RESEARCH ARTICLE

Synthesis and Thermomechanical Behavior of SiC/Si Compounds Derived from Wood Waste

M.L. Benavides-Salinas, D.L. Mayta-Ponce, F.A. Cuzziramos-Gutiérrez, G.P. Rodríguez-Guillén, F.A. Huamán-Mamani*

The traditional method of manufacturing SiC compounds is associated with a serious environmental problem, mainly due to the need for large amounts of energy (generally derived from oil) to reach processing temperatures (typically above 2500 ºC). In addition, the chemical reaction that gives rise to the formation of SiC has CO and CO_2 as by-products. Therefore, in this work an alternative method to manufacture SiC/Si composites using waste from the wood industry as the main raw material was developed. SiC/Si composites were fabricated by infiltration of molten silicon into carbon preforms at 1500 °C. The carbon preforms were obtained by pyrolysis (in an inert Ar atmosphere) of four types of resin-carbon mixtures. The carbon used in the mixtures was obtained by pyrolysis of sawdust powder.
The mechanical and thermomechanical behaviour in uniaxial compression was studied at a constant compression rate of 0.05 mm/min at different temperatures (ambient, 1100 °C and 1400 °C). The maximum resistance values found were in the range of 58 and 384 MPa, while the Young's modulus values were between 40 and 120 GPa. The porosity found in the materials was between 1 and 4%. Finally, the fabricated compounds presented a homogeneous microstructure of interconnected silicon carbide in gray contrast and dispersed and unconnected whitish phases of uniformly distributed silicon.

Introduction

Silicon carbide (SiC) is considered one of the most promising candidates in a variety of industrial applications due to its combination of desirable properties and its low cost, which is why it has been a focus of interesting research in the field of porous materials [**1**-**3**]. The traditional manufacture of Silicon Carbide, either in bulk, powder or pieces of predefined shapes and sizes; are associated with various environmental problems, because: (i) They require very high processing temperatures, typically above 2500 °C, and to reach these temperatures, petroleum-derived fuels are required, (ii) the chemical reaction that gives rise to the SiC molecule has by-products such as greenhouse gases such as CO and CO_2, (iii) for the manufacture of SiC parts, in predefined shapes and sizes (tubes, bars, plates, profiles), the solid state reaction known as sintering is traditionally applied [**1**,**2**]. This technique of joining powder particles is carried out at very high temperatures (above 2000 °C), due to the high thermochemical resistance of the SiC powder, and (iv) extractive manufacturing processes (cutting, roughing, polishing, milling, among others) carried out on SiC blocks or pieces are usually very complex and generate a large amount of waste, due to the intrinsic fragility of SiC [**6**].

The accumulation of waste from extractive manufacturing processes also aggravates the environmental problem of the SiC manufacturing and transformation industry. However, several efforts are being made in the investigation of alternative manufacturing methodologies for SiC. The most studied investigations are those that use vegetable precursors as raw material. These precursors go through thermal calcination processes (to obtain carbon) and reactive infiltration of metallic Si in carbonaceous preforms to obtain SiC pieces [**7**,**8**].

From the above, in the present work several Carbon-Organic binder mixtures were studied, to advance in the knowledge of the volumetric relationship in the mechanical response of SiC/Si compounds at variable temperatures.

Experimental

Manufacture of SiC/Si compounds

Sawdust powder from Capirona-type wood (collected in sawmills in the city of Iquitos, Peru) was used as initial raw material. The sawdust powder was dried in an oven at

Grupo de Investigación en Ciencia y Tecnología de Materiales, Departamento de Ciencias Naturales, Universidad Católica San Pablo, Urb. Campiña Paisajista Quinta Vivanco s/n, Arequipa 04001, Peru

*Corresponding authors:
E-mail: mirandabenavides7@gmail.com

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100 °C for 24 hours, then it was sieved through ASTM mesh no. 80. The sieved sawdust was pressed in a 30 mm diameter metal steel mold and subsequently the pressed sawdust was subjected to a thermal calcination process in an inert argon atmosphere, up to temperatures of 800 °C for 1 hour.

After calcination, the carbon obtained was ground in a planetary mill and sieved through an ASTM mesh no. 140. The sieved carbon powder was used for the manufacture of various carbon-binder mixtures, in this work phenolic resin (CAS no. 9003-35-4) was used as a binder. Four types of mixtures were prepared, whose volumetric proportions C-B are shown in **Table 1**.

Table 1. Volumetric ratio used in the preparation of carbon preforms.

Type	Carbon (C)	Binder (B)
A	0.7	1
B	1	1
C	1.5	1
D	2	1

The C-B mixtures were pressed in a cylindrical steel mold with a pressure of 5 tons. The samples were subjected to the calcination process under the same conditions (800 °C, 1h) obtaining carbon preforms.

