



Mechanical Behaviour of Biomorphic Silicon Carbide Derived from Malaysian Timber

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Abstract

The biomorphic silicon carbide (bioSiC) is an alternative to the expensive commercial silicon carbide by using wood waste from the furniture industry which is identified as an attractive option for carbon precursor due to its availability and its anisotropic nature. This ceramic material is fabricated by pyrolysis of wood waste from Kapur, Dark Red Meranti and Kempas in an inert atmosphere leaving an amorphous carbon preform. The amorphous carbon is infiltrated with molten Si at elevated temperatures forming a porous bioSiC with a microstructure that is analogous to the wood precursor. Scanning electron microscope (SEM) analysis showed that the silicon infiltrated both larger and smaller vessels by retaining the original wood structure without being affected by the closure of the small pore and reduction of pore diameter. The X-ray diffraction (XRD) results revealed that SiC is produced without excess silicon after etching. The increasing holding time of infiltration enhanced the formation of bioSiC by up to 3 hours. However, a longer reaction of 5 hours reduced the bioSiC formation at an infiltration temperature of 1500 °C. The flexural strength, compressive and modulus of Dark Red Meranti samples were much higher than those of Kapur and Kempas due to the higher forms of silicon carbide.

Discipline: Material Science, Mechanical Engineering

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1 Introduction

Wood ceramics are new porous carbon materials obtained by carbonising wood and impregnating it with ceramic slurry. Biomorphic SiC (bioSiC) is one of the wood ceramic types fabricated by pyrolysis and infiltration of silicon of wood preform. Natural wood possesses a porous complex microstructure that cannot be obtained by artificial materials; therefore, the characteristic of final bioSiC inherited from the natural wood precursor has been a matter of interest in the last decade. In addition to natural wood, wood-derived cellulosic preform such as sawdust (Bringas-Rodríguez, et al., 2020), paper and cardboard increase significantly as the option for preform precursors. BioSiC with a similar microstructure to natural wood is obtained with high toughness and strength, lightweight, good mechanical strength and large surface area. A current trend in ceramic fabrication is to produce ceramic materials with characteristic properties of multiple and hybridised functions, multi-component, multi structural with low-cost natural materials, as bio-preforms are one of the solutions to improve the material function as they have many inspiring properties such as resistance and adaptability, hybridization and hierarchical organisation. These characteristics will produce materials that can fulfil the physical or chemical demands of any application's requirements. BioSiC fabricated by Si melt infiltration is one of the most popular techniques due to several advantages such as the additives are not necessary, a complex shape can be obtained, low processing temperatures and faster fabrication; thus, it lowers the fabrication cost compared to other fabrication techniques (Orihuela, et al., 2017).

In recent years, the application of silica carbide has been extended to a new era. The lightweight silicon carbide ceramic with fiber has been applied in space, aircraft, energy and transportation industries. So far, in the automobile industry, SiC ceramic has been used as glow plugs in diesel engines, and as a piezoelectric multilayer actuator for high pressure used in rail fuel injection, ball, and roller bearings. Recently, Carbon/Silicon Carbide (C/SiC) has been used as brake disc material in luxury cars and sports cars. In comparison to conventional grey cast iron brake discs, the C/SiC brake disc has reduced by 50% of the weight. Another advantage is it improves brake response and maintains high friction at elevated temperatures. Carbon ceramic brake disc has already been applied in expensive commercial vehicles (Okada, 2009; Jürgen Rödel et al., 2009). Orihuella et al. (2020) studied the effect of bioSiC as a diesel particulate filter and they found that the key factors that affect the permeability results are average pore size, alignment of pores and tortuosity.

The aim of this study is to obtain porous bioSiC ceramics, originating from a wood precursor (Dark Red Meranti, Kapur and Kempas) using the Silicon melt infiltration method. Thermogravimetric (TGA) analysis, X-ray powder diffraction (XRD) as well as scanning electron microscopy (SEM) are performed for the characterization of bioSiC. The physical characteristics and mechanical performance of bioSiC at different holding times are also discussed.

