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Processing of Ultralight Porous Al_2O_3-Ceramics by Biotemplating of Bamboo

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Abstract. Biomorphic alumina ceramic with a long, large and oriented growth of alpha alumina grains suitable for the devices such as prosthetic implants, dental implant and control drug release etc. Ultralight porous alumina was synthesized from a carbonaceous preform derived from bamboo using biotemplating technique. Carbonaceous preform (C-preform) of bamboo precursor was prepared by controlled thermal processing in a muffle furnace for pore formation. Al_2O_3 was inserted into C-preforms by sol-gel method applying repeated infiltration of low viscous Al_2O_3-sol and then dried and sintered at 1500°C in air atmosphere resulting of an ultra-light monophasic Al_2O_3 ceramics with replication of the pore structure of the bamboo C-preforms. The microstructure of porous Al_2O_3 was characterized by scanning electron microscopy (SEM). Open hierarchical porous structures surrounded by alumina struts web with nominal porosity of 80% inside the biomaterial are visualized which lacking of interconnectivity and low strength.

Keywords: Bamboo precursors, Carbonaceous preform, Ultralight cellular alumina.

1. Introduction

The development of highly porous ceramics with a cellular porosity has attracted increasing interest in the recent years for its specific properties such as high surface area, high permeability, high-temperature stability, low weight and low thermal conductivity. Porous ceramics are potential for a variety of applications including filters for molten metals and hot gases, light-weight structural components, electrodes, sensors, bioreactors, catalyst carriers, radiant burners as well as porous implants in the area of biomaterials [1-2].

Artificial porous materials are costly, less available, limited compatibility, specific to Medical/surgical procedure with period of application/usage, relate to characteristics of host with high demand to support in the body natural conditions [3-4]. Compared to synthetic porous materials, natural biomaterials are often available at low cost and causes less health and environmental hazard problems with renewable materials and increased concerns about the environment for people producing the composites as compared to glass/Ceramic materials based composites [5]. The primary reason for the development of biocomposites from natural materials by biotemplating is flexible to the of type/distribution of the reinforcing phases in the composites, surgical techniques, and sterilization methods and the possibility to obtain biocomposites having a wide range of mechanical and biological properties [6].

Biotemplating techniques, in which biological materials are used directly as template structures for high-temperature conversion into technical ceramic materials, overcome this time rate problem [7]. Recently porous ceramics with tailored morphologies and structures synthesized from carbonaceous preform (C-preform) derived by replicating of plants have received wide attention for unique combination of anisotropic structure and properties [8–13]. The retention of parent plant micro-cellular anatomical features and macro-structural integrity in the C-preform plays a very important role in promoting reactivity and producing diverse microstructures bearing on the type of parent

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biostructure [14]. Previous works on biotemplating was mainly focused on the preparation of biomorphous carbide ceramics, e.g. SiC via a reaction of the biological material derived from biocarbon with different Si-infiltrants such as Si-melt, Si/SiO-gas, Si-containing polymers as well as SiO_2-sols [15-17]. Several investigations have been also carried out on the synthesis of biomorphous oxide ceramics. Preparation of Al_2O_3- and ZrO_2-fibers by oxidizing hydrated cellulose fibers impregnated with solutions of aluminum chloride and zirconium chloride was done earlier [18-19].

Porous ultrahigh alumina ceramics is considered as potential biomaterials and can be produced from Bamboo by biotemplating. Bamboo, the cheapest cellulosic and long fiber can be effective to prepare porous ultrahigh alumina ceramics [20]. In the open literature, due to the certain limitations of artificial materials we want to develop bamboo as biotemplating materials for cellular alumina production is very scarce. Thus the present work studied the fabrication of porous alumina retaining the morphological and macro and micro-structural features of the bamboo precursor.

