; oxide bond; mechanical properties; gas permeability; thermal shock resistance.

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#### **1. INTRODUCTION**

Currently, porous SiC ceramics have been a focus of interesting research in the field of porous materials due to their excellent structural properties, high strength, high hardness, and superb mechanical and chemical stabilities, particularly at high temperatures and hostile atmospheres. Hence porous SiC ceramics have been considered as an ideal candidate for catalyst supports, thermal insulators, high temperature structural materials, kiln furniture, seal pumps for automotive water pumps, composite armour protection, heat exchangers, bio-implants. bearings. abrasion-resistant components such as blast nozzles and centrifuge tiles, burner technology, furnace heating elements, medical implants (hip replacement, orthopedic implants) hydrogen-permselective membranes and hot gas particulate filtration in different industrial processes like integrated gasification combined cycle (IGCC) and coal combustion [1-21]. The use of porous SiC ceramics for hot gas filtration in the coal gasification process especially for pressurized fluidized bed combustion (PFBC) and integrated gasification combined cycle (IGCC) is increasing in order to limit the emission of some corrosive or toxic particles into the environment. For such applications, the ceramic filters must be able not only to resist the chemical attack at high temperature (600 - 900°C) and high pressure (6 -10 atm) by a variety of gases such as O<sub>2</sub>, HCl, H<sub>2</sub>S, Cl<sub>2</sub>, SO<sub>2</sub>, NO and H<sub>2</sub>O, but also to withstand the mechanical stress or thermal shock in the pulse cleaning process [22-24]. There are mainly four methodology developed for manufacturing of porous SiC ceramics: replication, reaction bonding, partial sintering, and expandable microspheres [25-40]. Among them, the reaction bonding technique is a promising method, which involves carbothermal reduction techniques, silicizing techniques [28-29], etc. The processing of porous ceramics using preceramic polymers is another promising route for making porous SiC ceramics [30-37]. Synthesis of hierarchical porous materials from biological templates by carbothermal reduction techniques continues to generate significant interest because biological structures exhibit excellent strength at low density, high stiffness and elasticity, and high damage tolerance integrated into these structures through the evolutionary process [38-401. All the above mentioned process required sinterina temperature hiah and costlv environment which enhance the processing cost. Clay bonded SiC filter has been successfully

operated at temperature 620-845°C in an oxidizing atmosphere for 5855 h [22-24]. But they suffer from several disadvantages like (i) Failure due to softening of the bond during service and corrosion. (ii) bond depletion. (iii) bond destabilization, (iv) cracking/spalling due to mismatch of thermal expansion coefficient, etc. Therefore clay bonded SiC filters damaged during long term operation at higher temperature. It is expected that such a problem would not arise for self bonded SiC ceramics [41-42]. Unfortunately, submicron powders and high temperature are required for production. Moreover oxidation remains another problem for high temperature operation of self bonded SiC filters in an oxidizing atmosphere [43-44]. In order to realize low temperature fabrication of porous SiC ceramics, secondary bond phase was considered to bond SiC particles. Utilizing the oxidation derived silica to bond SiC particles. Zhu and co-workers prepared SiC reticulated porous ceramics [45]. Following this. She et al. developed unique oxidation bonding technique for fabrication of porous SiC ceramics with superior oxidation resistance [46-49]. The key feature of the process is that SiC powder compacts are heated in air instead of an inert atmosphere. At the heating stage, surface of SiC get oxidized and SiC particles bonded each other by oxidation derived SiO<sub>2</sub> glass, which crystallize to cristobalite at temperature above 1300°C. The technique is referred as oxidation bonding technique. Following this technique various oxide bonded porous SiC ceramics are prepared and characterized [50-101]. The oxidation bonding process is characterized by a low processing temperature and a short sintering time, and is suitable for the preparation of large-size and complex-shaped components. In comparison with the porous SiC prepared by other methods, oxide-bonded porous SiC retains its strength up to very high temperatures and has excellent thermal stability [49]. This method has several advantages compared to the porous SiC processina ceramics fabricated by other techniques. The advantages includes (1) good oxidation resistance because of air sintering of the ceramics (2) low sintering temperature (3) cost effectiveness of raw materials because of coarse staring SiC powders. However, the main drawback of this powder processing route is the agglomeration of the sintering additive and poor dispersion of pore former in the starting materials, which degrades the mechanical and physical properties of the porous ceramics. To avoid these problems colloidal processing route was used to prepare porous SiC ceramics [59].

Sol-ael processina is considered as an alternative approach to fabricate ceramic composites owing to its advantages, such as fine-scale mixing between bond phase additives with ceramic particles and lower processing temperature. Inorganic sol was considered as an effective dispersant for ceramic powders hence enhanced stability of ceramic suspensions by changing their surface potential and also provide high bond strength to ceramic bodies due to the formation of its solid gel at low temperature [96-100]. The addition of a sintering additive via the sol-gel route and a pore former by in situ polymerization was considered as an appropriate choice to overcome the inhomogeniety of the starting particles and expected to enhance and control the properties of the final ceramics. Ebrahimpour et al. used this combination of solgel and in-situ polymerization techniques to fabricate a porous ceramic with the desired filtration properties and oxidation resistant properties [74-77]. Traditional colloidal powder processing methods, are often time consuming, requires careful control over the suspension rheology. Assuming that the porosity of the starting compact is uniformly distributed, an infiltration technique was considered to distribute the secondary oxide bond phase homogenously into the final ceramics with negligible shrinkage and without any crack. Recently an infiltration technique is used for fabrication of oxide bonded porous SiC ceramics in which liquid precursor of secondary bond phase is infiltrated into the porous powder compact of SiC followed by heat treatment [86-88,92-94]. Extensive research work has been carried out to fabricate a wide range of porous SiC ceramics by employing various processing methods. However very limited information about the permeability of porous ceramics are available in the literature [57,81,84,90-91]. Particularly, there have been even less attention to the gas permeability behaviour of porous SiC ceramics with the micrometric pore size [101-110].

The application of SiC membranes for water cleaning and liquid separation processes has received recent attention of the scientific community. It has been proved that SiC microfiltration membranes are extremely suitable for filtration of back wash water, surface water and produced water from oil and gas industries [111-121]. Hagen et al. compared silicon carbide membranes with polymeric membranes for particle removal of prefloculated and prefiltered dam water in pilot scale [112]. The SiC dead-end filters are used for pool water filtration in public

swimming pools [113]. In addition to dead-end filters, tubular cross-flow filters which are especially interesting for filtration of water with high solid loadings, e.g. industrial wastewater or produced water from oil and gas exploration [114,116-118]. It is reported [117] that the SiC membranes have high thermal (up to 800) and chemical (pH 0 - 14), stabilities and they also have very high permeate flux due to high porosity (about 45%). Saint -Gobain's made R-SiC membranes have been demonstrated [118] to effectively clean water from various sources (i) ground water containing As (ii) municipal swimming pool water (iii) produced water from oil and gas exploration. SiC membranes used for such application are mainly fabricated by ceramically-bonded silicon carbide (CSiC), recrystallised silicon carbide (RSiC) or by reaction-bonded silicon carbide (RBSiC). In all these methods SiC is sintered at high temperature with expensive atmosphere and delicate instrumentation and hence the methods are costly. In order to avoid such problems oxide bonding technique is considered as a simple technique for fabrication of macoporous SiC membrane. Very recently S.C. Kim et al. reported fabrication of oxide bonded SiC microfiltration membrane, in which either clay or glass frit are used as bond phase additives [119-120].The literature review indicate that OBSC ceramics have huge potential in air and liquid filtration application.

The objective of this article is to present an overview on processing and properties of porous SiC ceramics by oxidation bonding technique. The effect of processing parameters on the microstructure, porosity, mechanical, thermal shock resistance, oxidation resistance properties, permeability air and particulate filtration characteristics are discussed. Finally, we suggest unresolved issues that should be addressed for future advances in this field.