2 Literature Review

The replica method is based on the copy of the original foams concerning its pore shape and strut structure and this method is the most appropriate technique to produce open-cell ceramic with pore sizes ranging from 10 μm to 5 mm. The production of silicon carbide from wood was first carried out by Ota et al. (1995) with alkoxide solutions via sol-gel chemistry. Many researchers have processed SiC from various types of wood as wood derived from porous ceramic has an anisotropic pore structure, resulting in anisotropic properties.

The effect of microstructure on the fracture behaviour of biomorphic ceramic materials at room temperature is being studied by using precursor from pine wood due to the homogeneous tracheidal pore structure with a mean pore diameter of 20 μm (Greil et al., 2002). The processing technique applied is Si vapour infiltration by using three different vapour sources; Si, SiO_2 and CH_3SiCl_3 at the reaction temperature of 1600 $^\circ\text{C}$ for 8 hours of holding time. The authors found that SiC by SiO infiltration had the highest porosity which was 80 %. The fracture behaviour showed similar to that of fibrous monolithic and also laminated composite ceramic. Another characteristic of anisotropy is that the fracture behaviour depends on the load direction in relation to the cell structure. Other alternative processing methods have also been studied (Greil et al., 1988) on various types of wood precursors which included pine, maple, beech, oak and balsa. Infiltration was done at 1600 $^\circ\text{C}$ by liquid infiltration process using TEOS (Tetraethylorthosilicate) for 4 hours holding time. They found that Si infiltrated into the pore channel with a maximum of 30 μm . The larger pore did not contain residual silicon even though the formation of carbon into silicon carbide was already completed.

Zolfran et al. (2004) used pine and beech as carbon precursor prepared SiC by Si melt infiltration at 1550 $^\circ\text{C}$. They found that the microscopic morphology of the biomorphous SiSiC ceramics was similar to materials manufactured by the conventional LSI process. The spontaneous wetting and infiltration of Si on porous carbon preform occurred when a pore was brought into contact with silicon melt and three different phases were formed. They were SiC from the reaction of Si with the biomorphous carbon, solidified Si filling the pores of the initial wood structure and isolated regions of amorphous residual carbon. Two different phases of SiC were also observed, which included coarse SiC-grains with average grain diameters of about 15 μm at the Si/ SiC interface and a nano-grained SiC-phase with average diameters below 100 nm.

Research carried out by Hou et al. (2007) studied the effect of holding time on the properties of bioSiC derived from beech wood. In that study, SiC was prepared by infiltration of liquid silicon into carbon preform at 1550 $^\circ\text{C}$ for 10, 30, 45, 60 and 90 minutes, subsequently removing Si at 1700 $^\circ\text{C}$ for 20, 40 and 60 minutes. According to their research findings, there was a coarse surface on the pore wall due to the formation of crystalline SiC. They observed that increasing reaction time decreases the porosity while enhancing bending strength and fracture toughness. However, when removing Si time was increased, both bending strength and fracture toughness decreased while density and porosity increased. This reduction is attributed to the transformation of excess Si into

gaseous Si, leaving the resultant SiC. They concluded that the improvement in bending strength and fracture toughness is related to the unreacted silicon that improves the strut and increases the density of resulting bioSiC. Their result also indicated that the strength and toughness of the axial samples are significantly higher than those of the radial samples because of anisotropic pore orientation derived from the microstructures of the original wood.

Other alternative processing methods have also been developed to form bioSiC from wood and other cellulose-based preforms. Qian et al. (2004) studied biomorphic SiC prepared by carbothermal reduction of oak wood. Silica sol was prepared from ethanol solutions of tetraethoxysilane [$\text{Si}(\text{OC}_2\text{H}_5)_4$, TEOS], distilled water and hydrochloric acid at a suitable molar ratio of TEOS:H₂O: HCl by a sol-gel process. Carbothermal reduction of the as-prepared silica/charcoal composites was carried out in a static argon atmosphere in a graphite furnace at elevated temperatures for 4-8 hours of holding time. Although this process has fewer processing steps where pyrolysis and infiltration are combined, the SiC yields are far lower than that of molten silicon infiltration. They found that heating at 1600 °C for a longer holding time may occur due to small initial SiC particles (1–5 µm) resulting in the densification of the strut material; thus, it improves mechanical strength without destroying the final structure of SiC. Vladimir et al. (2020) produced porous SiC by carbothermal reduction of TEOS using plane tree fruits as carbon precursors. They found that the carbon obtained through carbonising the PTS precursor and activated at 850 °C during longer holding time (1 and 2 h) in a reducing atmosphere at 1400 °C possesses β-SiC (cubic) nanowires.

