Nanostructural Characters of β-SiC Nanoparticles Prepared from Indonesian Natural Resource using Sonochemical Method

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Abstract. Silicon carbide (SiC) nanoparticles become one of the interesting non-oxide ceramics due to their physical and chemical properties. For an extended period, SiC nanoparticles have been prepared by several methods that usually performed at high temperatures ranging from 1200 - 2000 °C from inexpensive commercial precursors. In this work, we prepared SiC nanoparticles from the low priced precursor of Indonesia natural resource using the sonochemical method at a temperature that is lower than 1000 °C. To produce samples with particular characters, we varied the sintering holding time (1, 10, and 20 hours) and the sintering temperatures (850, 950, and 1050 °C) during the synthesis. The samples were then characterized using XRD, SEM-EDX, TEM, and FTIR. The XRD data analysis showed that the samples have a dominant phase of SiC in the form of \(\beta\)-SiC with a 3C-SiC structure and SiO\textsubscript{2} phase in a low composition within a good agreement with the EDX characterization. Interestingly, the sample prepared at the sintering temperature of 850 °C for 1 hour showed a non-crystallite phase. Using a Scherer’s equation, the particles of the samples sized from 13 to 18 nm, which were validated by SEM and TEM images. Furthermore, the FT-IR spectra presented several peaks, i.e., at wavenumbers of 482.2 and 1150 cm\(^{-1}\) representing Si-O-Si bonding and also at 798.5 cm\(^{-1}\) regarding with Si-C bonding.

1. Introduction

Silicon Carbide (SiC) is an important non-oxide ceramic with unique properties such as mechanical toughness and chemical resistance so that it is utilized for numerous applications [1]. SiC is used intensively in military, aerospace, automotive, nuclear industry, electronic and optoelectronic devices such as solar cell, detector, modulator, and a semiconductor laser, specifically for high frequency, intense radiation, or high temperature [2,3].

Two most familiar SiC crystal structures are \(\beta\)-SiC crystal structure with 3C-SiC phase and \(\alpha\)-SiC crystal structure with 2H-, 4H-, and 6H-SiC phase [4]. The \(\alpha\)-SiC components start to form at approximately 2000 °C or above, and the \(\beta\)-SiC components start to form under 2000 °C. The SiC synthesis could be performed by different methods, for example, a carbothermal method with 1450 °C sintering temperature [5], a sol-gel method using Tetraethyl orthosilicate (TEOS) [6], and solar furnace method with 1500 °C sintering temperature [2].

The temperature and materials required for SiC synthesis are still relatively expensive. Hence, it is necessary to conduct research and synthesis of SiC at low temperature, i.e., under 1000 °C, by using.
affordable local materials. One method that can be a candidate for the SiC synthesis is a sonochemical method [7]. The sonochemical method is an efficient and interesting method to form a mini-emulsion since it could be broken down into a nanometer-sized material of around 20-200 nm by ultrasonic waves [8]. This method is useful for reducing the particle size, controlling morphology, and producing crystal with excellent phase purity and crystallinity.

This research utilizes local materials for the SiC synthesis. The SiC synthesis utilized pyrophyllite that has been synthesized into SiO$_2$ with a purity of up to 98.2 % as the primary material; the produced silica has a nanometer size ranging from 40 until 90 nm [9].

2. **Experimental Method**

2.1 **Synthesis of SiC Nanoparticle**

The SiC synthesis was performed via a sonochemical method utilizing SiO$_2$ and sucrose (C$_{12}$H$_{22}$O$_{11}$). In the synthesis, 3 g C$_{12}$H$_{22}$O$_{11}$, 1 g SiO$_2$, ethanol, and distilled water were mixed for 45 minutes until it became homogeneous at a speed of 350 rpm. The process was continued by a sonication process applied to the solution for 4 hours. Next, the sample was drained at 180 °C for 1 hour. Next, the solution was sintered at 850, 950, and 1050 °C in Ar gas atmosphere for 1, 10, and 20 hours.

