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Production of Porous Silicon Carbide Ceramics by Starch Consolidation Technique

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Abstract. Silicon carbide is used to produce abrasive and high-temperature structural ceramic materials due to its mechanical and chemical properties. The possible applications of porous silicon carbide ceramics are diesel engines catalysers and molten metal filters. In the last years the starch gained importance as a pore-forming and consolidation agent, due to it is environmental friendly and easily processing. The current work uses starch (corn, rice and potato) as pore forming and consolidation agent. The samples sintered at different sintering times were characterized by density and microstructure (XRD, SEM). The results show that the samples presented porosity between 20 and 40% and the microstructures obtained is homogeneous with a pore size similar to the starch particle added.

Introduction

Dense silicon carbide is used to produce materials for high-temperature structural applications due to its mechanical performance. Silicon carbide porosity can be adjusted, achieving a good porosity-mechanical strength relationship according to the application. [0-0]

Porous silicon carbide ceramics have been produced by several methods, which are denominated as replication methods, pre-ceramic polymers and recently gelcasting. The replica method was used in two aspects: the transformation of a natural matrix (live) and a polymeric sponge in a ceramic material. Natural matrix was studied by several researchers.[0,0,0] The porous silicon carbide derived from wood has the same mechanical behavior of the source material, in the axial direction the mechanical properties are superior compared to the perpendicular direction, thus producing anisotropic bodies with high compressive strength in the axial direction. [0, 0] The polymer sponge replication method is cited in a study where, the bodies obtained had the porosity between 73% and 87%. [0]

In the case of pre-ceramic route many studies can be cited. [0-0] In the first study, pre-ceramic polymers were combined with several pore-forming materials (nylon, carbon fiber etc.), resulting in materials with a similar porosity but with different shapes of pores, this indicates that it is possible to tailor the microstructure (the shape of pores) to the application of the porous ceramics. [0] In another work a mixture of polysiloxane, sintering aids and 5% of micro-spheres are studied. The obtained porosity ranged from 32% to 64%, controlled by the amount of polysiloxane in the mixture. [0] Finally in other study was concluded that by increasing the amount of sacrificial phase in the mixture porosity increases too. In the same study was show that increasing the sintering time the microstructure changes due to the transformation of $\beta \rightarrow \alpha$ grains of silicon carbide. The obtained porosity in this study ranged from 35 to 95%. [0]

Starch showed important role as a pore-forming agent, due to environmental friendly issues, easy processing, burnout without generation of toxins and commercial availability. The control of process permit to choose size, shape and fraction of porous in the material until reach 50%, above this level the porosity is hard to control. This problem was caused by agglomeration of the starch that decreases the homogeneity of samples. [0,0]

In the current work different starches were used as pore and gelling agent; the samples were densified in two conditions to provide a difference in the microstructure; density and porosity are measured by Archimedes method, the microstructure using Scanning Electronic Microscope (SEM) method and X – ray diffraction (XRD) were evaluated.

Experimental Procedure

In this study a powder containing 90% SiC (B17C – HC Starck), 6% Y_2O_3 (HC Starck) and 4% Al_2O_3 (CT-3000SG Alcoa) was milled in an attritor milling (4 hours at 300rpm). And dried in a rotoevaporator, the obtained powder was sieved through a 325 MESH sieve. This mixture was denominaded BC (basic composition). Before mixture with BC powder the starches were characterized by laser scattering.

The suspensions were prepared with 50% in weight of solid contend (a mix of 20-30% in volume of starch related to BC) and distilled water. The mixture was made at magnetic stirrer. The suspension containing starch was poured in cylindrical molds and heated in a thermostatic bath at different gelling temperatures for each starch. [0] The molds were kept in the gelling temperature for 2 hours. The samples were demoulded and dried (24 hours at 40 º C) in a kiln. The samples obtained are summarized in table 1.

Removal of starch was performed using a furnace with air atmosphere, with a heating rate of 1ºC/min until 300ºC during 1 hour. The sintering was performed using a graphite resistance furnace (**Astro** 1000, 4560,FP 20, Thermal Technology Inc.) and a sintering profile (5ºC/min until 400ºC/min in vacuum and 15ºC/min until 1850ºC in argonium as atmosphere), with holding time of 1 and 4 hours. Porosity was measured using Archimedes method in distilled water. The microstructure is evaluated by Scanning Electronic Microscope (SEM). The XRD was performed with 2θ varying between 10º a 90º.