3 Method

3.1 Materials

The precursor materials chosen were waste wood (Kapur, Kempas and Dark Red Meranti) collected from a sawmill. Wood samples were cut into rectangles of 15 cm x 4cm x 2.5 cm with a long dimension parallel to the axis of the wood grain. Prior to pyrolysis, the wood samples were dried in an electric oven for 24 h to remove moisture that will affect the pyrolysis process.

3.2 Method and Fabrication

The wood sample was pyrolyzed in a tube furnace with an argon flow. This process involved two heating stages with a slow heating rate to avoid the cracking of samples. The first stage involved heating the sample to 500°C at a heating rate of 1°C/min with 1mL/min of argon flow rate. The temperature was held up for 30 minutes and increased to 850 °C at a heating rate of 2 °C/min and 2 mL/min of gas flow rate.

Carbon preform was placed into an alumina crucible together with excess silicon powder (Aldrich, 99% purity, average diameter of 44 µm). The ratio was higher than the stoichiometric amount as there were silicon losses at high temperatures due to the high vapour pressure of silicon (Bautista, et al, 2011). The heating process was carried out in a tube furnace under argon flow at 1500 C at 1 to 5 hours holding time. The resulting template was composed of Si/SiC. Chemical

etching with a solution of hydro-fluoric (HF) and Nitric Acid (HNO) was used to remove excess silicone. The fabrication of SiC blocks is presented in the schematic diagram in Figure 1.

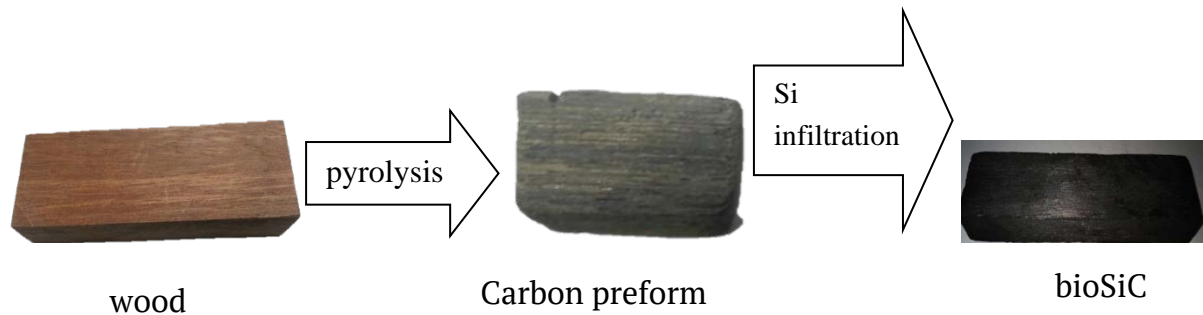


Figure 1: Processing method of producing wood-derived bioSiC.

3.3 Characterization

Thermogravimetric analysis was performed by a thermal analyzer (Model Netzsch TG 209 F3) under flowing nitrogen gas at a flow rate of 100ml min⁻¹ with a heating rate of 10 °C min⁻¹ from room temperature to 1000 °C. Alumina powders were used as a reference sample to determine the mass loss during pyrolysis. Density was measured by the Archimedes method (ASTM C373-88) whereas Scanning Electron Microscope (SEM-Hitachi S-2500) operated at 20kV and 20mA was used for microstructure analysis. The phase composition was determined by X-ray Diffraction (XRD) on a RigakuUltima III) X-ray diffractometer using Cu K α radiation produced at 35kV and 20mA. Three-point flexural strength was performed using ASTM C1161 configuration B with inner 20 mm and 40 mm outer span with a 1 mm/min crosshead speed.