Finally, the carbon preforms obtained were cut into discs with a thickness of 3 mm that were subjected to a reactive infiltration process under vacuum and at a maximum temperature of 1550 °C for 1 hour. The Si:C volumetric ratio was 2.96 : 1 (30% excess of silicon with respect to the stoichiometric amount for the amount of carbon to be infiltrated).

Physical, morphological and mechanical characterization of SiC/Si compounds

Physical characterization consisted of determining the geometric density, real density and porosity of the manufactured compounds, using a Micrometrics helium pycnometer, model AccuPyc II 1345, of German manufacture.

The morphology of the samples was studied by observations of a CoolingTech optical microscope, model 1600X, on previously polished surfaces.

On the other hand, uniaxial compression tests at a constant compression speed of 0.05 mm/min and at variable temperatures (room temperature, 1100 °C and 1400 °C) were carried out to determine the maximum strength and stiffness of the manufactured compounds. These tests were carried out in a Spanish-made electromechanical multi-test machine (MicroTest EM1/50/FR series) with a 50 kN capacity cell. The machine had a high temperature oven coupled to carry out the thermomechanical tests. The samples tested were nominally 2.5x2.5x5 mm^3.

To ensure the thermal equilibrium of the samples, the heating rate to the target temperature was 10 °C/min and the duration of the isotherm was thirty minutes before performing the test.

Results and discussion

Physical and morphological characterization

Fig. 1 shows the average values (in blue letters) of the geometric densities, real densities and porosity of the manufactured SiC/Si composites.

The densities obtained were from 2.37g/cm^3 to 2.62 g/cm^3. On the other hand, the compounds presented porosities in a range of 0.98% and 4.29%.

Fig. 2 shows the polished optical microscopy micrographs of the SiC/Si compounds, in all cases two well-differentiated phases could be seen, on one hand, the interconnected SiC phase in gray contrast and, on the other hand, phases isolated, dispersed and disconnected whitish corresponding to uniformly distributed silicon.

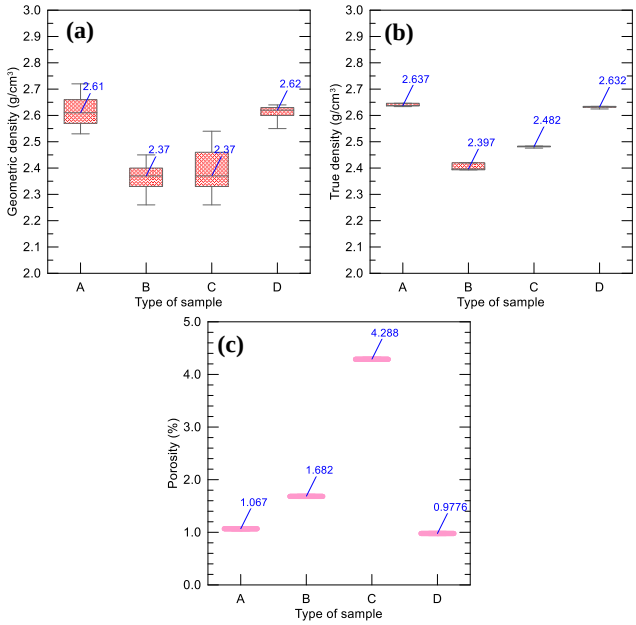

Fig. 1. Values of: (a) geometric density, (b) real density and (c) porosity for SiC/Si compounds studied.

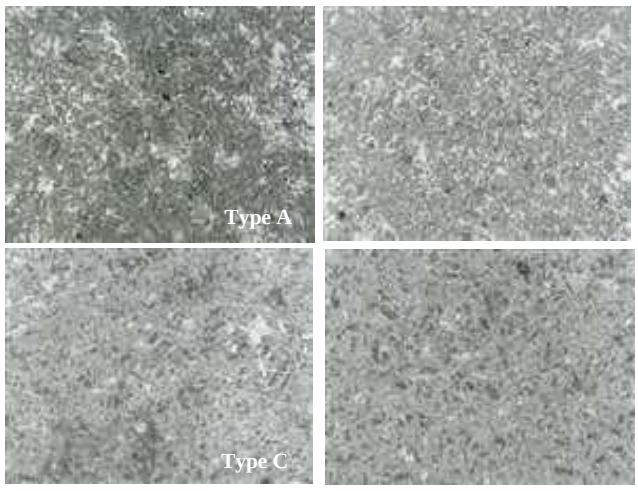

Fig. 2. Optical microscopy micrographs for the SiC/Si compounds studied.

