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Preparation Silicon Carbide Pours and Studies their Characterization

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Abstract: In this research preparation pours silicon carbide by addition saw dust in different percentage (1, 3, 5, 7) wt.% to the SiC matrix by using cold isoststic press technique, then the sintering temperature was 1250°C, polyester polymer and glass powder was added to the composites as binder and to assist lowering the sintering temperature. Then make the tests of measurement the density, porosity, vickers hardness, thermal conductivity and scanning electron microscopy. The apparent density increase with the saw dust at 5 and 7 wt.% it showed the retention of small pore sizes and fine particles. The porosity was in range of 33-48% it increased with the saw dust ratio, sintering temperature within 1250°C and with present polyester as binder addition. The hardness decreases with increase porosity in the samples. Thermal conductivity decrease with increase the saw dust ratio. SEM result show that, the microstructure were seen to be improving to the pours silicon carbide grains surrounded by a glassy-rich phase.

Key words: Porous silicon carbide, saw dust, porosity, thermal conductivity, retention, Iraq

INTRODUCTION

Porous Silicon Carbide (SiC) that has grand potential for overcoming of industrial applications including filtration for gas and water, absorption, grinding materials, thermoelectric conversion, etc. (Liden *et al*., 1995; Chen *et al*., 2012) due to their high mechanical strength, low thermal expansion coefficient and good thermal shock resistance.

Bonding techniques in general inexpensive, low-temperature processing route to prepare macro porous SiC ceramics with porosity from 15-60%. The sintering temperature was in arranging 850-1550°C, dependent on the bonding composition. Mullite ($3Al_2O_3.2SiO_2$), Silica (SiO_2), Silicon Carbide (SiC), Silicon Nitride (Si_3N_4), cordierite ($2MgO.2Al_2O_3.5SiO_2$), Silicon (Si) and frit phases have been investigated as bonding materials for porous SiC ceramics (Soy *et al*., 2011).

A decreased temperature of sintering means that this processing method will be modified towards the lower temperature applications of silicon carbide. So, synthesized micro porous SiC ceramics of low temperature can be used for filtering aggressive liquids and gases (Schwetz, 2000; Streitwieser *et al*., 2006).

The benefits of effective low temperature sintering of silicon carbide are directly obvious as a lower processing temperature, even if only a small reduction, relates to a huge cost saving when considered on the mass-scale of manufacture (Wang *et al*., 2005). Current examples of low temperature SiC uses are varied and include ball bearings; scratchy, thermal isolation in furnaces and cutting tools (Wang *et al*., 2005; Castillo-Rodriguez *et al*., 2006). Several searches were about synthetic porous silicon carbide by various methods. Fukushima *et al*. (2009) prepared porous SiC which are sintered at 1500-1800°C by using different additive and weight ratios of Al_2O_3/Y_2O_3, the pore size and grain size increased with increasing sintering temperature. However, the linear shrinkage and porosity did not change during heating.

Dey *et al*. (2011) prepared porous silicon carbide by an infiltration technique with good flexural strengths. Bai *et al*. (2012) preparation silicon carbide porous ceramic used Fe_2O_3 as pore-forming agent. Maity *et al*. (2012) synthesized porous silicon carbide from cellulosic bio-precursor with a mean fracture strength value of 303.9 MPa. Li *et al*. studied the effects of molding pressures, bonding phase contents and silicon carbide particle sizes on the flexural strength of SiC-based porous ceramic (Pastila *et al*., 2001).

To this finish, the present study work is undertaken to develop a new class of silicon carbide/sawdust and studies the effect of various operational variables.

MATERIALS AND METHODS

Experimental parte

Composites material: Silicon Carbide (SiC) used as matrix which supplied by (NICE Ltd., India) as particle size 30 μm. The SiC matrix reinforced by saw dust with different weight percentages (1, 3, 5 and 7 wt.%). Dried saw dust in case to remove residual moisture. Unsaturated polyester resin (manufactured by Ciba Geigy-Northern Polymers Ltd., India), glass powders obtain from (Saint Govion) with particle size ≥30 μm used as binder.

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Composite fabrication: In this study, silicon carbide matrix and 2% glass powders mix together. Then these mixture reinforced by saw dust with different percentages (1, 3, 5 and 7 wt.%).

Each mixture was made thoroughly with 5% Unsaturated Polyester (UP) binder solution then pressing cylinders of 25 mm diameter and 3 mm thickness by uniaxial pressing using stainless steel dies at pressure 13 tons.

The samples were dried by electrical dryer at 110°C for 6 h. Then fired at 1250°C with a soaking time for 2 h. The tests carried out which are Apparent Density and Porosity, Water Absorption Ratio, Hardness (HV), Thermal Conductivity and Scanning Electron Microscopy (SEM).