4 Result and Discussion

4.1 Physical Characterizations

Figure 2 depicts the TGA and DTG analysis curves of Kapur, Dark Red Meranti and Kempas. Generally, the mechanism in the conversion of wood into carbon is quite similar to all three kinds of wood where the curves could be divided into three major stages according to the rates of weight loss which fall gradually followed by a sharp decrease and slightly decompose at the last stage. However, some differences may exist due to differences in the cell wall structure of different wood samples (Yan et al, 2011). At the end of the decomposition process, the total residual mass left of Kapur, Dark Red Meranti and Kempas was about 15 %, 17.5 % and 4.5 %, respectively. The differences in the inherent structures and chemical nature of the three components in each type of wood possibly accounted for the difference in total mass residual left. The variation in weight loss is related to the differences in the type of chemical composition (cellulose, hemicelluloses and lignin), especially when more weight loss occurs in the wood that has lower lignin content (Randrianarisoa, 2011). The variation of chemical composition is high between different species and within species as a result of environmental and genetic factors. Factors of age, growth and stress also influence the chemical composition within the trees (Barnet et al., 1988; Greil, 2001).

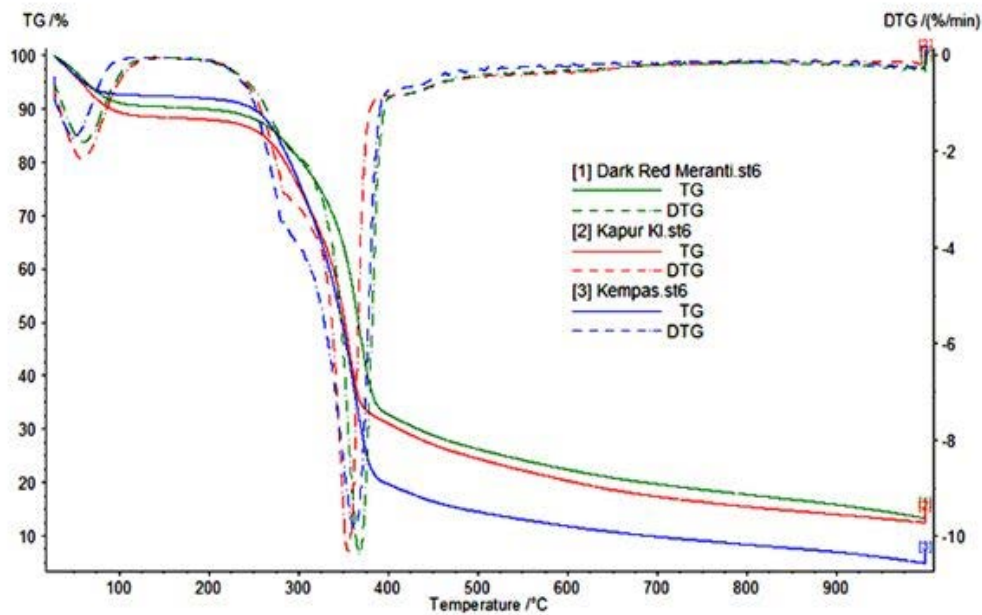


Figure 2: TGA and DTG comparison for Kapur, Dark Red Meranti and Kempas.

Porous silicon carbide is produced from pyrolyzed wood as a precursor as shown in Figure 3. Most of the main elements of the wood tissues such as ray cells, tyloses, fibre, parenchyma from those three types of wood are still present. The wall structure of wood is made up of an amorphous matrix of hemicellulose and lignin with randomly woven fibrils. The woven fibril networks act as thermosetting polymers and as a result, the composite pore wall material of wood burns upon pyrolysis (Greil et al., 1998). During pyrolysis, the microfibril is broken up into carbon and dissociated from its cellulose matrix to form an unstructured carbon matrix, commonly called amorphous carbon.