2. Experimental

2.1. Conversion into C-preform of Bamboo precursors by thermal processing

At first sun-dried bamboo after removing the outer hard layer were cut into short lengths (0.02 -0.05 m). The bamboo samples were then oven-dried at 65°C for 24 hours overnight and weight and dimension of this oven dried bamboo were measured. Oven-dried bamboo samples were converted into C-preforms by applying a controlled thermal processing. The bamboo samples were placed in stainless steel close chamber and reduced in an electrically heated muffle furnace under self-generated atmosphere allowing the gases and volatiles to escape through a cold trap where tarry materials condensed. Temperature was raised at different rates as 2 °C/min up to 400°C, and then 1°C/min up to 800 °C and 2 °C/min up to 900°C. The samples were held for 1hour at this pick temperature. The active C-preforms derived from bamboo were recovered after cooling the furnace and weight of the preforms were measured.

2.2. Alumina sol infiltration into C-preform and drying

The alumina sol was prepared by mixing alumina powder (Fluka) by making a 5% weight of aqueous solution. The bamboo C-preform samples, taken in a small beaker, were placed in a vacuum glass chamber fitted with a dropping funnel containing the alumina sol. When the vacuum reached steady state after a certain period of time, the alumina sol was allowed to fall on to the samples drop by drop. The bubbling was appeared during infiltration. At the end of the infiltration, the bubbling was ceased and the vacuum was released. The samples were taken out and were wiped free of any loose sol adhering to the exterior surfaces. Normally eight to ten cycles of infiltration treatment was carried out on sample depending upon weight gained of alumina in preform and sample dimensions. After the infiltration process of each cycle, alumina infiltrated carbon preforms were dried in air and after that in an oven (100-120°C) for 2 hours to 4 hours as per sample dimensions.

2.3. Conversion into cellular alumina ceramic by Sintering

Alumina infiltrated C-preform samples were taken in a crucible. The sample containing crucible was placed in a high temperature furnace for sintering the alumina sol infiltrated oven dried C-preforms and heating was applied. Samples were heated slowly (2°C/minute) in air to sintering temperature of 1500°C. The materials were allowed to stay for 60 minutes at the sintering temperatures for complete oxidation of carbon and to sinter the ceramic. Then the furnace was cooled down to 800°C with a rate of ~2°C/minute followed by furnace cooling to room temperature (Figure 1). Finally cellular ceramics sample was taken from the furnace.

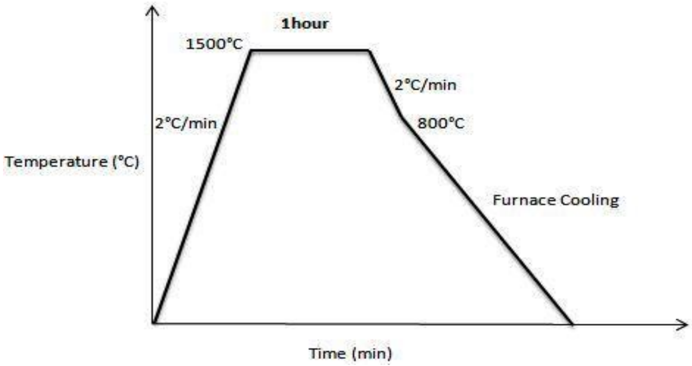


Figure 1: Sintering cycle of the Ceramic Alumina sample

2.4. Characterization of cellular ceramics

In a FESEM, Electron beam emitted from a field emission electron gun which uses secondary electrons that condense to a fine probe to examine microscopic structure for surface scanning with much higher resolution and three-dimensional appearance of its images with its large depth of field. The signal electrons emitted from the specimen are collected by a detector, amplified, and used to reconstruct an image. Field emission scanning electron microscope (FESEM) was employed to study of the structure of raw bamboo; C-preforms derived from bamboo and cellular alumina ceramics morphologies with a long, large and oriented growth of alumina. This test was carried out to determine/Characterize the pore within cellular ceramic, and to detect the presence of any micro defect in the composite. Images in the FESEM are recorded digitally with ability to generate an image by averaging multiple scans for the same area to reduce background noise in imaging.

3. Results and discussion

3.1. Weight change of bamboo carbon preforms formation and infiltration

The raw bamboo shorts losses its weight after drying overnight and after C-preform formation. Percentages of this losing weight have presented in (Table 1). Bamboo is composed of cellulose, hemicellulose, lignin, and water. The weight loss occurs after drying of the removal of water in Bamboo and it is around 10%. When the dried samples were heated to 900°C at different heating rate in controlled environment, weight loss occurred drastically due to the removal of the hydrogen, oxygen and other volatile matters from cellulose, hemicellulose, and lignin in bamboo and it is around 90%. Finally a porous carbon preform was obtained with desired bamboo template structure.