#### 2. EXPERIMENTAL

Oxidation bonding technique is an unique process for fabrication of porous SiC ceramics at low temperature. Several steps were used, these includes i) powder mixing, ii) pressing/shape making iii) pore former removal and iv) sintering. In the fabrication process commercial SiC powders of variable particle size from submicron to micron size were mixed well with or without pore former ( graphite, starch, PMMA, etc) with or without additives (Al<sub>2</sub>O<sub>3</sub>, MgO, etc) and with or without other oxides (Y<sub>2</sub>O<sub>3</sub>, Sc<sub>2</sub>O<sub>3</sub>, SrO, CeO<sub>2</sub>,

etc) and pressed at a varving pressure of 23-90 MPa. The powder compacts were than heat treated at temperatures ranging from 1100-1600°C in air with hold time 1-4 h. The main composition of raw materials and processing conditions used in the formation of oxide bonded porous SiC ceramics and their material, mechanical properties are summarized in the Table 1. However, the main drawback of this powder processing route is the agglomeration of the sintering additive and poor dispersion of pore former in the starting materials, which degrades the mechanical and physical properties of the porous ceramics. Therefore Ebrahimpour et al. used combination of sol-gel and in-situ polymerization techniques with a reaction bonding process to develop mullite-bonded porous SiC ceramics. Sol-gel processing is a well-known method to fabricate ceramic composite due to the low cost and high homogenous mixing. In addition, in-situ polymerization is a promising technique to synthesize organic-inorganic composites with enhanced mechanical properties. This method was used to fabricate a porous ceramic with the desired filtration and oxidation resistant properties [74-76]. In this method, SiC particles and calcined alumina powders were coated by alumina sol subsequently; polyethylene was layered on particles using in situ polymerization. By heating the green body, polyethylene was removed as a result of the reaction of the polymer with oxygen and consequently pores were created. Mullite was formed by the reaction between alumina and silica derived from SiC during the sintering process to bond SiC particles together.

То achieve significant performance enhancements, good dispersion of the sinterina additive as well as pore former into the starting materials are required. Assuming that the porosity of the starting compact is uniformly distributed, an infiltration technique should result in homogenous formation of a secondary oxide bond phase with the possibility of producing a crack free final ceramics with reduced or shrinkage. Inspired by negligible these possibilities a systematic investigations was carried out on the incorporation of oxide bond precursors into SiC powder compacts by an infiltration based technique. The oxide bond phases that are used include (a) mullite, (b) cordierite and (c) yttrium aluminium garnet (YAG) (d) silica [86-88,92-94]. The reasons behind selection of these bond phase systems are their useful properties ~ high refractoriness, low

oxvaen diffusion coefficient. low thermal expansion coefficient, low dielectric constant, etc. Other important properties are: high strength that can be retained up to a very high temperature, good thermal and chemical stability, in addition having possibility of synthesizing these materials at low temperatures. a-SiC powders of different grit sizes with or without pore formers were mixed in a acetone medium and the mixtures were ball milled for 24 hrs for homogeneous mixing. The powder mixture was dried in air followed by further drying at 60°C for 24 h. The drv powder mixtures was mixed with 15 wt% solution of polyvinyl alcohol which was used as adhesive, then pressed in a hydraulic pressing at 23 MPa gauge pressure to produce rectangular bar samples. The pressed samples dried at 100°C and subsequently heat treated at 1100°C in air medium for 4 h to obtain porous SiC structure with sufficient handling strength after removal of pore former. Porous SiC compacts were kept into an evacuator and and kept for two hours under the evacuated condition. The precursor sol was added through an inlet which was directed to disperse the sol into the porous compacts under the high capillary action of pores of bulk SiC compacts. After sol incorporation the samples were taken out from the sol and kept for dry under RT and then at 100°C for 24 hours. During this step, the incorporated sol was transformed into gel state due to evaporation of solvent part. Infiltration technique was repeated for several times until the weight gain of sol-gel incorporated porous SiC compacts became constant. The final saturated sol-gel incorporated SiC compacts were sintered at 1300-1500°C to obtain porous SiC ceramics bonded by secondary oxide bond phases.

In another approach mullite- and YAG- bonded porous SiC ceramics was also prepared using sol-gel coated or co-precipitated method. SiC powder suspension was prepared by dispersing a fixed wt% of SiC powders in de-ionized (DI) water followed by ultra-sonication for 30 minutes. Secondary bond phases in the form of sol precursor were added to the SiC powder suspension, the colloidal mixture was stirred for another one hour. The amount of bond phase precursor sol was adjusted in such to yield a definite wt% of secondary bond phase in the final ceramics. The SiC powders dispersed in mullite sol maintained at pH~2 and at 7 to obtain mullite coated SiC powder and SiC co-precipitated with mullite respectively. The SiC powders dispersed in YAG sol was maintained at pH~9 by adding 1 (N) solution of ammonia to obtain precipitation of colloidal particles of YAG on the surface of SiC particles. The coated and co-precipitated powders were filtered, washed four times with DI water and dried at 100°C for 24 hour. The dry powders were then calcined at a particular temperature to remove volatile components. The calcined coated or co-precipitated powders were sieved using sieving net to make uniform powder size and then pressed in bar shape. Fig. 1 shows the schematic processing of porous SiC

ceramics by sol-gel coating method. The bar samples were dried at 100°C for 24 hours and finally sintered at 1300°C for mullite system and at 1450°C for YAG system. Oxide bonded porous SiC ceramics (OBSC) were named as SBSC, MBSC, CBSC and YBSC for silica, mullite, cordierite and YAG as bond phase respectively. The effect of different secondary bond phases on the microstructure of porous SiC ceramics are presented in Fig. 2.

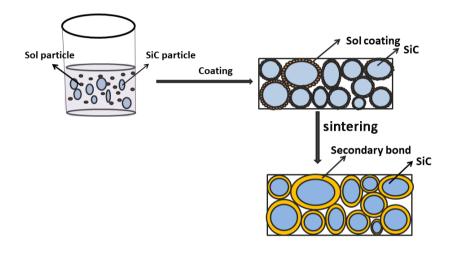


Fig. 1. Schematic of fabrication porous SiC ceramics by sol-gel coating process

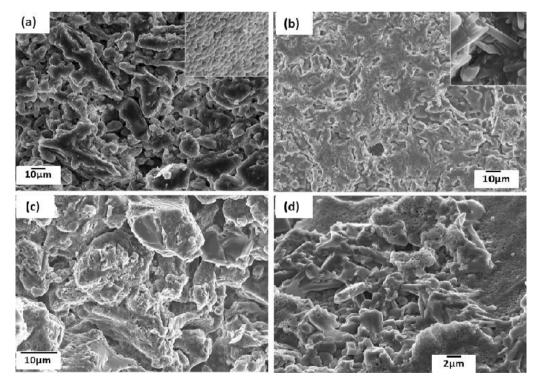


Fig. 2. Effect of secondary bond phases (a) silica (inset shows fish scale morphology of cristobalite) (b) mullite (inset shows needle shape morphology of mullite) (c) cordierite and (d) YAG on the microstructure of porous SiC ceramics