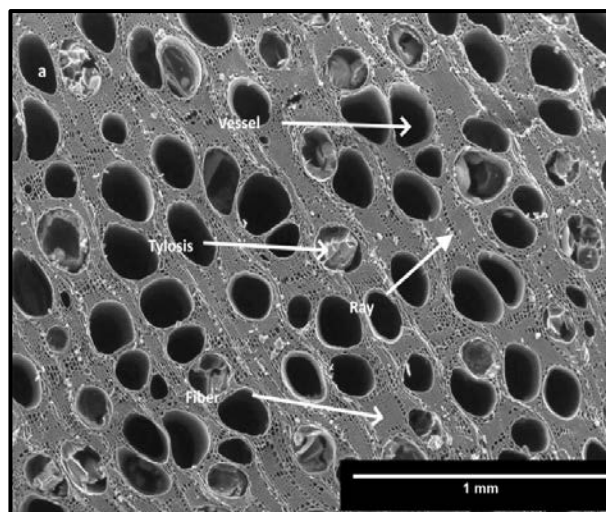


Figure 3: SEM Micrographs of Kapur carbon preform which their retained structure.

The formation of bioSiC involves packing pyrolyzed carbon in a bed of silicon powder and heating it up to 1500 °C. The reaction of carbon with silicon melt occurs as soon as silicon powder is melted into liquid at 1414 °C and infiltrated into the pores. The primary layer of SiC occurs at the

surface of the pore structure; it occurs very fast which was less than 2 seconds (Fukushima, et al, 2008). Infiltration starts with the combination of capillary effect and reactive wetting. The driving forces from both factors lead to the infiltration of silicon into small and large pores followed by a reaction of carbon with liquid Si. The general reaction mechanism of silicon with carbon into bioSiC can be expressed as



where C and Si represent carbon and silicon. All the pores in the samples produced are blocked by the formed SiC layer during the silicon infiltration process, as shown in Figure 4(a). The existence of excess Si in the sample can be removed by the leaching process which is the oxidation of Si by Nitric acid and dissolution of silicon dioxide by Hydrofluoric acid leading to the absence of Si residues. The final result is SiC composed of crystalline SiC and unreacted carbon which can be clearly observed in Figure 4(b).

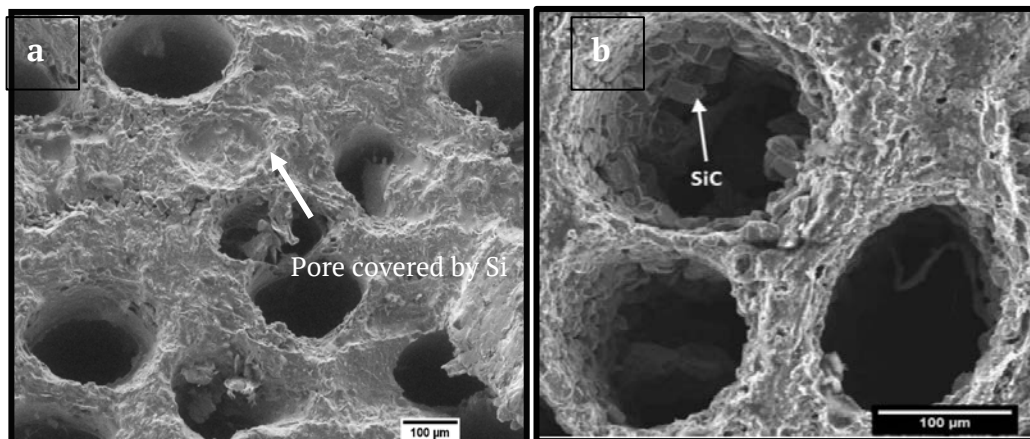


Figure 4: SEM micrograph of (a) Kapur sample before etching; (b) bioSiC derived Kapur at 1500 °C cross-section view.