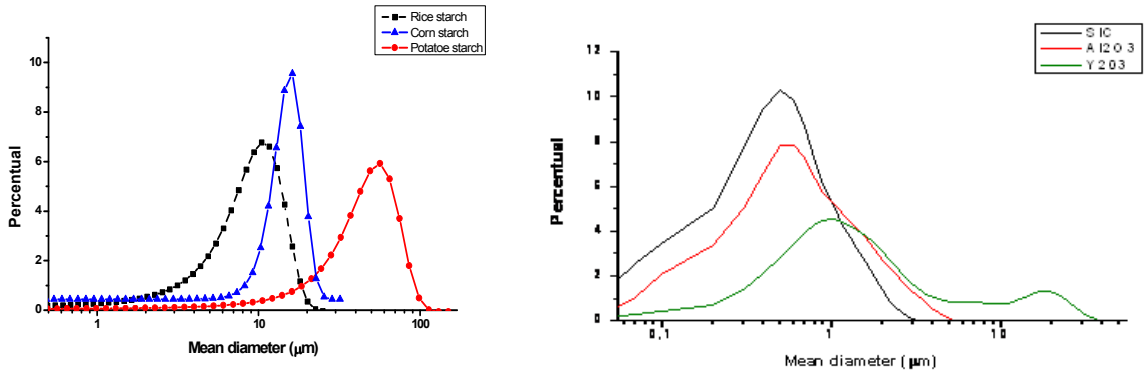
Table 1: Studied Compositions:

Sample	Starch	Starch contents [%volume]
BC	-	0
20C	Corn	20
30C	Corn	30
20P	Potatoe	20
30P	Potatoe	20
20R	Rice	20
30R	Rice	30

Results

The particle size distribution of starches evaluated by laser scattering is presented in Fig. 1(a). Where can be observed that, any starch powder presented a characteristic distribution of mean particle size, with particles mean size about 44,5 µm, 12µm, 8,3µm for potato, corn and rice. The starting materials had their particle size distribution analyzed Fig. 1(b), they had mean particle size of 0.46µm (SiC), 0.69 µm (Al_2O_3) and 3.06µm (Y_2O_3), it is noted that the Y_2O_3 has a bimodal distribution, while the SiC and Al_2O_3 have a monomodal distribution.

Porosity values of the samples after sintering are presented in Fig. 2. Samples without addition of starch presented relative density about 93% and 98%. All samples made with satrch addition showed porosity between 20 and 40%, the samples produced from potato starch had the highest porosity.This study shows that the increase in starch quantity did promote significant increase in sample porosity (about 10%). When the sintering time was increased to 4 hours the difference of the final porosity obtained varies about 10% depending on the starch used. But can be observed that 20P sample do not present reduction in porosity.



(a) (b)

Fig.1: Particle distribution (a) corn, potato and rice starch and (b) the elements of the basic composition.

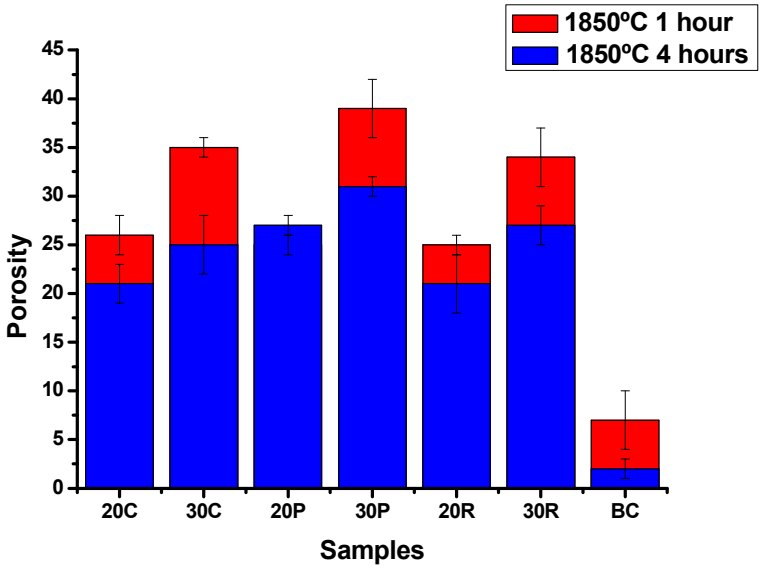


Fig. 2: Porosity of the samples after sinterization.

X-ray diffraction of samples after sintering were show in Fig. 3, increasing the sintering, the peaks related to the oxides decrease, disappearing in some cases and this phenomena can be related with the formation of a amorphous liquid phase in grain boundary. In relation to the silicon carbide can be said that the polytypes found are those expected for sintering temperature used in the work.