#### Table 1. Effect of addition of other oxide on material and mechanical properties of porous OBSC ceramics

SiC (d₅₀ in µm)	Bond type/ [Ref]	Additives (d₅₀ in µm)	Pore former	Sintering temperature (°C)	Porosity (vol%)	Pore diameter (µm)	Flexural strength (MPa)
0.6-2.3	Silica [46]	-	-	1100-1400/1 hr	28-41	-	60-190
2.3	Mullite [49]	Al <sub>2</sub> O <sub>3</sub> (0.2)	Graphite (5,10 and 20)	1450/0.5-21hr	36-75	2.5-12	10-52
0.3 to58	Silica [49]	-	Graphite (5,10 and 20)	1450-1500/1-5 hr	31-36	2.5-12	40-133
0.34	Silica [50]	-	Microbead(20)	1400/1hr	40	25-30	65
20	Mullite [51-53]	$AI_2O_3$ (0.6) and $Y_2O_3$ (5.0)	Graphite (10)	1300-1550/4hr	34-57	1.6-8 (Bimodal)	5-28
20	Cordierite [62]	Al <sub>2</sub> O <sub>3</sub> , talc, clay	Graphite	1360/2hr	28-60	1.9-4.9 (Bimodal)	10-55
10	Cordierite [60]	Al <sub>2</sub> O <sub>3</sub> , (0.6) MgO	Graphite (5,10, 20)	1200-1400/2-4hr	28-66	2-8	3-71
10	Cordierite [63]	$AI_2O_3$ (0.6) MgO, CeO <sub>2</sub>	Graphite (10)	1250-1400/2hr	28-56	2.4-3.2	4-36
10	Mullite [65]	$AI_2O_3(0.6)$ preheat-treated aluminosilicate	Graphite (10)	1350–1550/ 2 hr	35-53	1.6-4	14-87
90 and <b>0.5</b>	Mullite [66]	Al <sub>2</sub> O <sub>3</sub> , (0.4) MgO, CaO, SrO	C template (20)	1450/2	44-49	-	6-44
		2 = 0, (= )			-		Superior st, SrO
65 and <b>0.5</b>	Mullite [67]	AI, ALN, $AI_2O_3$ , $AI(OH)_3$	-	1450-1500/1-2 hr	35-46	-	5-19kNm/kg
65	[72]	AI(OH) <sub>3</sub>	-	1450-1500/1-2 hr	46-54	-	3-14
65, 0.7	SiC bonded [70]	Si (1)	Carbon black	1700-1800/1 hr in Argon	38-46	5-25	3-42
20	SiC-cordierite composite [55]	Clay, talc, alumina	Graphite	1200-1400/2 hr	27-60	-	9.3-54.6-26
10	Sodium borate [73]	$Na_2B_4O_7$	Starch	650-800/30 min	47-64	15-25	3-37
2.4	Mullite [78]	Porous Alumina column (37.3)	Graphite (18.7)	1300-1500/3hr	30-47	-	22-56
23	Cordierite-mullite [80]	Al <sub>2</sub> O <sub>3</sub> (11.7) MgO,	Graphite (18.7)	1300-1450/3hr	18-50	2.98	13-64
125-7	Glass [81]	Glass (61 wt.% SiO <sub>2</sub> , 24% Al <sub>2</sub> O <sub>3</sub> , 5% CaO, 2%Na <sub>2</sub> O, 2% K <sub>2</sub> O, and 6% other materials)		850/1hr	32-65	6-127	-
22.8	Cordierite [82]	Diatomite (4.2) Talc (2.6) $Al_2O_3$ (27.3)	Graphite (18.7)	1150-1400/3-7 hr	27-46	3	25-65
22.4	Silica [85,90]	Silica sol (with and without)	-	1300/4hr	26-36	5-7	29-48
22.4	mullite	$AI_2O_3$ and Silica	-Petroleum coke (11)	1500/4hr	30	5	47
	[86]	Only Al <sub>2</sub> O <sub>3</sub> (6.5)		1500/4hr	29-56	6-9	9-34
22.4	Cordierite [91]	Al <sub>2</sub> O <sub>3</sub> (6.5) MgO,	Petroleum coke (11)	1350/4hr	32-68	6-50	5-54
4.5-99	Mullite [87-88]	Mullite sol	Graphite (12)	1300-1500/4 hr	31-48	2-30	16-49
22.4, 53,	Cordierite [93-94]	Cordierite	Graphite (12)	1300-1400/3 hr	30-49	4-22	13-38
99		sol	Microbead (8,50)			· ===	
22.4, 53	yttrium aluminum garnet (YAG)		Graphite (12)	1300-1500/3 hr	29-41	5-30	8-41
99	[92]	sol	Microbead (8,50)				

Microbead = poly(methyl methacrylate-co-ethylene glycol dimethacrylate)

#### **3 RESULTS AND DISCUSSION**

#### 3.1 Mechanism of Bond Formation

SiC oxidizes during thermal heating with the formation oxides of silicon. Depending on the oxygen potential this oxidation may be active with the formation of SiO or, passive with the formation of SiO<sub>2</sub> [122-123]

$$SiC + O_2 = SiO + CO_2$$
(1)

$$\operatorname{SiC} + \operatorname{O}_2 = \operatorname{SiO}_2 + \operatorname{CO}_2 \tag{2}$$

The active oxidation is associated with a critically low partial pressure of oxygen, where as adequate supply of oxygen at the reaction front ensures passive oxidation with the formation of protective SiO<sub>2</sub> scale and gain in weight. This SiO<sub>2</sub> may be vitreous at the formation temperature which under suitable heat treatment conditions devitrifies to the cristobalite phase. In a porous body the percolation threshold for permeation of molecular oxygen is around 16-22 vol.% [124-125]. If the porosity can be maintained above this percolation threshold limit, molecular oxygen permeates to oxidize SiC particles with the formation of SiO<sub>2</sub>. The silica scale under controlled heat treatment conditions converted to cristobalite that join the neighbouring SiC particles as shown in Fig. 2a. Cristobalite with fish-scale morphology was observed in the microstructure which was also in silica bricks [126-127]. observed The cristobalite has the characteristic fish-scale morphology arises from the grain boundaries of the original quartz grains [126]. Oxides like alumina, magnesia, etc., were mixed with starting SiC powder for the formation of other oxide bond like mullite, cordierite at low temperature which led to change in material and mechanical properties. In presence of Al<sub>2</sub>O<sub>3</sub>, there is a strong thermodynamic possibility of formation of mullite  $(CTE = 5.4 \times 10^{-6})^{\circ}C)$  which has nearly matching coefficient of thermal expansion with SiC (CTE = 4.7 x  $10^{-6}$ /°C) .The oxidation derived SiO<sub>2</sub> reacts with alumina to form mullite at a temperature over 1410°C. The mullitization between SiO<sub>2</sub> and  $\alpha$ - Al<sub>2</sub>O<sub>3</sub> is explained by solution precipitation mechanism. SiO<sub>2</sub> does not form a viscous liquid phase at 1400°C but shows a viscous softening. Due to the superfacial softening, the  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> particles penetrate into viscous SiO<sub>2</sub> glass, leading to the nucleation of mullite. At 1450°C, the viscosity of SiO<sub>2</sub> glass decreases and more  $AI^{3+}$  ions gets dissolved into viscous SiO<sub>2</sub> glass. Above 1500°C, the mullite formation occurs by the reaction between cristobalite and  $\alpha$ - Al<sub>2</sub>O<sub>3</sub>.

Because of short diffusion distances achieved by the viscous flow assisted sintering, the rate of mullitization is accelerated drastically [128-129]. Formation of mullite during sintering of porous SiC is described by following equations.

$$SiC + 2O_2 \rightarrow SiO_2$$
 (amorphous) +  $CO_2$  (3)

$$AmorphousSiO_2 \rightarrow Cristobalite$$
(4)

$$3Al_2O_3 + 2SiO_2 \rightarrow 3Al_2O_3.2SiO_2$$
 (mullite) (5)

After enough mullite is formed, the interfaces of Al<sub>2</sub>O<sub>3</sub> -mullite appear. SiO<sub>2</sub> SiO<sub>2</sub>-mullite and and  $\alpha$ - Al<sub>2</sub>O<sub>3</sub> inter-diffuse across the mullite layer and the formation of mullite is diffusion controlled [129]. It has been proved that with increasing amount of alumina open porosity decreases, and the flexural strength increases significantly. Sintering at 1300°C or above resulted in the formation of needle shaped mullite phase between the SiC particles at the contacting points as shown in Fig. 2b. The needle shaped morphology of mullite phase has been reported by several researchers [130-131] The mullite bond phase was seen to be of ~2-4 µm in width and 20-40 µm in length in the ceramics prepared by infiltration of mullite precursor sol [91]. Lee et al. obtained needle-like mullite phase of 1 - 2 µm in length and 0.1 - 0.3 µm in width by sintering at 1300°C for 2 hours; the length and width of the mullite phase had grown to 10 - 20 µm and 1 - 2 µm respectively when the sintering temperature was raised to 1600°C [131]. Average activation energy of mullite was obtained as ~1200-1275 kJ mol<sup>-1<sup>-1</sup></sup> produced through reaction of powder sample of Al<sub>2</sub>O<sub>3</sub> and oxidation derived SiO<sub>2</sub> from SiC [132].