The phase transformation of biomorphic SiC was investigated using X-ray diffraction analysis. The results were plotted after removing residual Si; therefore, there was no significant peak observed corresponding to Si which indicated that excess Si was successfully removed during etching. Figure 5 shows the X-Ray Diffraction pattern of carbon preform and bioSiC derived Kapur for 1-5 hours holding time. The SiC phase reference of this study is in good agreement with the standard SiC with reference to JCPDS 01-074-2307. XRD profile showed that the amorphous carbon at $2\theta=23^\circ$ disappeared when prolonging the holding time. It was observed that the intensity of SiC at $2\theta = 35.6^\circ$ significantly increased when the holding time was 3 hours to 5 hours which was due to the crystallinity change of the sample from amorphous to highly crystalline. However, the highly crystalline structure of the sample at $2\theta = 35.6^\circ$ can only be seen after 4 hours of holding time. The higher crystallinity of samples is indicative of a greater crystalline ordering and increase in SiC concentration as the crystal structure can be seen clearly in the pore wall as shown in Figure 3(b). Hou et al. (2007) found that the first fastest stage of conversion of carbon into silicon carbide is less

than 10 minutes reaction time which involves spontaneous wetting and infiltration that form the primary layer of SiC on the surface. The carbon atom and Si must permeate through the primary layer of SiC to form the new SiC. Further increase of holding time will use the transformation of unreacted carbon to continue the form of SiC by diffusion process (Zolfrank et al., 2004).

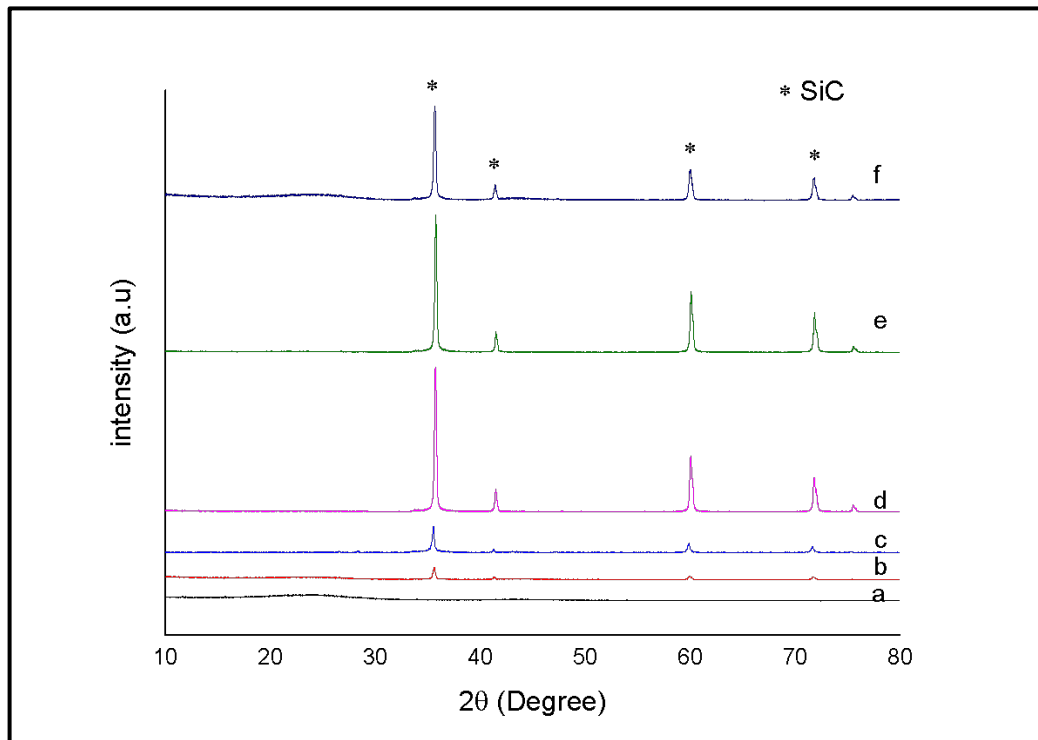


Figure 5: X-Ray diffraction pattern of (a) carbon preform; BioSiC derived Kapur infiltration at 1500 °C for (b) 1 hour, (c) 2 hours, (d) 3 hours, (e) 4 hours and (f) 5 hours.