Testing techniques

Density and porosity testing: Density and porosity are measured by Archimedes method using a Sartorius Density Determination Kit. The 1 h was allowed for the sample to be fully saturated in the distilled water. The expression for Archimedes density:

$$\rho_A \approx \frac{m_{air}}{m_{sat}-m_{sub}}$$

Where:

m_{air} = Mass in air
m_{sat} = Mass when saturated in water
m_{sub} = Mass when saturated in air

A density of water of 1 g/cm^3 at room temperature. The geometric density (ρG) was determined by taking the sample's dimensions and calculated as follows (Uhlmann *et al*., 1976):

$$\rho G = m/V$$

The Apparent Porosity (AP): The apparent porosity was calculated using the following equation:

$$AP(\%) = \frac{W_s-W_d}{W_s-W_n}\times 100$$

Where:

W_d: = Weight of the dry sample
W_s: = Weight of sample being infiltrated with water
W_n: = Weight of sample being immersed in water (Uhlmann *et al*., 1976)

Vickers Hardness (HV): Vickers Hardness (VH) measurements were performed a diamond indenter in the form of a right pyramid with a square base and an angle 136°C between opposite faces is forced into sample in a load F. The two diagonals of indentation on the surface of the sample after removal of the load is measured and its half the indentation diagonal L. Hardness in porous materials is usually described using an equation:

$$H_v = 1.854\times F/L^2$$

And 1.854 is a geometrical Constant of the diamond pyramid.

Thermal conductivity (K): Thermal conductivity is one of the more important properties for the ceramic insulation and refractory which is refers to the quantity of heat transfer through the body.

In this research Lee's disk method was used for determining the thermal conductivity (K) (Philip and Fagbenle, 2014). This investigation was carried out using 40×20 mm cylindrical specimens according to the standard specifications of the instruments using Lee's disk type (Griffin and George Ltd., Germany).

Scanning Electron Microscopy (SEM): Scanning Electron Microscopy (SEM) is a more advanced form of microscopy which is prepared with a field emission gun operating between 200 V to 30 kV used to analysis a material's chemical composition, morphology and phases. The samples were put on a carbon tape and mounted on the stage of the microscope. Secondary electrons were used to take images at a voltage of 16.0 kV and a magnification range of continuous form 6× to 100.000 ×s at various spots.

RESULTS AND DISCUSSION

Density and porosity: As show in Fig. 1 which is represent the value of the densities and porosity with additive percentage for samples. Figure 1 shows the results that the theoretical density increase with increase the addition of sawdust, the determination of the theoretical density of the ceramic cannot be verified as a highly accurate estimate. This is because the value for the theoretical density was calculated using the density of the initial components however upon the present of the glass, its density will not simply be a manipulation of these primary values (Can *et al*., 2006).

Figure 2 shows that apparent density of the composite decreases with addition of sawdust. The decreasing density of sintered samples beyond certain percentage is a well-known phenomenon related to the grain coarsening and pore coalescence, so can say the decrease in densification can be assign to the trapping of

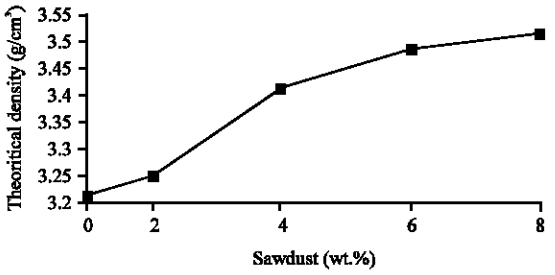

Fig. 1: Theoretical density of porous SiC with sawdust ratio

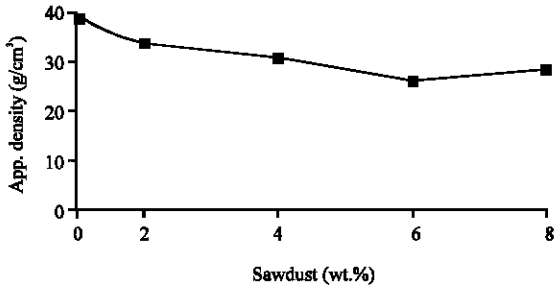

Fig. 2: Apparent density of porous SiC with sawdust ratio

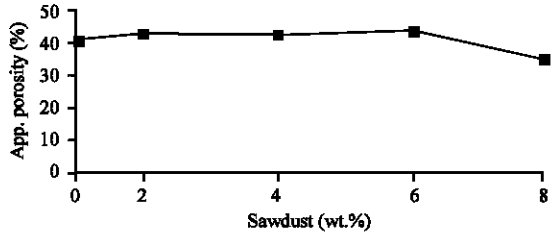

Fig. 3: Apparent porosity of porous SiC with sawdust ratio

oxygen vacancies by the creation of defect associates which lead to a decrease in concentricity of free oxygen vacancies. Also, this behavior can be explained by the different specific surface areas of the powders (Can *et al*., 2006; Liden *et al*., 1995).