Table 1: Weight loss of Bamboo shorts after preform formation

Sample	Weight of Raw sample	Weight after drying (gm.)	Percentage of weight loss after drying	Weight after carbon preform (gm.)	Percentage of weight loss after C-preform
Bamboo	1.3050	1.1860	9.1187%	0.1136	90.42%

Compare to the previous research, our weight loss percentage is higher 90.42% to ensure porous carbon preform to the weight loss of Jute sample 75.20% [21]. It is found from another research by PK Mandal and R Majumdar that the weight loss of Jute sample and Cane stick is 75.20% and 69.35% respectively [22]

The carbon preform sample derived from bamboo was infiltrated with alumina from alumina sol in several cycles. Infiltration of alumina in carbon preform is a critical step for the fabrication of porous alumina ceramics. After completion of each infiltration cycle, the sample was dried and measured the weight gain (Table 2). It can be seen that the weight gain in grams was increased with increasing infiltration cycle and it is around 70% after the final infiltration cycle. Weight gain of alumina infiltrated was related to drying after each infiltration. Drying temperature and time should be selected in a way that no crack can be formed during drying.

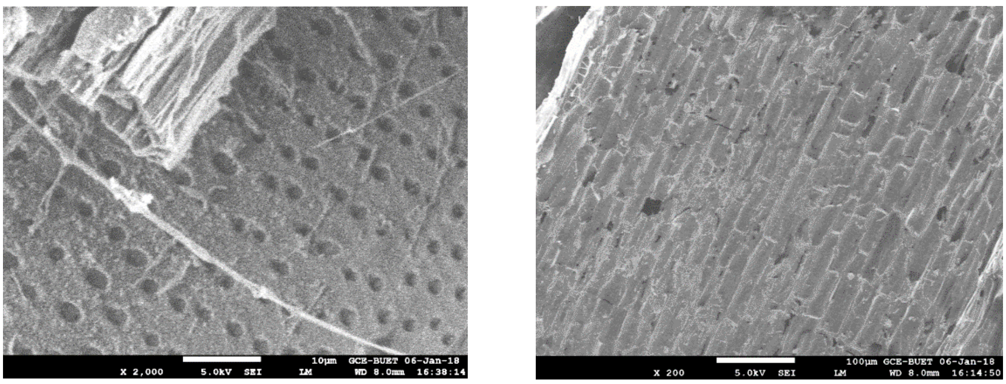
Table 2: Weight gain of C-preform sample after alumina Sol infiltration

Sample	1st Cycle (gm.)	2nd Cycle (gm.)	3rd Cycle (gm.)	4th Cycle (gm.)	5th Cycle (gm.)	6th Cycle (gm.)	7th Cycle (gm.)	8th Cycle (gm.)	Percentages of weight gain after final infiltration
Bamboo	0.1768	0.2225	0.2630	0.3084	0.3320	0.3570	0.3610	0.3750	69.71%

PK Mandala, R Majumdar and KK Mukherjee also found that the weight gain percentage of Jute sample after infiltration is 68.1% [21] which is similar to our findings 69.71% to develop a heavy mass of ceramic alumina sample.

3.2. Microstructural analysis

Figure 02 shows the physical structure of cross-sectional and longitudinal section of raw dry bamboo. Bamboo contains parallel fibers that are reinforced along the axial direction of the culm which is evidence from the cross-sectional view (Figure 2a). From the longitudinal section, cellular structure is observed in (Fig. 2b)



(a) (b)

Figure 2: Showing the microstructure of dry raw bamboo sample (a) cross-section and (b) longitudinal section.

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The dried bamboo sample was converted into C-preform and structure of the sample is shown in (figure 03). SEM image illustration for C-preform samples are showing elongated channel pores formed due to the leaving of organic and volatile matters resulted from the heat treatment at 900°C in an inert and self-generated gases. This pours are filled with the accumulation of alumina from alumina sol over cycles of sol-infiltration to fabricate porous alumina.