2.2 **Characterization**

X-Ray Diffraction (XRD, Philips in X’Pert PRO type), Fourier Transform Infrared Spectroscopy (FT-IR, Shimadzu in Prestige-21 type), Scanning Electron Microscopy (FEI in INSPECT-S50 type), and Transmission Electron Microscopy (FEI in Talos F200X TEM type) characterized the SiC nanoparticle synthesis.

3. **Results and Discussion**

3.1 **Diffraction Pattern and Crystal Structure**

![Figure 1. X-Ray diffraction pattern of synthesized SiC nanoparticles.](image-url)
Figure 1 presents the sample 1 sintered for 1 hour and at 850 °C, sample 2 with a 1-hour sintering time at 950 °C, sample 3 that is sintered for 1 hour at 1050 °C, sample 4 sintered for 10 hours at 850 °C, sample 5 sintered for 10 hours at 950 °C, and sample 6 sintered for 20 hours at 850 °C. The XRD result shows no formation of SiC peaks in sample 1 with a consistent amorphous structure. However, multiphase was found in other samples with SiC and SiO2 peaks forming the diffraction pattern. Sample 5 showed an optimum condition of SiC synthesis via a Sonochemical method while sample 6 showed a decline in peak intensity due to the over prolonged sintering time. Sintering temperature and time affect the intensity of material and tend to reduce the material intensity if its melting point is passed [10]. A sintering process is usually performed in 1 to 1000 minutes [11].

Two phases were formed during the analysis, i.e., Low-cristoballite phase (SiO2) and Moissanite-3C (3C-SiC) with PDF numbers of 01-076-0939 [12] and 01-075-0254 [13], respectively. The sample showed several SiO2 crystal peaks at angle 2θ of approximately 31.27° with (101) hkl field and 44.56° with (212) hkl field, while SiC peaks were displayed at angle 2θ of around 35.96° with (111) hkl field, 42.7° with (020) hkl field, and 56.62° with (202) hkl field.

3. 2 Particle Size

A Scherrer equation measured the average crystal size on the SiC nanoparticle synthesis. Based on the XRD results, the smallest angle of diffraction formed was $\theta = 18.001^\circ$ ($2\theta = 36.003^\circ$) with the $\beta = 0.0096$ (radian). Such calculation presented the average crystal size on a nanometer scale of approximately 13 up to 18 nm.
The SiC crystal size has been inevitably confirmed by SEM, TEM, and EDX characterization presented in Figure 2-4. The Energy Dispersive X-Ray (EDX) test was conducted to analyze the constituent elements of the material. Figure 2 and 3 displays an agglomeration in the sample due to its nano size. It affected the SEM and TEM morphology test results that showed non-uniform sizes of the sample, i.e., approximately 10 nm up to 18 nm. Aside from that, it is already certain that the particle already had a nano size. In other words, the SiC nanoparticle was already formed. Figure 4 is a spectrum of the EDX test performed on the SiC sample showing a smaller Si element concentration than that of the C element of 44.05 % and 50.94%, respectively. Also, the EDX identified another element of O in a concentration of 5.01 so that it the SiC synthesis could be regarded as successful, which is proven by the formation of two phases, namely SiC and SiO₂ phases.

3. 3 FT-IR Characterization Results

Figure 5 displays the FT-IR test results on the SiC sample. All samples showed the existence of a Si-C functional group at the wavenumber of 798.5 cm⁻¹ with different transmissions, and a Si-O-Si functional group was found at the wavenumbers of 482.2 and 1150 cm⁻¹. Such findings are in good agreement with the database of experimental results of the researchers, in which the Si-C functional group formed at a wave number of around 754 until 840 cm⁻¹ [14–17].

4. Conclusions

To conclude, in this research, the β-SiC has been successfully synthesized from SiO₂ and C₁₂H₂₂O₁₁ via a sonochemical method at low temperatures with nanoscale particles of around 10-18 nm. The XRD test results showed a multiphase formation of SiC and SiO₂. According to the FT-IR test results, the sample displayed a formation of a Si-C functional group. Hence, the SiC was well-synthesized by a sonochemical method.
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