Mechanical and thermomechanical properties

Fig. 3 shows stress vs. curves. deformation for SiC/Si composites manufactured from sawdust powder from capirona wood.

The mechanical behavior of the SiC/Si compounds showed a higher resistance at a temperature of 1100 °C with a maximum value obtained of 409 MPa. While, at room temperature and 1400°C, the maximum resistance values obtained were 182 MPa and 313 MPa, respectively.

A semi-ductile behavior is observed at 1400 °C, which can be attributed to the presence of liquid Si in the sample. As the temperature increases, the silicon gradually softens (as it approaches its melting point) causing a significant deformation in the material, obtaining a decrease in the modulus of elasticity.

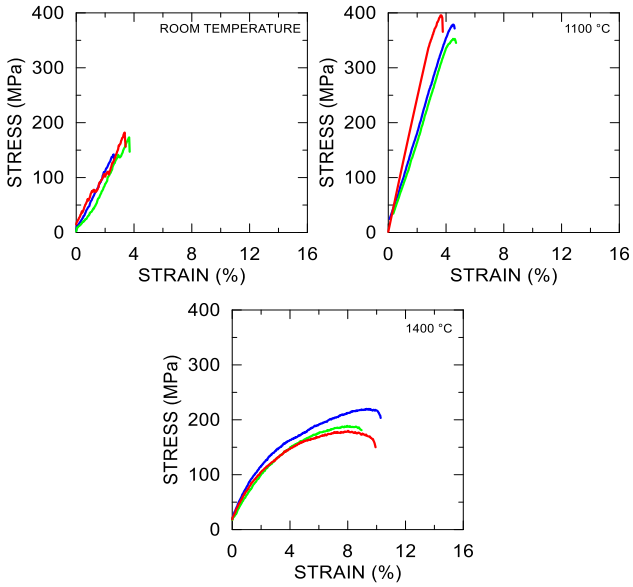

Fig. 3. Effort curves vs. deformation of SiC/Si composite evaluated in thermomechanical tests at (a) room temperature, (b) 1100 °C and (c) 1400 °C.

Fig. 4 shows the upper and lower limits for the set of maximum resistance values under conditions of three different temperatures for each type of mixture.

The behavior of the SiC/Si compound is brittle at room temperature, a premature fracture of the material is observed due to the presence of discontinuities in the compression surface

At 1100°C, a systematic increase in the maximum mechanical resistance can be seen. This behavior is due to the thermal equilibrium reached, which allows an accommodation of the surface discontinuities, obtaining a result more typical of silicon carbide.

Finally, at 1400°C, a decrease in the maximum resistance is observed, which is due to the isolated silicon present in the structure, which, being close to its melting point, undergoes softening. This indicates that the distribution of silicon is also present at the grain boundaries, causing slippage in the structure and a decrease in mechanical strength.

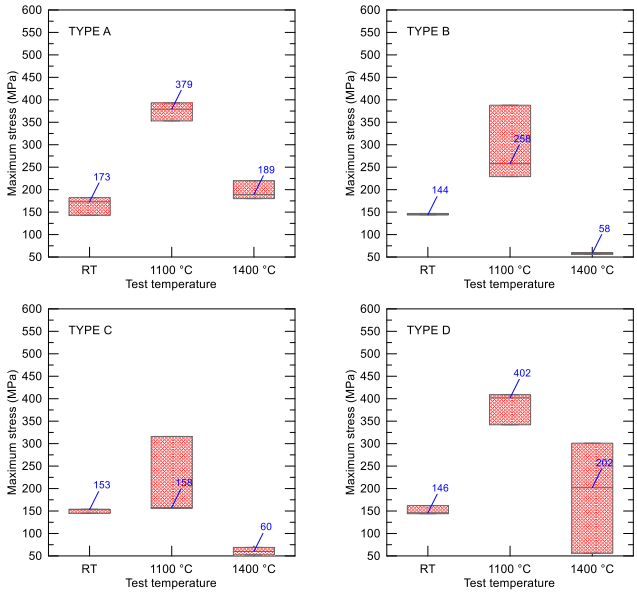

Fig. 4. Maximum strength values as a function of test temperature of SiC/Si compounds (a) type A, (b) type B, (c) type C and (d) type D.

