Structural Properties of Porous Silicon Carbide Bonded with Bentonite, Prepared by a Gas-Emitting Chemical Reaction Method

Lara A Shalal and Shihab A Zaidan

University of Technology-Applied Science Dep.

E-mail: as.20.77@grad.uotechnology.edu.iq

Abstract. Direct foaming and bonding techniques were used to fabricate porous silicon carbide ceramics. Silicon carbide is characterized by a high melting temperature; it is always referred to as being bonded with low melting point materials such as bentonite. The technique of generating gases from the reaction of hydrochloric acid HCl with aluminum was used in silicon carbide-bentonite suspension, and by means of which high porosity bodies were prepared by adding different bentonite (10,15,20 and 25 wt.%). After molding them, it is dried and fired at 1100°C. The results of the X-ray diffraction analysis was founded that the main phases appearing after the firing process are α and β–SiC with quartz and mullite, where a part of (β–SiC) is transformed to (α-SiC) and transformation a part of Quartz to Tridymite, which means an increase in the stability of the crystalline phases. The porosity of specimens was decreased with bentonite increases after the firing process, and the values ranged between 62.3% and 72.3%. The compressive strength was increased with bentonite additions increases in the range from 4.35 MPa to 5.08 MPa. From the above results, it is clear that this type of porous (SiC-bentonite) composites can be used in fluid filtration with high efficiency, especially at high temperatures.

Keyword. Porous Ceramics, Silicon Carbide, Bentonite, Direct foaming.

1. Introduction

Porous ceramics have many suitable properties, which combine the characteristics of porous ceramic materials, such as low density, lightweight, low thermal conductivity, low dielectric constant, thermal stability, high specific surface area, high wear resistance, high permeability, and high resistance to chemical attack. Porous ceramics are commonly used in molten metals, industrial hot gas filters, and diesel exhaust filters [1]. The pore size and porosity percentage are controlled by the particle size distribution of the starting ceramic powder, manufacturing technology, binder type, and sintering conditions. The decrease in porosity will decrease with the increase of manufacturing conditions (such as pressure, sintering temperature, and time) and will also affect manufacturing effects, such as the quantity and type of additives, green density, and sintering conditions (temperature, pressure atmosphere, etc.) [2]. Direct foaming usually generates bubbles inside the ceramic slurry containing SiC powder, thereby producing SiC foam. In this technology, a ceramic or precursor suspension is foamed with a foaming agent to produce stable foam, which is then dried and sintered to obtain a large-pored SiC ceramic. The bubbling agent can be a volatile liquid, a solid form, which is a gas escapes, or a gas that can be generated in situ by a chemical reaction, or it can be added to the liquid...
mixture by mechanical stirring or bubbling (gas injection). The nucleation and growth of bubbles in the precursor liquid or ceramic slurry are affected by the presence of suspended particles and the viscosity of the slurry. Blowing agents are divided into physical and chemical blowing agents. Chemical foaming agents produce gaseous products through chemical reactions, while chemical reactions do not accompany physical foaming agents, and the bubble/foam generation process is reversible [3]. In the past few decades, several methods have been developed to prepare porous SiC ceramics using direct foaming. In the presence of surfactants, porous SiC ceramics can be produced by combining the thermosetting properties of the matrix polymer with in-situ foaming agents [4], or apply pressure drop technology [5], or forming by steam box [6]. The intensity of the reaction can be determined and controlled by determining the concentration of the acid used. The temperature during the process, the surface area of the particles involved, and the catalyst's design can determine the degree of change in hydrogen production in cubic centimeters over time. When aluminum reacts with dilute hydrochloric acid, the chemical reaction can be estimated, as shown in the following chemical equation [7].

$$\text{Al} + \text{HCl} \rightarrow \text{AlCl}_3 + \text{H}_2$$

The reaction rate between Al and dilute acid will determine the production of hydrogen. The chemical activity will become active after (3s), until (10 minutes) continue to remain active, as the time approaches 14 minutes, its activity begins to decline, and after (14 minutes), the activity becomes linear (zero) Mole H2). During the 14-minute reaction, the weight of Al will decrease from (110 g) to (90 g). This method is a simple and inexpensive alternative, depending on the strength of the acid used. The reaction process is a compilation of dilute hydrochloric acid and aluminum metal's corrosion and corrosion properties to produce chemically active hydrogen [8].