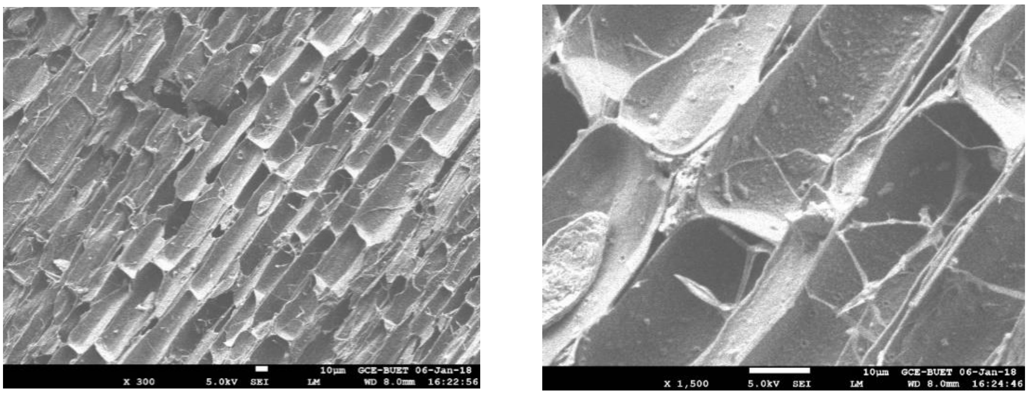

Figure 3: SEM image showing the structure of C-preform sample

The final alumina ceramics were fabricated from a thermal C-preform in sintering process at 1500^0C, but it was weak to measure the strength. SEM image of the alumina ceramic obtained are shown in (Fig. 04). SEM investigation reveals the porous structure of the alumina ceramics. The porosity oriented within its fiber where alumina particles gathered in this porosity and after sintering porous structure of alumina has formed. The effective properties of porous ceramics are not a simple function of porosity but depend on all features of the microstructure including pores between solid spheres, isolated (closed) spherical pores etc. Bamboo has microstructure of large number of open pore shown in (figure 3). Alumina phase also exhibited round pore. Although larger pore can be seen, the cellular morphology and structure with a longitudinal disposition of carbon preform, their orientation and interconnectivity are not perfectly maintained in the fabricated alumina ceramics.

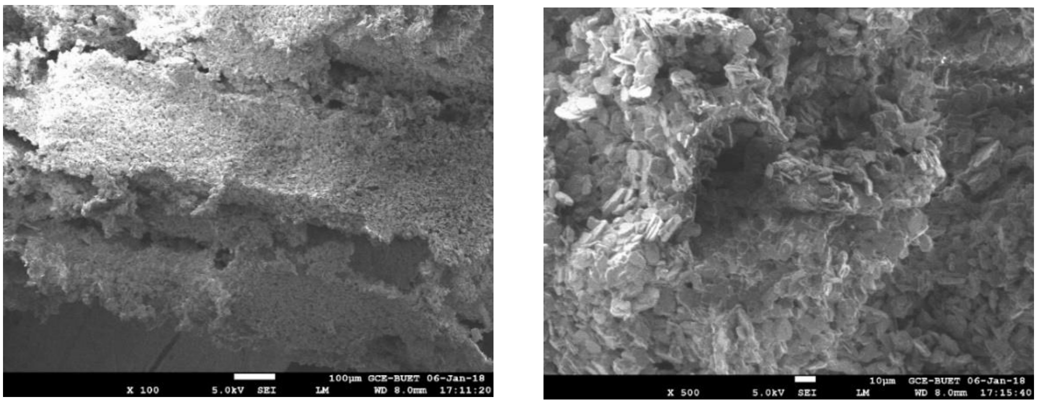

Figure 4: SEM image of sintered alumina sample

4. Conclusion

The specific aim of this study was to produce porous alumina ceramics in the image of bamboo. Carbon preform from the bamboo was successfully prepared by thermal process. The porous alumina was fabricated from the alumina infiltrated C-preform sintered at 1500^0C. Large and oriented growth of alumina grains porous network was found which is likely to be suitable for biomaterial. The pore network of sintered alumina with its strength is important criteria in biomaterial applications and can be obtainable with further study on particle size effect on pore size and shape, infiltration and proper sintering cycle.

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