By an addition of 5 and 7% sawdust, it showed the maintenance of small pore sizes and particles. This mainly due to the formation of liquid phase from a thin layer that exists on the surface of SiC particles. Also can say the decreasing of density with the increasing saw dust ratio, that the saw dust fired and resulted pores in the ceramic body. Figure 3 shows that, the porosity increases with sawdust ratio is increasing because the inter vacancies are caused by saw dust that leaves the specimen during firing.

The obtained porosity was in range of 33-48% and it increased with the sintering temperature within 1250°C in

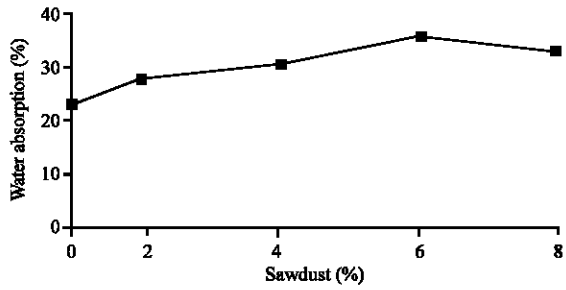

Fig. 4: Water absorption porous SiC with sawdust ratio

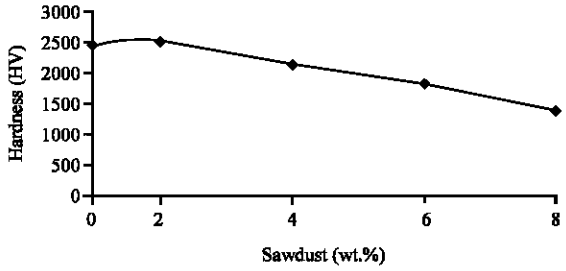

Fig. 5: Vickers hardness of porous SiC with sawdust ratio

the present polyester in the initial mixture and glass phase resulted at sintering process of porous SiC which was filled few pores inside the ceramic body due to the covalent Si-C bonds, porous SiC ceramics needed to sintering at high temperatures or (and) with the addition of sintering additives which have restricted the application of porous SiC.

Figure 4 shows the increasing of the water absorption ratio with increasing the saw dust ratio. New processing track that conquer the problems are the ceramic polymer processes, during which the polymer precursors change into ceramic materials. This process has an benefit of requiring usually low temperature of 1000-1200°C.

Hardness: When compared with the Vickers hardness of pure α-SiC (24 GPa), generally, the hardness of the samples is expectedly less. The presence of the weaker additive phase lowers matrix hardness as shown in Fig. 5.

The hardness of the samples is seen to be of a medium level in reference to other structural ceramics and is of a lower magnitude than was targeted. From a micro structural viewpoint, this follows as the more porous a material, the less resistant it be comes to plastic deformation. The relationship between hardness and porosity mean the

additive inverse of density (Tekkaya and Lange, 2000). The results of the measurements depend on the porosity of the matrix structure in the vicinity of the point of indentation. It can be say that the hardness increases with geometrical density and decreases with porosity, the lower value was taken due to an assumed SiC-glass-SiC layered structure whereby the softer glass layers compress between the SiC layers (Cho *et al*., 2009).

Thermal conductivity: Thermal conductivity decreases with sawdust additions as show in Fig. 6, the thermal conductivities, mainly influenced by porosities. Thermal conductivity through a solid is controlled largely by phonon transfer this relies on the extension of thermal vibrations through the crystal lattice a cross a temperature gradient and is reduced when large numbers of point defects and extended defects (e.g., dislocations) are present (Philip and Fagbenle, 2014).

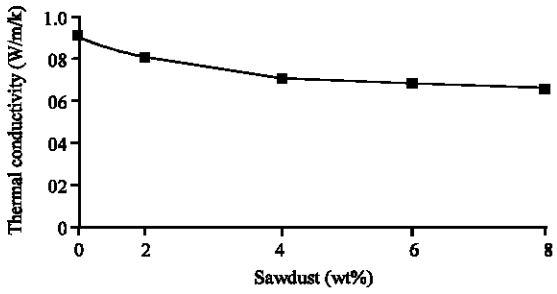

Fig. 6: Thermal conductivity of porous SiC with sawdust ratio

Thermal conductivities also decrease from 0.8-0.65 W/m/K with the porosity ratio increasing from 33-48 %, this result is rational, higher ratio of air in samples meaning higher porosity and air have low thermal conductivity (Jang and Sakka, 2008).

Consider the effect of temperature on a sample with constant porosity with indication to the theory of heat conduction naturally second, consider the porosity of a material which are Pores act as main disruptions to the crystal lattice (i.e. are large defects) and thus have a strong influence on thermal conductivity (Philip and Fagbenle, 2014).