4.2 Mechanical Characterizations

The flexural strength of SiC-derived Kapur, Dark Red Meranti and Kempas as a function of holding time from 1-5 hours at 1500 °C was determined by flexure tests at room temperature as presented in Figure 6. It can be seen that the flexural strength of bioSiC-derived Kapur and Dark Red Meranti enhanced with the increase of holding time of up to 3 hours and then decreased when the holding time reached 4 hours. The bioSiC-derived Kempas, however, started to decrease at a holding time of 2 hours. It was found that the highest flexural strength was obtained at 3 hours from bioSiC-derived Dark Red Meranti, which was about 53 MPa. This enhancement of 44.1 % was more likely due to the presence of a greater amount of bioSiC phase and a lesser amount of unreacted carbon content. The density obtained at this holding time was 1.9279 g/cm³ with a porosity of 57.47 % since the density was linearly related to the flexural strength while porosity was inversely proportional to the strength. The improvement of flexural strength with the effect of holding time was followed by bioSiC-derived Kapur with about 33.3 MPa which increased by 71.7% at 3 hours. Finally, the lowest value went to bioSiC-derived Kempas with 29.9 MPa at 3 hours holding time.

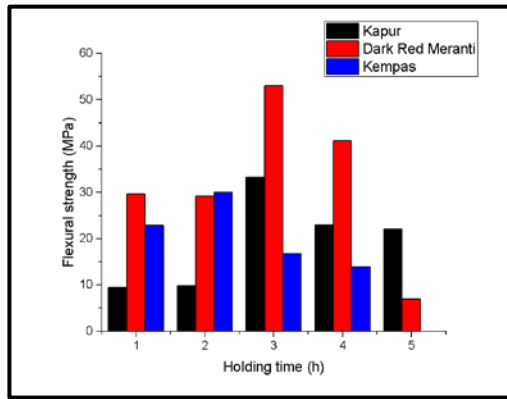


Figure 6: Flexural properties of BioSiC derived Kapur, Kempas and Dark Red Meranti at 1500 °C.

Figure 7 shows the compressive strength of Kapur, Dark Red Meranti and Kempas at 1500 °C for different holding times. The maximum compressive strength for both bioSiC derived from Dark Red Meranti and Kapur occurred at 3 hours holding time with 231.9 MPa and 190.8 MPa, respectively. Further increasing the holding time would decrease the compressive strength by 47.2 % and 11 % for Dark Red Meranti and Kapur, respectively. On the other hand, Kempas experienced the highest compressive strength at 2 hours of holding time. The compressive strength of porous bioSiC ceramics was mainly influenced by porosity and SiC compositions. In addition, the amount of bioSiC increased with the increase of holding time of up to 3 hours. As a result, a larger wall thickness would be produced and hence, resulted in higher compressive strength. Figure 7 shows a representative SEM micrograph of bioSiC from Kapur at 3 hours of holding time which showed the wall became thicker due to the formation of bioSiC.

The increase in wall thickness can be explained by the decrease in pore size with the increase in holding time. The measurements of pore size in the longitudinal and transverse direction of the bioSiC sample were done as shown in Figure 8(a) and compared to the original pore size as shown in Figure 8 (b). It is evident that the pore size decreased from 214 μm to 168 μm in the longitudinal direction, as well as 130 μm to 109 μm in the transverse direction, respectively.

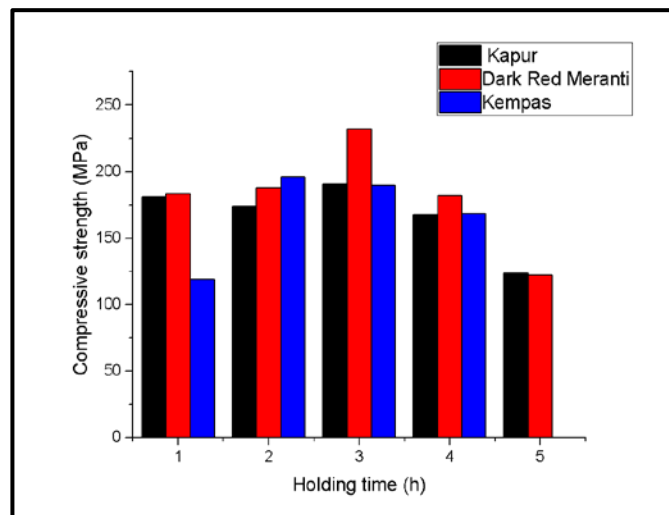


Figure 7: Compressive properties of BioSiC derived Kapur, Dark Red Meranti and Kempas at 1500 °C.