If MgO is present, there is a possibility of formation of cordierite  $(2MgO.2Al_2O_3.5SiO_2)$ ; cordierite may be another important oxide bond phase due to its low thermal expansion coefficient (~1.5 x 10<sup>-6</sup>/ °C) and low cost [133-134]. During heat treatment of powder compacts of SiC,Al\_2O\_3 and MgO, silica reacts with MgO and Al\_2O\_3 to form cordierite below1350°C. Sintering of SiC is described by series of reactions as follows:

$$MgO + Al_2O_3 \rightarrow MgO.Al_2O_3(spinel)$$
 (6)

$$2M_gO + 2Al_2O_3 + 5SiO_2$$

$$\rightarrow 2M_gO.2Al_2O_3.5SiO_2 (cordierite)$$
(7)

$$2(M_g O.Al_2 O_3) + 5SiO_2$$

$$\rightarrow 2M_g O.2Al_2 O_3.5SiO_2 (cordierite)$$
(8)

The mechanism of formation of cordierite (Mg<sub>2</sub>Al<sub>4</sub>Si<sub>5</sub>O<sub>18</sub>) from mixed oxides is well documented [135-137]. At around 1250°C MgO and Al<sub>2</sub>O<sub>3</sub> of the spinel dissolves into SiO<sub>2</sub> and subsequently turns into  $\alpha$ -cordierite (orthorhombic structure) from the solid solution at its limit of solubility. It is reported that reaction between spinel and SiO<sub>2</sub> proceeds via the formation of a µ-cordierite phase (a low temperature metastable modification of cordierite with hexagonal structure) as а stuffed derivative of β-quartz phase. From TG-DTA analysis of powder compact of SiC+ Al<sub>2</sub>O<sub>3</sub> + MgO, Liu et al. observed three exothermic peak at 1255.4. 1292.3 and 1346.7 °C [62]. The peak near 1255.4 °C was explained due to the formation of  $\alpha$ -cordierite, which was also confirmed by the phase analysis of the samples sintered at different temperatures. They also found that the increase of  $Al_2O_3 + MqO$  additions results in more cordierite, spinel and Mg-Al-Si-O glass. Cordierite and spinel can enhance the strength of porous SiC ceramics, but the strength is impaired when excessive Mg-Al-Si-O glass exists in the porous ceramics, because of fragile nature of glass. Microstructure indicated well necks formation developed made of reaction-derived and cordierite silica as shown in Fig. 2c. At higher magnification spherical-, and lethal shaped morphologies are seen and EDS analysis indicated presence of Mg, Al, Si and O elements which supports the cordierite phase formation [93]. The cordierite crystals were found to be 3-5 µm in length and 0.5-1.0 µm in width and spherical shaped cordierite was found be of ~1 µm diameter. Moftah El-Buaishi et al. also observed the spherical shaped cordierite crystals of dimension 1 µm and some agglomerated particles of ~10 µm [138]. For MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> cordierite-based glasses activation energy crystallization is estimated by many authors and found to vary over a wide range, ~ 270 to 630 kJ mol<sup>-1</sup> [139-141]. Average activation energy of  $\mu$  and  $\alpha$  phase of cordierite was obtained as 644.22±33.33 and 849.75±24.34 kJ mol<sup>-1</sup> produced through reaction of powder sample of Al<sub>2</sub>O<sub>3</sub>, MgO and oxidation derived SiO<sub>2</sub> from SiC [91]. The micrographs of YAG incorporated ceramics showed fish-scale, spherical-, rod or needle- shaped morphologies for silica, YAM or YAP and Y<sub>2</sub>Si<sub>2</sub>O<sub>7</sub> phases respectively as shown in Fig. 2d.

#### 3.2 Effect of Other Additives

In order to lower the sintering temperature of porous SiC ceramics, alkaline earth metal oxides, Y2O3 has been chosen as sintering additive as they have lower eutectic temperatures  $Y_{2}O_{3} AI_2O_3$ – $SiO_2$ (1360°C),  $Sc_2O_3 - Al_2O_3 - SiO_2$  (1450°C), MgO-Al\_2O\_3 - SiO\_2 (1355°C) and CaO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> (1170°C) SrO-SiO2 (1358°C) [142-145] when Al<sub>2</sub>O<sub>3</sub> exists, SrO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> can form eutectic liquid at lower temperature than 1358°C. Addition of small amount of Sc<sub>2</sub>O<sub>3</sub> accelerated the rate of solid state diffusion to form a ternary Sc-rich aluminosilicate, resulted enhanced mullite formation at temperatures between 1350 and 1550°C [142]. Choi et al observed that in presence of SrO the flexural strength of MBSC ceramics was improved because the addition of SrO formed a denser strut than the other alkaline earth metal oxides (MgO, CaO) [66]. In the powder processing route the cordierite phase was accompanied by the phase of spinel (MgO·Al<sub>2</sub>O<sub>3</sub>), which impaired the thermal shock resistance of porous SiC ceramics due to the high thermal expansion coefficient of spinel. To avoid this problem Liu et al. used CeO<sub>2</sub> as sintering aids and observed that with addition of 2.0 wt.% CeO<sub>2</sub> a large amount of cordierite but only a trace of spinel at 1250°C. Due to the enhancement of neck growth by the addition of CeO<sub>2</sub>, the porous SiC ceramics exhibited better mechanical properties than the ceramic prepared without CeO<sub>2</sub> [63]. Typical microstructure of mullite bonded porous SiC ceramics with addition of other additives are shown in Fig. 3 which exhibits strong neck formation with connected porous structure.

Fig. 4 shows the XRD pattern of silica-, mulliteand cordierite bonded SiC porous ceramics. For silica bonded porous SiC ceramics the main peaks were due to SiC and cristobalite. In case of mullite bonded porous SiC ceramics SiC, cristobalite, mullite was detected as the major crystalline phases. Similarly for cordierite bonded porous SiC ceramics peaks of cordierite were detected along with major peaks of SiC and cristobalite.

#### 3.3 Effects of Particles Size of SiC and Pore Former and on Porosity and Pore Diameter of Final Ceramics

For application of porous ceramics as gas filter, a desired pore size is needed to achieve high particulate filtration efficiency. This can be easily

obtained by oxidation bonding process through incorporation of various pore forming agent like. saw dust, rice husk, starch, yeast, graphite, organic particulates, etc. During heating in air these particulates burn out, leaving pores in the OBSC ceramics. For oxide bonding of porous SiC ceramics polymethylmethacrylate (PMMA; microbeads), sodium bicarbonate, graphite pore formers are commonly used. Chun et al. reported variation of porosity in a range of 19 to 77 vol.% using microbead (of  $d_{50}=20 \ \mu m$ ) as pore former in SBSC ceramics simply by the variation of amount of the pore former [50]. Gnesin et al. prepared highly porous SiC ceramics (with porosity up to 70%) by the use of varied amount of sodium bicarbonate pore former [146]. She et al. used SiC particle of size 0.3 µm, observed average pore diameter of 3.4, 5.3 and 7.6 µm on using graphite of particle sizes 5, 10 and 20 um respectively [48]. They calculated the pore diameter theoretically from the schematic diagram using following equation and the result matched excellently with the experimental results.