Scanning Electron Microscopy (SEM): Figure 7 shows the SEM image of silicon carbide samples sintered at 1250°C for 1 h. The sintering process facilitates melting of glass as (glue) to bind SiC particles together.

Figure 7a SiC particles are well-bonded to each other by glassy phase. The morphology of the open pores created between silicon carbide particles is unequal, whereas spherical closed pores are shown the bonding

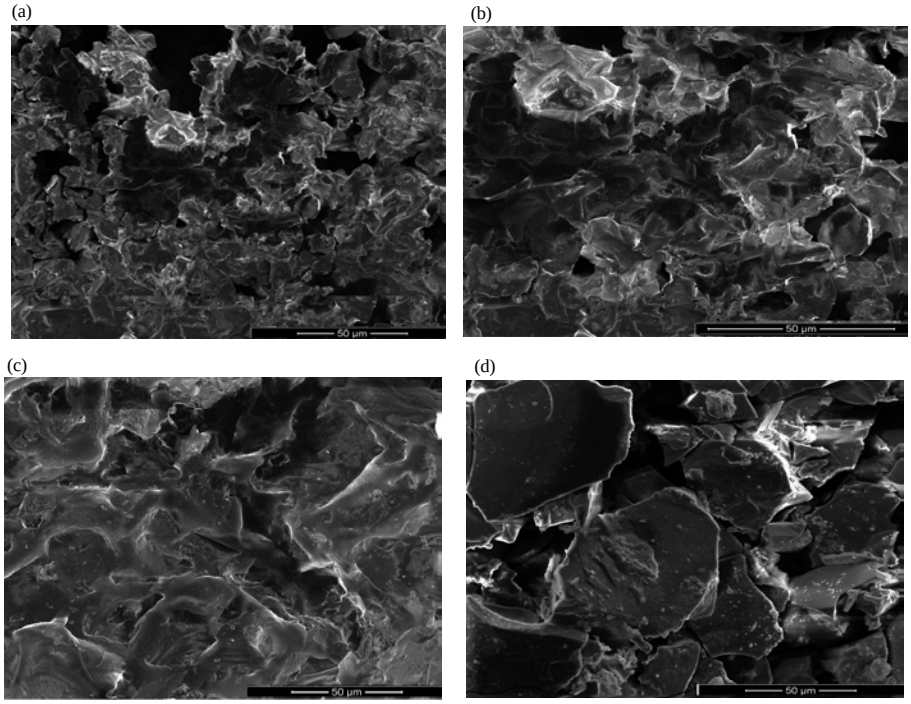

Fig. 7: The SEM image of porous silicon carbide samples sintered at 1250°C for 1 h: a) 1 wt.% sawdust; b) 3 wt.% sawdust; c) 5 wt.% sawdust and d) 7 wt.% sawdust

glass phase. The samples with (1% glass powder) reveal the lowest porosity, that refer to the large size ratio between silicon carbide particles and glassy phase, resulting in dense green sample as well, the densification of silicon carbide particles need much higher amount of glass phase due to its larger surface area.

Figure 7b shows open porosity of samples with (3 wt.%) contents of sawdust and particle sizes of glass powders that sintered at 1250°C for 1 h. With the additive of sawdust, the porosity of silicon carbide samples increase. Also can be say that the saw dust leaves in their sites vacancies which is represent as porosity as show in the image.

Figure 7c microstructure was of particular interest as it presented a grain structure with a brighter phase consistently present inter-granularly, the darker phase can be seen to have a primarily spherical shape and an approximate sub-micron grain size. The small pores shaped through the glass melt process (Goodhew *et al*., 2000).

For samples with the (5 wt.%) sawdust which has the open porosity increases with the saw dust percentage and particle size of silicon carbide, it was due to the lower density of green sample with particle size under the equal pressure. Figure 7d shows the usual pore size distribution of the porous silicon carbide samples. It takes on a fine and unimodal pore distribution.

CONCLUSION

Decreasing density of sintered samples beyond certain percentage is a well-known phenomenon related to the grain coarsening and pore coalescence and due to the presence of liquid phase from a thin layer that exists on the surface of SiC particles.

The sawdust fired resulted pores in the ceramic body, so can say the decreasing of density with the increasing saw dust ratio and that lead to increasing in the porosity of the samples as that result showed.

Measurements of hardness depend on the porosity of the matrix structure in the vicinity of the point of indentation, so, the hardness of the samples is expectedly less due to the increase the porosity. Thermal conductivities decrease from 0.8-0.65 W/m/K with the porosity ratio increasing from 33-48 %.

The morphology of the pores formed between silicon carbide particles is unequal whereas closed pores are shown on bonding glass phase, brighter phase consistently present inter-granularly and the darker phase can be seen to have a primarily spherical shape and an approximate sub-micron grain size.

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