2. Materials and methods
The primary material used to prepare the porous body is the commercially available α-SiC powder (99.5% impurity) produced by Struers Company, with a particle size of 10 µm < particle size < 38 µm. Iraqi bentonite raw material has been used as a binding material. These raw materials are provided by the "National Geological Survey and Mining Company." Put the bentonite into a ball mill for 5 hours to obtain a uniform powder and make the ceramic porous. The chemical composition of the oxides is listed in Table 1 by XRF or chemical wet analysis.

| Oxide | SiO₂ | Al₂O₃ | Fe₂O₃ | TiO₂ | CaO | MgO | Na₂O | K₂O | Loss on ignition %
|-------|------|------|------|------|-----|-----|------|-----|-----------------
| Bentonite | 55.31 | 14.62 | 5.71 | 1.17 | 3.43 | 3.31 | 1.11 | 0.72 | 14.62 |

2.1. Traifawi, Western Desert, 35 km to the South of H3-Iraq
The reactant that chemically generates hydrogen is a pure aluminum fine powder, provided by Merck (≥90%) (63µm) in Darmstadt, Germany, and the concentration of hydrochloric acid is (35-38%), provided by Thomas Baker. Silicon carbide is mixed with bentonite (10, 15, 20, and 25 wt. %) and Al powder, and then water and dilute HCl acid are added to the mixture. The Al powder reacts with dilute HCl acid to generate hydrogen to carry out a chemical reaction as bulk Space hole, as shown. In Figure (1) As the temperature increases, the reaction between many possible continuous and concurrent reactions will accelerate [10]. After the reaction is completed, it is put in a drying oven with a temperature of (250°C) for a specific period according to the amount of material and then roasted at (1100°C).
According to ASTM (C373), the traditional Archimedes method is used to measure the sample's apparent porosity. A cubic sample is used to determine the compressive strength test, according to ASTM (C-773). Calculate the apparent porosity and compressive strength of the fired sample using the following equations:

\[
A.P\% = \frac{w_{s-w_d}}{w_{s-w_i}} \times 100
\]  

Where:
- A.P: Apparent porosity %,
- \( w_{s-w_d} \): The mass of the dried sample (g),
- \( w_s \): Mass of sample immersed in water (g), and
- \( w_i \): Mass of sample immersed in water (g).

\[
\sigma = \frac{F}{A}
\]

Where:
- \( \sigma \): Compressive strength (MPa),
- \( F \): Maximum applied load (N),
- \( A \): The cross-sectional area of the sample (mm\(^2\)).

Use (Shimadzu XRD-6000 system, copper, and NF type radiation source) to perform X-ray powder diffraction (XRD) research on all prepared solid samples.

3. Results and discussion

Porosity is one of the essential parameters that can characterize ceramic particles in general and porosity in particular. Figure (2) shows the decrease in the porosity from (72.3%) when adding (10%) bentonite to (62.3%) when adding (25%) bentonite. The reason for this decrease is due to the low melting temperature of the bentonite, which works to bind the SiC grains together through the formation of the liquid crystal phase [11]. An increase in bentonite means an increase in the liquid glass phase, which works in two directions.

First: penetration between the granular boundaries and inside the pores.

Second: the binding of silicon carbide particles. The type of pores, their shape, and distribution are among the important factors that affect the determination of the nature of porous bodies in different applications. The compressive strength is one of the most critical mechanical properties determining the extent of bonding between the ceramic body particles. Porosity is the most crucial factor in decreasing mechanical properties in general and compressive strength in particular. As for the effect of adding bentonite on the prepared ceramic objects, it is shown in Figure (2), where it was noticed that the increase in the compressive strength value from (4.35 to 5.08 MPa) for the percentage of addition from (10 wt. % to 25 wt. %) when firing at a temperature of (1100 °C) because this temperature is insufficient to achieve the entire liquid glass phase. The open pores visible on the specimens' surface are shown in Figure (3), from which the decrease in the porosity of the specimens fired at 1100 °C can be evaluated. Also, pores size was reduced with bentonite increases.
Figure 2. Apparent porosity and compressive strength as a function of bentonite weight percentage additives.