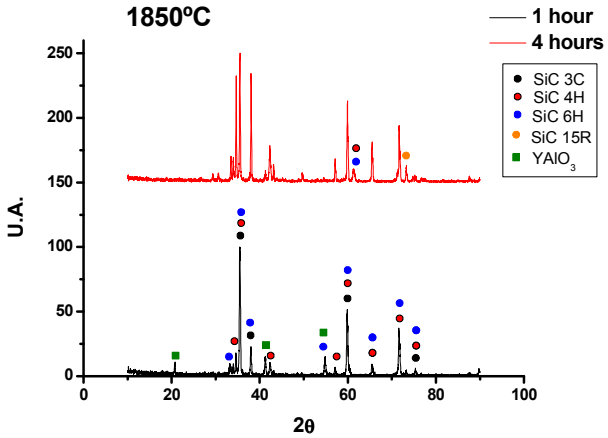
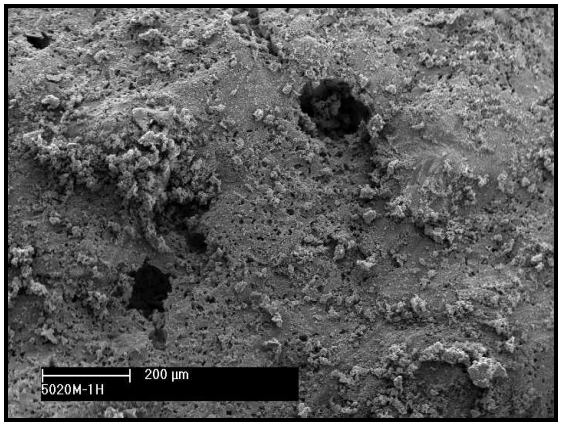
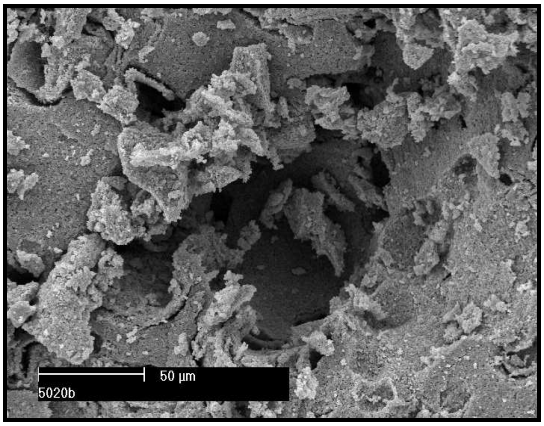


Fig. 3: XRD of porous SiC samples sintered at 1850°C for 1 and 4 hours

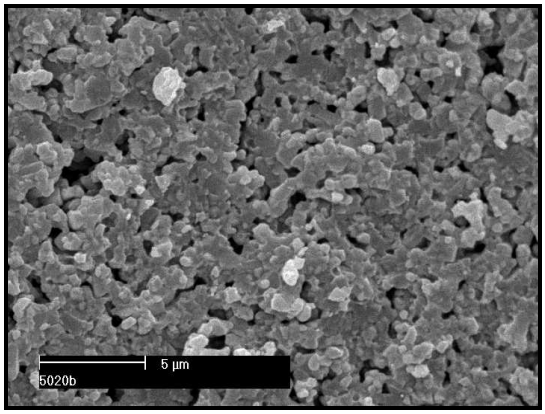
Fractured surface of samples were analyzed by SEM and the obtained images are presented in Fig 4. In Fig. 4 (a), is possible to observe porous and cracks created by compressive test, the cracks had started in the porous and spread through the sample (20C – 1850ºC /1hour). The porous morphology is showed in Fig. 4(b) (sample 20P – 1850ºC / 1 hour); the mean size of porous was about 40µm. This value was close of potato starch particle size and agrees with reports in the literature.[0] In Fig. 4(c), residual porosity is observed in the ceramic matrix of sample 20P – 1850ºC /1hour).



(a)



(b)



(c)

Fig. 4: Micrograph of the sample 20C/1850ºC /1 hour (a), 20P/1850ºC /1 hour (b), and the ceramic matrix 20P/1850ºC /1 hour (c).

Conclusion

Studied samples presented porosity range of 20% to 40%. The results show a strong relation between the starch content and the porosity. This effect indicates that the porosity of the sample can be tailored by changes in the starch contents. The microstructure had a homogeneous porosity.

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