Ding et al. noticed change in pore size distribution pattern from monomodal to bimodal

with increase of graphite particle size from 5 to 20 µm, with an average pore diameter 1.6 and 8.0 µm [53] respectively. It was explained that since 20.0 µm SiC particles were used, the size of the pore by stacking of SiC particles should be theoretically 8.3 µm, provided that spherical SiC particles are in dense cubic stack. When graphite particles of 5 and 10 µm were used, the sizes of the pore by burning out of graphite closely match with pores by stacking of the SiC particles, so that there is no remarkable difference between them in the average pore diameter. When graphite particle increased to 20 µm, the pores formed by burnout of graphite are much larger than those from stacking of SiC particles; as a result bimodal pore size distribution was obtained. CBSC ceramics showed variation of porosity from 28 to 66 vol% with increase in amount of pore former fraction from 0 to 0.60 prepared from powder mixture containing SiC, Al<sub>2</sub>O<sub>3</sub> MgO and graphite. It is also reported that with the increase in graphite particle size from 5 to 20 µm pore diameter increased from 1.2 to 7.5 µm but porosity remained almost same on keeping the amount of pore former constant [62]. On the other hand, the pore diameter of OBSC ceramics was found to be strongly dependent on

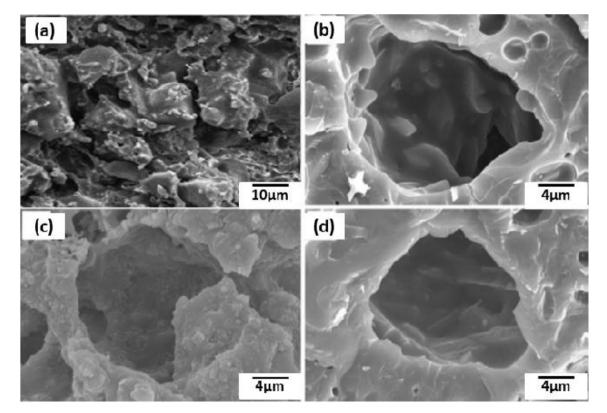


Fig. 3. Typical SEM micrograph of porous mullite bonded SiC ceramics prepared with (a) 1.5 wt.% Y<sub>2</sub>O<sub>3</sub> sintered at 1450°C for 4 h [51] (b) 4 wt% MgO (C) 4 wt% CaO and (d) 4 wt% SrO sintered at 1400°C for 2 hr [66]

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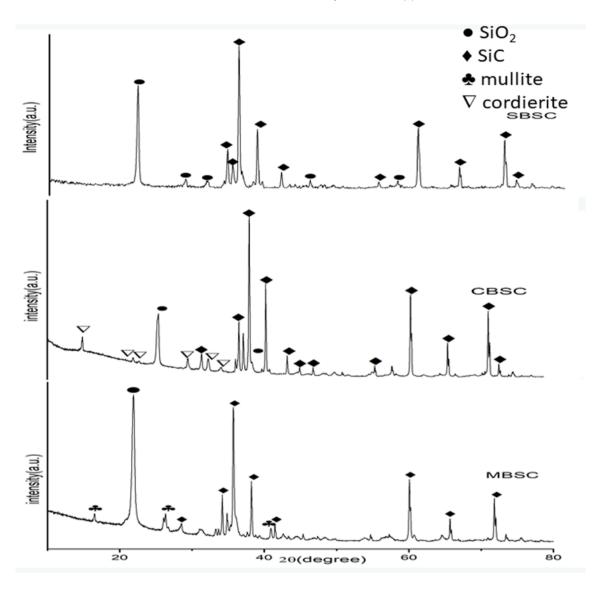


Fig. 4. XRD pattern of silica-, mullite- and cordierite bonded SiC porous ceramics

the size of the starting powders. She et al. reported mean pore diameters of 2.5, 3.5, 7.2 and 11.6 µm for the samples prepared using 6-, 10-, 27- and 58-µm SiC powders [49]. Baitalik et reported mean pore diameter of 3.5, 10.6 and 22.3 µm and 4.8, 11.9 and 21.5 µm for the cordierite-infiltrated porous SiC samples prepared using 22, 57 and 99 µm SiC powders sintered at 1300 and 1400 °C respectively [93]. With increase of porosity from 29 to 56%, pore size increased from from 5.5 to 10.6  $\mu$ m as reported for mullite bonded porous SiC ceramics [90]. Similar observations were made for the cordierite bonded porous SiC ceramics, with increase of porosities from 30.2 to 55% pore size changed from 5.5 to 17.9 □m with further increase of porosity to 55%, the pore size increased to ~49 µm [91].

Eom et al. studied [72] the effect of incorporation of submicron size SiC powder (in addition to the coarse SiC powder) on the microstructure, densities and mechanical properties in case of reaction sintering mullite bonded SiC ceramics. The porosity was increased with increase in amount of pore former due to the increased number of pores as shown in Fig. 5. The average pore diameter did not changed significantly with amount of pore former, but they get changed significantly with changing pore diameter of the starting pore former. Hence, porosity, pore sizes can be controlled by selecting the size, amount and type of pore former, sintering temperature, size of starting SiC powder, sintering additives and bond phases in the final ceramics.

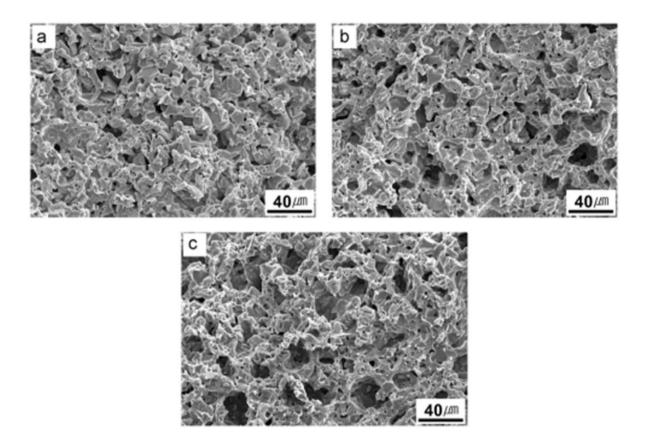


Fig. 5. Effect of starch content on the microstructure of porous SiC ceramics (a) 40 (b) 43 and (c) 46 wt%. [73]

#### 3.4 Effect of Sintering Temperature

Sintering temperature and hold time are other important processing parameter for oxide bonded porous SiC ceramics. The heat treatment temperature has significant effects on % SiC oxidation degree, porosity and pore size distribution of the porous SiC ceramics. A gradual gain in mass was observed at >1000°C in the thermogrametric analysis of the powder mixtures, because of on set oxidation of SiC. Although SiC might began to oxidise at >800°C, it became noticeable only at 1100°C [147]. During sintering of the SiC compact, SiC get oxidized forming silica. The degree of oxidation (f) of SiC can be expressed as [148]:

$$f = 2C_M \times 100 \tag{9}$$

where  $C_M = (M_S' - M_S)/M_S$ ,  $M_S'$  and  $M_S$  being the mass of SiC before and after oxidation. The oxidation degree of SiC depends on several factors (i) sintering temperature (ii) particles size of the SiC powder (iii) amount of additives (Al<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub>+MgO) (iv) addition of other oxides like Y<sub>2</sub>O<sub>3</sub>, CeO<sub>2</sub>, alkaline earth metal oxides, etc. It is

reported that oxidation degree of SiC rises steeply with increase in sintering temperature and hold time. She et al. observed [43] sharp rise of vol% of SiO<sub>2</sub> from 17.0 to 31.9 with increase in sintering temperature from 1100 to 1400°C. Similarly Ding et al. observed [48] increase in f from 11.2 to 33.4% with increase in temperature from 1400-1550°C. With increase of temperature, the diffusion rate of oxygen toward SiC through SiO<sub>2</sub> film is accelerated in the early stage of the sintering so that more oxygen can further react with SiC to form SiO<sub>2</sub>. Furthermore, the higher sintering temperature can speed up the viscous flow of oxidation derived SiO<sub>2</sub> [58]. Liu et al. [62] noticed with increase in graphite content from 0.25 to 0.6 (weight fraction) oxidation degree sharply increased from 30 to 49 % keeping the other processing parameter constant for bonded porous SiC ceramics. cordierite Oxidation degree was also found to be influenced by the particle size of the pore former. With increase of size of the pore former from 5 to 20 um oxidation degree was found to reduce from 30 to 26.7%. It is proved by several authors that the oxidation degree of SiC is almost independent on the amount of pore former but it strongly depends on the particle size of SiC [46].