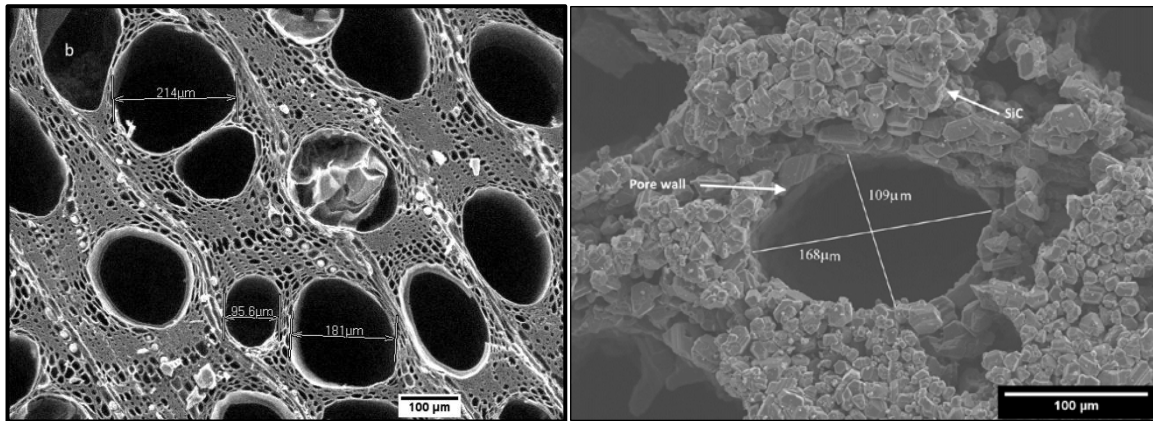


Figure 8: SEM micrograph of the pore wall structure of (a) original structure; (b) Kapur at 3 hours holding time.

The trend of data points obtained from compressive strength indicates that the compressive strength decreases with the increase of porosity, which is in good agreement with the literature. In porous SiC ceramics fabricated by partial sintering technique, it was reported by Fukushima et al. (2008) that the maximum compressive strength is 513 MPa with 39% porosity. Furthermore, the compressive strength of 61 MPa with 54% higher porosity was obtained by Eom et al. (2007) depending on the processing parameters. Based on various processing parameters in the replica technique, a very low compressive strength of 0.3 MPa with 90% porosity was reported by Yao et al. (2006). On the other hand, a better compressive strength of 70 MPa with 55% porosity (Greil et al., 1998) was also reported. In an investigation into porous SiC ceramics processed by sacrificial preform technique done by Eom et al. (2008), it found that the maximum compressive strength was 600 MPa with 30% porosity and a minimum compressive strength of 4.8 MPa with 78% porosity. In another study done by Kim et al. (2011), it was also proven that the compressive strength data of porous bioSiC ceramics processed by direct foaming obtained a higher strength at low porosity while the minimum strength obtained was 51 MPa at about 62% porosity.

5 Conclusion

The effect of holding time from 1 hour to 5 hours was investigated on the conversion of carbon preform into bioSiC. Increasing holding time during infiltration enhances the formation of bioSiC up to 3 hours; however, a longer reaction to 5 hours reduces the bioSiC formation. This is because the longer the reaction time, the heating that allows the capillary to flow the excess silicon into the structure is prolonged. This may cause cracking of bioSiC, and thus reduces the performance of bioSiC. In contrast, a longer reaction time increases the crystallinity of bioSiC. Results from Scanning electron microscopy and X-ray diffraction indicate the transformation of carbon into bioSiC as many bioSiCs are formed at the inner surface of the pore walls. The bioSiC derived from Dark Red Meranti has the highest mechanical strength compared to Kapur and Kempas. Therefore, the temperature at 1500 °C and 3 hours of holding time are the optimum parameters for the bioSiC to have a better mechanical performance in terms of compressive and flexural strength.

6 Availability of Data and Material

Data can be made available by contacting the corresponding author.

7 Acknowledgement

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