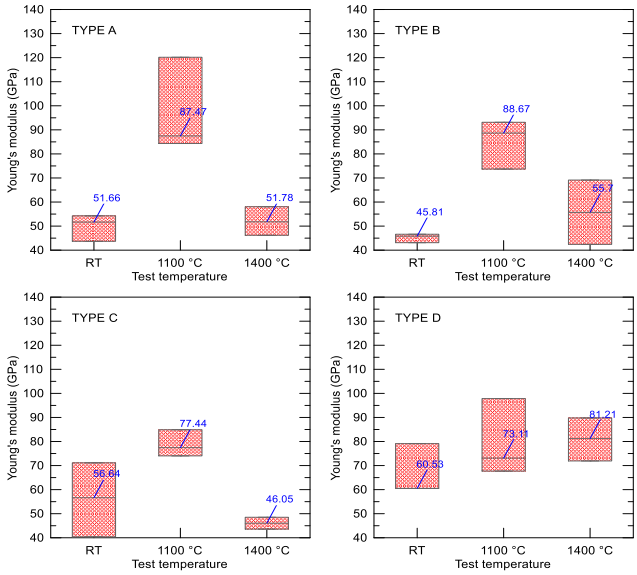

Fig. 5. Young's modulus values as a function of test temperature for SiC/Si compounds (a) type A, (b) type B, (c) type C and (d) type D.

Fig. 5 shows the Young's modulus of the SiC/Si compounds under conditions of three different temperatures for each type of mixture.

The mechanical behavior of the material at room temperature presents a decrease in stiffness, this could be originated in the discontinuities in the compression surface present in the sample. When subjecting the sample to compressive forces, these discontinuities generate internal cracks causing a premature failure of the material, therefore, a decrease in the slope in the curves stress vs. deformation.

On the other hand, at 1100°C this phenomenon does not occur thanks to the thermal stability reached by the sample, which allows the increase of the slope where silicon carbide predominates.

At 1400°C, the silicon present in the compound begins a softening phase, (as the temperature approaches the melting point of 1450°C) the metallic phase is the one that controls the mechanical response process, reducing the value of Young's modulus, compared to the behavior observed at 1100 °C.

Also, can be observed that the samples that present a lower porosity, also present a lower value of the Young's modulus (A, B and C) that can be seen, specifically, in the results of the tests carried out at room temperature and at 1400 °C.

Conclusion

SiC/Si composites were successfully manufactured using capirona wood sawdust powder as the main raw material through a pyrolysis process, followed by reactive infiltration. The fabricated compounds presented a homogeneous microstructure of interconnected silicon carbide in gray contrast and dispersed and disconnected whitish phases of uniformly distributed silicon.

The density and porosity found in the composites ranged from 2.37 g/cm^3 to 2.62 g/cm^3, and 1 to 4%, respectively. The mechanical and thermomechanical behavior in uniaxial compression at different temperatures (ambient, 1100 °C and 1400 °C) was evaluated.

The maximum strength values found were in the range of 58 and 384 MPa, while Young's modulus values were between 40 and 120 GPa.

The mechanical behavior at room temperature was brittle for all the studied compounds, due to the discontinuities in the compression surface of the samples, at 1100 °C the mechanical response was controlled by the SiC phase, while at 1400 °C, the Silicon controlled the process.

The mechanical behavior was similar for the four types of mixtures C-B studied; therefore, it could be suggested that the mechanical response of this SiC/Si composite material is independent of the type of mixture manufactured, at least in the range of proportions of this work.

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Conflicts of interest

"There are no conflicts to declare".

Keywords

Thermomechanical behavior, SiC/Si compounds, wood waste, silicon carbide, mechanical properties

Supporting information

Supporting informations are available online at journal website.

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(a) Scientific article

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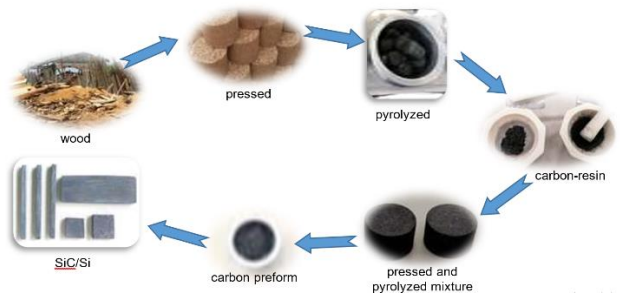
Authors biography

Bachelor of Materials Engineering. Thesis student in the Materials Science and Technology research group (CITEM) of the San Pablo Catholic University.
Research lines: composite materials, sustainability in materials

Graphical abstract



SiC/Si composites were manufactured using waste from the wood industry, sawdust was pressed in a 30 mm diameter metal steel mold and subsequently the pressed sawdust was subjected to a pyrolization process, up to temperatures of 800 °C for 1 hour.
After calcination, the carbon obtained was grounded and sieved.
The sieved carbon powder was used for the manufacture of various carbon-binder mixtures, the carbon preform were pressed and pyrolyzed. Finally, this preforms were infiltrated with Silicon powder to obtain SiC/Si composites.