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Eom et al. observed 2.3 mass% of SiO<sub>2</sub> on oxidation of 65  $\mu$ m- SiC at 1550°C for 2 h, whereas the oxidation of 0.5  $\mu$ m- SiC under the same conditions resulted in 48.1 mass % of SiO<sub>2</sub> [71]. In mullite bonded porous SiC ceramics prepared by infiltration route at 1500°C the % SiC oxidation degree was reduced from 65 to 7 % with increase of particle size from 4.47 to 99  $\mu$ m [96]. A similar trend was also observed for samples prepared at 1300°C, where the % SiC oxidation degree decreased from 40 to 3% with increase of particle size from 4.47 to 99  $\mu$ m.

For silica bonded SiC Ceramics the value of f varied from 12.2 to 13.06 for variation of fraction of pore former from 0.14 to 0.38 [84]. Mullite bonded SiC ceramics showed variation of f from 28-36.76 for variation of fraction of pore former from 0.15 to 0.51 [90]. Similarly with variation of fraction of pore former from 0.23 to 0.76 the cordierite bonded porous SiC ceramics showed variation of f from 22 to 39 [91]. It was observed that sample showed no significant changes in f when the pore former volume fraction was not very high. It is therefore likely that when the amount of pore former was not very high, the pore or void space that were created by burning of sacrificial pore former were not able to influence the process of SiC oxidation significantly, and consequently the % SiC oxidation degree showed no significant changes. But when the pore former amount was significantly higher, the pores or void spaces created by the pore formers were also higher which created higher surface area for SiC particles for oxidation and hence f was increased. Quanli et al. [149] also observed an increase in weight and rate constant and decrease of oxidation activation energy with decrease of particle size of SiC powders. In case of MBSC ceramics, decrease in f was observed with increase in amount of alumina in the starting powder mixture. This was because the coverage of surfaces of SiC particles by some fine alumina powders leading to the reduction in the surface area and thus f was reduced [48]. Same was observed during formation of CBSC also ceramics, where the oxidation degree of SiC was found to decrease from 48.4 to 42.5% with increase in weight fraction of Al<sub>2</sub>O<sub>3</sub> and MgO from 0.1 to 0.3 [62]. It was noticed that the oxidation degree of SiC increases significantly with addition of  $Y_2O_3$ ,  $CeO_2$  and alkaline earth metal oxides [63,66]. This was due to the formation of glass of various compositions at low temperature. Ding et al. noticed [51] an increase of % oxidation degree of SiC from 24 to 35% for MBSC ceramics prepared with 1.5 wt %  $Y_2O_3$  at 1500°C. With increase of oxidation degree amount of cristobalite formation increased and porosity decreased and hence mechanical properties improved to some extent.

# 3.5 Mechanical Properties of OBSC Ceramics

The mechanical strength of porous ceramics strongly on porosity and depends the microstructure. Therefore, the factors controlling the porosity are indirectly controlling the mechanical strength. For the porous SiC ceramics the factors affecting the strength includes the grain size, bonding necks between particles, bond phases, porosity, pore size distribution, pore shapes and flaws. The main parameters are the porosity and the bonding necks. The porosity depends on sintering temperature, forming pressure, content of pore former, etc., while bonding necks could be reflected from the densification of porous SiC ceramics. Table 1 shows that the flexural strength of oxide bonded porous SiC ceramics decreases with increase of porosity irrespective of the type of bond phase.

According to the minimum solid area (MSA) model [150], the strength of a porous material is related to its porosity by the expression:

$$\sigma = \sigma_{o} \exp (-bP)$$
(10)

where  $\sigma$  is the strength of a material of porosity P,  $\sigma_o$  is the strength of the completely dense material and b is a constant that depends on the shape and alignment of the pores. For the OBSC ceramics, the relation between strength and porosity of the ceramics sintered at a given temperature fits well with the Eq. (1) [47,48,50]. Strength decayed exponentially with increase in porosity as shown in Fig . 6. By fitting equation 1 with the plot of flexural strength vs. porosity, the values of  $\sigma_o$  and b are obtained. The value of  $\sigma_o$  and b obtained by different authors for oxide bonded porous SiC ceramics are summarized in Table 2.

For oxide bonded porous SiC ceramics with different oxide bond phases, different values for b could be estimated from the strength-porosity relationship. She et al. obtained the value of b = 6.5 to 7.1 for SiO<sub>2</sub>-bonded porous SiC ceramic materials with porosity varying between 28 and 41 vol% [47]. Chun et al. reported still higher

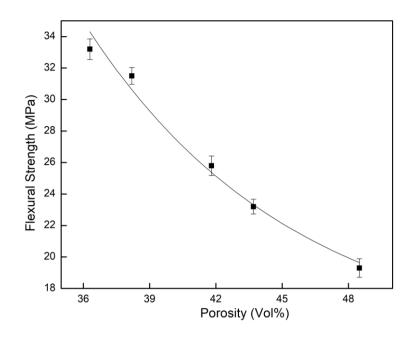


Fig. 6. Plot of strength vs. porosity for mullite bonded porous SiC ceramics [87]

value of b = 7.5 to 8.0 for the same material with porosity ranged from 19 to 77 vol.% [50]. Cordierite bonded porous SiC ceramics of porosity varying in the range of 28 to 60 vol% exhibited a value of b = 4.8 [55]. She et al. changed the porosity of the MBSC by varying the green density through the employment of different die pressing pressure, keeping the composition and sintering temperature and other processing parameter same and they reported value of b = 4.36,  $\sigma_0$  = 190 MPa [48]. The cordierite bonded porous SiC ceramics prepared by conventional powder mixing method showed  $\sigma_0$  =132 MPa and b=3.1 [82]. Bai et al. reported *b*=8.16 and  $\sigma_0$ =1237.04 MPa for cordieritemullite bonded porous SiC ceramics [80]. Liu et al. reported  $\sigma_0$ =501 MPa and b=6.97 for cordierite bonded porous SiC ceramics prepared using 10 µm graphite powders as pore former [62]. Literature review indicated that processing conditions have a great impact on b and  $\sigma_0$ values of final porous SiC ceramics. The values of b were reported to be 6 and 9 for cubic and rhombic stacking respectively [151]. The results of Table 2 indicated that the exponential factor 'b' did not change significantly with sintering temperature and oxide systems and the pore shape remained predominantly cylindrical in the final ceramics sintered at different temperatures. The results also indicated that OBSC ceramics followed the established strength-porosity dependence model and it is possible to alter their porosities to obtain desired level of strength values.

The Young's modulus is theoretically predicted by a relationship similar to the expression given in Eq. (1) [152]

$$E = E_o \exp (-bP)$$
(11)

where E is the Young's modulus of a material of porosity P,  $E_o$  is the Young's modulus of the completely dense material and b is a constant that depends on the shape and stacking of the pores. By using the value of b obtained from Eq. 1, phase compositions of the sintered ceramics and the values of Young's moduli of the phases for a particular porosity level, the  $E_o$  values of the sintered ceramics can be approximated by the rule of mixture. The Young's modulus ( $E'_o$ ) of a completely dense ceramic material can be approximated by the rule of mixture [153]

$$E'_{o} = E'_{1}V_{1} + E'_{2}V_{2} + E'_{3}V_{3} +$$
(12)

where  $E'_1$ ,  $E'_2$ ,  $E'_3$ ,..... are the Young's modulus of the different constituent phases 1, 2, 3, .... of the dense ceramics and  $V_1, V_2, V_3$ ,..... are their respective fractional volume. The  $E'_o$  value were estimated using the values of Young's moduli of SiC [450 GPa], mullite [150 GPa], cordierite [70 GPa], cristobalite [72 GPa] glassy phase [72 GPa] [153-157] and their corresponding amount determined from X-ray and chemical analyses.