Figure 3. Surface topography of the prepared specimens after firing at 1100°C: (a) SiC-10 wt. % bentonite, (b) SiC-15 wt. % bentonite, (c) SiC-20 wt. % bentonite, (d) SiC-25wt. % bentonite.

The X-ray diffraction results of the materials used in preparing porous ceramics were checked and reconfigured with standard charts using the MATCH-Phase identification from powder diffraction. After mixing the ceramic materials (70S-30B %), added (0.05% Al), and completed the chemical reaction by adding HCl. The x-ray diffraction was examined, and the scheme of the mixture after the preparation process (before firing) is shown in Figure (4), in which all the components of the mixture appear, and from it, if new compounds appear, despite the possibility of (AlCl₃) compounding as a result of the reaction of aluminum with HCl acid. The reason for this is the incomplete crystallization of (AlCl₃) because it needs an increase in temperature. The reaction of aluminum with HCl increases when the surface area of aluminum powder increases, so the particles should form to be laminate than spherical [12].
Increasing the percentage of aluminum to (0.0625wt. %) led to the montmorillonite phase and decreased quartz intensity due to random combinations caused by HCl interaction with clay compounds. Hydronium cations are also inserted between the sodium montmorillonite layers through the cation exchange mechanism while maintaining the layer structure. The decrease in intensity and increase in the peak width of montmorillonite indicates a significant effect of the acid on it and the emergence of amorphous phases due to the dissolution of the crystal structure of montmorillonite [13]. Figure (5) Shows the results of X-ray diffraction when adding (0.0625 wt. %).

Also, an excess of silica remains in the quartz phase, and a portion of the quartz is converted to Tridymite, with very little aluminum being converted to aluminum oxide (gamma-alumina phase γ-Al₂O₃) unstable, so that it can transition into γ-Al₂O₃ when the firing temperature increases. With the Keep of silicon carbide without any changes in the structure, and therefore it can be said that the mixture formed is a composite material.

![Figure 4. X-ray deflection analysis of the (70S-30B %), 0.0125 %Al before firing.](image)

![Figure 5. X-ray deflection analysis of the (90S-10B) % +0.0625 %Al before firing.](image)

Specimens burring processes containing (70S-30B) added to (0.05% Al) when in Fig. (6) Firing at (1100 °C) temperatures led to phase changes, especially in bentonite, and the most prominent phase resulting from firing bentonite is the formation of mullite compound 3Al₂O₃. 2SiO₂. Increasing the proportion of aluminum to (0.0625% Al) led to more prominence in the intensity of mullite formed
with the decrease of quartz and changes in the phases of silicon carbide, which led to change in the intensity of some of them, as shown in the Figure (7). Several factors affect the emergence of this phase of this essential ceramic phase, namely the particle size, fire temperature, firing conditions, and the rate of high-temperature furnaces [14].

4. Conclusions
1. The main phases formed from the burning of bentonite with silicon carbide (α-SiC) are the transformation of quartz to tridymite and crystallite and the emergence of the mullite phase.
2. Adding bentonite to the suspension (SiC) speeds up the chemical reaction because the bentonite is alkaline.
3. The mechanism of the interaction of hydraulic acid (HCl) with aluminum has shown high efficiency in generating hydrogen gas, and there is a possibility to control the remaining bubbles of this gas inside the ceramic suspension to form closed and open pores.
4. Bentonite showed efficiency in bonding silicon carbide granules, thus improving mechanical properties and inhibiting silicon carbide's thermal conductivity.
5. Porosity played a significant role in some of the characteristics, such as mechanical properties.
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