Ref.	Type of bond phase	σ₀(MPa)	b	Porosity (Vol.%)	Strength (MPa)
47	silica	-	6.54 - 7.08	41-28	100-185
48	silica	190	4.36	28-45	52-26
50	Silica	-	7.95	40	65
55	SiC-mullite composite	213.4	4.8	27.6-59.6	9.3-54.6
58	mullite	-	-	30-42	5-23
62	cordierite	501	6.97	66-28	3-70.3
71	mullite	167	4.89	35-42	13-38
78	Mullite	140	3.74	33-47	24-41
80	Mullite-cordierite	1237	8.16	37-53	15.6-55.4
82	Cordierite	132	3.1	22-45	30.9-65
88	mullite	181	4.6	36-49	19-33
90	Mullite	192	5.79	33-54	10-34
91	Cordierite	270	5.13	31-69	6-54
93	Cordierite- silica	195	5.05	30-36	13-38

Table 2. Porosity, flexural strength, b (the empirical constant) and  $\sigma_o$  (the strength of corresponding non porous structure) of oxide-bonded porous SiC ceramics

To reduce sintering temperature and increase mechanical strength, various oxide additives along with alumina was added. The MBSC ceramics prepared from 20 µm SiC powder using Y<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> as additives resulted a flexural strength of 28 MPa at 44 vol% porosity and without Y2O3 addition resulted 24 MPa at 43 vol% porosity [54]. The addition of 4 wt% of alkaline earth metal oxides (SrO) found to improve mechanical strength of MBSC ceramics from 9 MPa at 49% poroity to 44 MPa at 46% porosity [66]. Mullite-bonded porous SiC (d<sub>50</sub>=65 µm) ceramics prepared at 1500°C using 47.3 wt% AI(OH)<sub>3</sub> powder showed flexural strength of 14 MPa at 47 vol% porosity [69]. Significant improvement of flexural strength was obtained with addition of submicron SiC powder with coarse SiC powder [72]. Ebrahimpour et al. reported 30% enhancement of mechanical strength on replacement a portion of alumina powder by sol-gel alumina precursor due to increase of homogeneity. Depending on the processing parameters the reported flexural strength values of mullite bonded porous SiC ceramics using alumina powder as additives were 9.8 MPa at 50 vol% porosity, 24 MPa at 43 vol% porosity, 39 MPa at 36 vol% porosity and 45.49 MPa at 29.54 vol% porosity [53-54, 57]. The cordierite-bonded porous ceramics prepared from powder mixture of  $\alpha$ -SiC (d<sub>50</sub> 10  $\mu$ m),  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, MgO and graphite (as pore former) by reaction bonding technique showed variation in flexural strength of ~70-3 MPa with variation of porosities ~28-65.6 vol% final in SiC ceramics[62]. With the increase of weight fraction of MgO+Al<sub>2</sub>O<sub>3</sub> mixture from 0.2 to 0.3, the final SiC ceramics prepared at 1350°C showed a decrease in flexural strength due to formation of more Mg-Al-Si-O glass which made ceramics more fragile due to brittle nature. With addition of  $CeO_2$  in the system an improvement of strength was obtained due formation of increased amount of cordierite phase with lesser amount of spinel phase [63]. The CBSC ceramics prepared by infiltration of cordierite precursor sol into powder compact of SiC ( $d_{50}=22 \ \mu m$ ) sintered at 1400°C showed flexural strength of 38.3 MPa at 30.6 vol.% [93-94] . Li *et al.* recommended oxide bonded porous SiC as suitable candidate for hot gas filtration application as support material. They studied the effect of the moulding pressures, SiC particle sizes and oxide bond phase contents on mechanical strength of porous SiC ceramics [71].

#### 3.6 Gas Permeation Behaviour

The gas permeability behaviour of the porous SiC ceramics can be well explained by Forchheimer's equation, the parabolic dependence of pressure drop ( $\Delta P$ ) through a flat medium with the resulting face velocity (v<sub>s</sub>) of the fluid [158]:

$$\frac{\Delta P}{L} = \frac{\mu}{k_1} v_s + \frac{\rho}{k_2} {v_s}^2 \tag{13}$$

For compressible flow of gases and vapors,  $\Delta P$  can be calculated from:

$$\Delta P = \frac{P_i^2 - P_o^2}{2P}$$
(14)

in which  $P_i$  and  $P_o$  are absolute fluid pressures at the entrance and exit of the medium [Pa], L is the medium length or thickness(mm),  $\mu$  is the

fluid viscosity [Pa.s] and  $\rho$  is the fluid density [kg m<sup>-3</sup>].

The terms  $k_1$  and  $k_2$  are the Darcian and non-Darcian permeability coefficients or constants, in reference to Darcy's law, which established a linear dependence between pressure drop and [159-161]. The permeability fluid velocity parameters obtained for the porous OBSC ceramics are presented in Table 3. Ding et al. [110] obtained variations in Darcian and non-Darcian permeability from 8.5 – 9.9 x  $10^{-13}$  m<sup>2</sup> and  $1.4 - 3.5 \times 10^{-6}$  m respectively for a variation in porosity in a narrow range of 56-61.5% for MBSC ceramics [110]. Liu et al. [69] measured the permeability parameters of cordierite bonded porous SiC ceramics at two porosity levels, viz. 39 and 53% and obtained the Darcian permeability 0.71 x  $10^{-13}$  and 0.88 x  $10^{-13}$  m<sup>2</sup> respectively with the nearly same non-Darcian permeability  $\sim 0.02 \times 10^{-6}$  m. The permeability behaviour of porous SiC ceramics was also compared with that of porous oxide ceramics [162]. From permeability studies it is observed that the sintering temperature and amount of pore former affect the permeability extremely by varying the texture of porous ceramics such as the open porosity, pore size distribution, and tortuosity of pore channels. Darcian and Non-Darcian permeability was found to increase with increase in amount of pore former during the fabrication of porous ceramics. At room temperature SBSC, MBSC and CBSC ceramics with porosity ~56% showed pressure drop of 12500, 5600 and 3000 Pa respectively at 0.05 m s<sup>-1</sup> face velocity [84,90-91].

#### 3.7 Particle Filtration Behaviour Studies

The applicability of ceramic filter as hot gas filter depends on the filtration performance. permeability, durability, etc. in actual hot gas filtration condition [101-110]. In most of the previous studies dust filtration were carried out with commercial SiC ceramic candle filters in advanced power generation process [16-21]. Filtration performance in terms of collection efficiency and pressure drop of coupon size samples are also evaluated using the laboratory test set up to filter nano aerosol particles [105-110]. Very few studies are reported in literature on evaluation of filtration performance of the ceramic filter using laboratory made test set up to filter coal fly ash particles in actual power generation conditions [101-104]. The particle collection efficiency of a ceramic filter was determined by measuring the particle number concentration with an aerodynamic particle sizer. The particle are captured by the porous ceramics expresses their particle filtration efficiency. The overall efficiency was determined from the total particle mass concentration at the inlet and the outlet:

$$E_{overall} = \frac{C_{inlet} - C_{outlet}}{C_{inlet}}$$
(15)

Choi et al. measured the particle filtration efficiency of ceramic filters fabricated with SiC powders of various sizes (10-200 µm) [106]. Fly ash powder was used as test particles and filtration velocity was 1 m/min. The samples prepared with SiC 25 and 50 µm powders exhibited excellent collection efficiencies for particles larger than 1 µm. But the samples made with SiC10, SiC100 and SiC200 powders had low collection efficiencies due to the formation of pinholes and cracks and their inhomogeneity. The E<sub>overall</sub> of the samples of MBSC ceramics with porosity ~ 46.0% and 56%, was found to be >99% [90] and the CBSC with porosity ~ 62% 99% exhibited the Eoverall where NaCl nanoparticles were used as test particles [91]. Woo et al. fabricated SiC ceramic candle (500 mm long, 60 mm OD and 40 mm ID) from SiC powder of d<sub>50</sub> =180 µm using clay and other additives and filtration layer was developed by SiC powder of  $d_{50}$  =14µm followed by sintering at 1400°C. The particle filtration efficiency and pressure drop of the candle filter was evaluated using laboratory made test set up. They used fly ash of  $d_{50}$  = 5 µm as test particles and kept face velocity 5-9 cm s<sup>-1</sup>. The pressure drop was found to increase with decrease of porosity, increase of face velocity and dust loading. They obtained variation of pressure drop between 6000-700 Pa at face velocity 0.07m/s. and ~100% filtration efficiency [101,102].

# 3.8 Thermal Shock and Oxidation Resistance Properties

Using the water quenching technique, the thermal shock behavior of the porous OBSC ceramics was evaluated as a function of quenching temperature, quenching cycles and specimen thickness. Thermal shock resistant of ceramic materials depend on flexural strength, elastic modulus, thermal expansion co-efficient, Poisson's ratio, shape of the samples, thermal conductivity, diffusivity, or emissivity, etc. [163-164]. The unified theory of fracture initiation and crack propagation [164] suggests that the

Material Characteristics		Permeability parameters					
Bond type	Porosity, P (%)	Flexural Strength (MPa)	Darcian permeability constant (k <sub>1</sub> , m <sup>2</sup> )	Non-Darcian permeability constant (k, m)	Pressure drop (Pa), (porosity)	Particle filtration efficiency (%)	[Ref.
Cordierite	39 – 53	11.1 -30	0.71-0.88 x 10 <sup>-13</sup>	0.02 -0.02x 10 <sup>-6</sup>	-	-	63
Mullite	25-62	3-24(at P 37-43.4 %)	0-10 x 10 <sup>-13</sup>	0-3.5 x 10 <sup>-6</sup>	-	-	57
Silica	35.6 –56.4	5.4-29.5	1.64 -18.4 x 10 <sup>-13</sup>	3.19-52.3 x 10 <sup>-8</sup>	27,000(46%)and 5000 (56%)	-	84
Mullite	29-55	9-34	$1.21 \times 10^{-13}$ to $1.60 \times 10^{-12}$	5.8-29.5 × 10 <sup>-8</sup>	15,000 at P 46% and 5700 at P 56%	99.9 (at P 46-56%)	90
Cordierite	30-55	14-54	0.8 -27 × 10 <sup>-13</sup>	5.8-29.5× 10 <sup>-8</sup>	4200 at P 46% and 3700 at P 56%	96.7-99.9 (at P 68- 62%)	91
Oxide	21.2-40.4	-	-	-	490-88259	99% (particle size > 1 µm	79
oxide	28.7-35.8	20	-	-	200-3000(without dust) 900-7500(with dust loading	99.9	101
glass	32-64.7		1.45-6.53× 10 <sup>-12</sup>		5		81
ŠiC	68-75		0.13-0.82× 10 <sup>-12</sup>	-	-	-	19
LPS-SiC	45-57	15-27	0.4-1.1× 10 <sup>-12</sup>	-	-	-	20
LPS-SiC	86	16.6 (compressive str.)	2.3-10 × 10 <sup>-11</sup>				21

#### Table 3. Permeability parameters of oxide bonded porous SiC ceramics

damage by thermal shock depends on the degree of resistant to nucleating fracture. Li et al. suggested that the strength of SiC based ceramic composite decreases due to intrinsic brittle nature and sensitivity to crack after thermal shock [165]. Thermal shock analysis of oxide bonded porous SiC ceramics [47,52,54] showed a gradual decrease in flexural strength with increase in temperature gradient and thickness of the sample due to formation of cracks. This was interpreted that when the quenching temperature was >  $200^{\circ}$ C, the tensile thermal stress caused crack formation in weaker necks which led to decrease in the load-bearing area and consequently in the fracture strength. With further increase in the quenching temperature and thus in the thermal stress, more and more necks were cracked. As a result, the measured strength decreased gradually with quenching temperature [52,54]. However, cracks were arrested at neck region during thermal shock analysis of SiC ceramics because of porous nature of materials which did not allow the damages been propagated properly and the ceramics reported to have excellent thermal resistance properties [52]. Moreover it was found that the fracture strength of the quenched specimens was not affected by the increase of the quenching cycles. Thermal shock resistance test of SiC composite also showed decrease of strength with guenching temperature up to certain temperature but with further increase of quenching temperature, a slow gain in strength was noticed which was explained by healing of some cracks [166] by oxidation derived silica glass. OBSC ceramics are having excellent oxidation resistance properties, due to the presence of SiO<sub>2</sub> layer on the surface of every SiC particles. She et al. observed only a weight increase ~ 0.5 %, on oxidizing OBSC ceramics in air at 1000°C for 50 h [49]. The thermal shock resistance in combination with excellent oxidation resistance properties suggests a potential advantage of porous SiC ceramics for several industrial applications.

#### **3.9 Other Application Areas**

Porous silicon carbide (SiC) ceramic filters are commercially used for the combustion gas cleaning including IGCC and PFBC systems to clean combustion gas. SiC filters are also found suitable for High-Efficiency Particulate Air (HEPA) and Ultra-Low Penetration Air (ULPA) filters to provide extremely high collection efficiency of submicron airborne particles that are present in radioactive, biohazardous and highly

toxic aerosols [167]. Porous SiC ceramic is becoming a potential microwave absorbing material because it has not only excellent microwave-absorbing property but also high mechanical strength and interconnected holes connected to the external. Additionally, porous ceramics have high thermal resistance and low volumetric heat capacity due to high porosity, low density and low thermal conductivity coefficient. Very recently porous SiC ceramics are used as super thermal insulation materials, energy conversion treatment and environmental technologies, for lithium polysulfide retention in Li-S batteries, for simultaneous removal of PM and volatile organic compounds, etc [168-170]. There are large numbers of areas where oxide bonded porous SiC ceramics could be used. Therefore constant efforts are going on the development of porous SiC ceramics for various applications industrial [171-172]. New mechanisms are required to balance properties of porous SiC ceramics processed by oxide bonding method for various industrial applications.

#### 4. CONCLUSIONS

Large numbers of articles are reported on the development of porous SiC ceramics and their application, this review mainly focuses on the processing and properties of porous SiC ceramics processed by oxide bonding method. This paper highlighted the synthesis of porous SiC ceramics in air at low sintering temperature 900-1600°C between where different ~ secondary oxide bond phases (Mullite, Yttrium aluminium garnet, Cordierite, glass, frit, silica, etc) bonded the SiC particles. The oxidation bonding technique involves several advantages includes low processing temperature, cost easy control over the porosity, effectiveness, pore sizes and distribution of pores, with superior resistance to oxidation, etc. By selecting the type pore former, and amount of sinterina temperature, size of the starting SiC powder, the porosity, pore size distribution and pore morphology can be controlled in the resultant SiC ceramics. Literature survey indicated that there are many issues on processing of porous SiC ceramics by oxide bonding method which need to be studied in detail that includes (1) the development of more cost-effective processing with homogeneous distribution of bond phase, narrow pore size distribution using low cost raw materials (2) investigate corrosion resistance and thermal shock resistance properties of the resulted porous ceramics (3) investigate other

important properties such as air permeability and particle filtration efficiency of the porous SiC ceramics (4) optimization of the processing parameters based on the desired required properties for specific application (5) further development of permeable filtration layer on the porous SiC support for further air filtration applications (6) development of further coating layer on the porous ceramic by some additives which can acts as the catalyst and adsorb some effluent gaseous. Therefore processing oxide bonded porous SiC ceramics with homogeneous formation of oxide bond phase along with full knowledge of air permeation, particle filtration, thermal and chemical shock resistance behaviour in real condition is essential. The processing of porous ceramics by oxidation bonding technique has been continuously improving to the requirement meet of microstructure. characteristics pore and properties as per the need of application areas. Further work should also be directed to explore new possible application areas of oxide bonded porous SiC ceramics.

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#### **COMPETING INTERESTS**

Authors have declared that no competing interests exist.

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