

CARBONIZED WOOD - A PRECURSOR FOR ADVANCED CERAMICS

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INTRODUCTION

The current technology for the production of high performance ceramics involves the preparation of expensive high purity, submicron powders that require special handling and highly controlled consolidation followed by a high temperature sintering step. In addition, expensive machining and finishing operations are often needed to obtain the specified dimensions of the final component.

This paper describes a novel processing concept that has the potential for producing advanced ceramics of net shape using wood and other natural fibrous plants as a precursor. We describe results from experiments using carbonized wood for the production of Silicon Carbide and Aluminum Nitride. Also described is the carbonization process used to yield a porous carbon material which maintains the cellular structure of the precursor wood while eliminating the formation of macro-cracks usually associated with activated charcoal.

Wood has a complex polymeric structure consisting of lignin and carbohydrate (hemicellulose and cellulose). The lignin has a basic structural unit of a methoxy-substituted propylphenol and usually comprises 18-35% of dry wood. Lignin imparts a woody, rigid structure to cell walls that distinguishes wood from other fibrous plant materials. The carbohydrates are the principal components of the cell wall and usually make up 65-75% of dry wood. The cellulose portion of the carbohydrates are typically 40-50% of the wood and is partially crystalline. The hemicellulose is a mixture of amorphous branched-chain readily hydrolyzable polysaccharides consisting of hundreds of sugar residues. In its elemental form wood consists of approximately 50% carbon, 44% oxygen and 6% hydrogen.

The thermal decomposition of wood in an inert atmosphere, unlike incineration, can be carried out under a highly controlled condition. Thermal analysis of wood in both air and nitrogen has been performed to determine the oxidative stability or decomposition characteristics of the different polymeric components. Typical decomposition temperatures fall in the 250-400°C range. The decomposition is an endothermic reaction which enables close control over the wood carbonization process.

Results from current experiments indicate that monolithic ceramic bodies can be produced from both wood and carbonized wood. Techniques used for the material characterization presented here include x-ray diffractometry, x-ray radiography and scanning electron microscopy.

EXPERIMENTAL

Three different woods were selected for the experiments presented in this work, Balsa (*Ochroma pyramidale*), Tulip Poplar (*Liriodendron tulipifera*) and Red Oak (*Quercus rubra*). Thermal treatments were carried out in one of two furnaces capable of controlled heating in an inert atmosphere. One furnace used an Inconel retort 16 inches deep which was used for carbonizing larger samples to temperatures up to 1100°C. A second furnace with alumina tube liner was used for conversion to silicon carbide (SiC) or aluminum nitride (AlN) at temperatures up to 1600°C. An argon

gas atmosphere was used for conversion to SiC and nitrogen for conversion to AlN.

Carbonization of the wood samples was typically conducted over a 24 hour period. Specimens were cut to size using standard woodworking tools (power miter saw or table saw) and then placed in the furnace retort with a nitrogen flow of initially several L/min then 1 L/min when the heating sequence was begun. Furnace ramp rate of 30°C/hr was used to bring the samples to 500°C at which point the wood had decomposed to yield a porous carbon specimen. This heating schedule was adequate for carbonizing specimens up to 8 in long. Longer times would be needed for larger samples.

Two methods for introduction of silicon were used. First was to place a wafer of semiconductor silicon on top of the end grain of carbonized Red Oak and carbonized Tulip poplar. Second was to use a vacuum to assist the impregnation of wood or carbonized wood with a colloidal silica sol. For both methods samples were then heat treated to 1500°C in an Ar atmosphere.

X-ray diffractometry was performed using a Cu source with spectral filtering by a Ni foil. Collected scans were compared to ICDD PDF data. Specimens were scanned whole without grinding them into a powder. X-ray radiographs were taken with a Faxitron cabinet system using Polaroid high speed film as a recording medium. SEM was used to investigate the morphology of the carbonized wood and the wood-ceramic that was produced. Since both were electrically conductive, no conductive coatings were used.

RESULTS AND DISCUSSION

The carbonization process yielded bodies of glassy carbon with a porous microstructure retained from the original wood. No macro or micro scale cracking was observed in those specimens carbonized using the above heating schedule. Figure 1 is a photograph of carbonized Balsa wood and a similar sample before carbonization. Shrinkage due to carbonization is evident. We found an 80% weight loss and up to a 40% volume shrinkage. Linear dimensional changes were found to be dependent upon the orientation of the cellular structure with values ranging from 15% to 35%. Different wood species had different reduction in dimensions owing to their unique cellular microstructures. The carbonized Balsa wood has a calculated porosity of over 95% which provides a very low density structural carbon.

Specimens heat treated with silica or silicon were identified as SiC by x-ray diffractometry. Figure 2 is a diffraction pattern from a sample of carbonized Tulip Poplar combined with silicon to form SiC. High intensity, narrow peaks are evident of highly crystalline ceramic matching the powder diffraction file for Moissanite (synthetic SiC PDF #29-1129). A similar result was found for the sample prepared from Red Oak and silicon.

The specimens infiltrated with silica sol also produced ceramic monoliths of SiC. Diffraction patterns were obtained from Red Oak wood with silica which was carbonized and converted to ceramic by one thermal process. Though the peaks were not as intense or narrow as found in for the silicon experiments, it is clear that SiC is present. Peaks between 20° and 30° 2-theta is an indication of unconverted silica. Those peaks

were found to change greatly in amplitude when the scanned area was rotated suggesting that the remaining silica has a preferred orientation. Experiments from other wood species and carbonized woods infiltrated with the silica sol gave similar results.

A x-ray radiograph of carbonized Red Oak, Red Oak wood-SiC and carbonized Tulip Poplar-SiC is shown in Figure 3. The variation seen in the brightness of the images is an indication of the difference in absorption between carbon and SiC. The Red Oak-SiC specimen is much more absorbing than the carbonized Red Oak. The cellular microstructure is clearly maintained. The penetration of the silicon into the carbonized Tulip Poplar can readily be investigated by viewing the specimen shown in Figure 3. This technique is being used to optimize silicon impregnation for thorough conversion of the microstructure to ceramic.

Microstructural characterization by SEM indicates the cellular structure of the wood is maintained by carbonization without the production of cracks. This technique is also enabling the study of the carbon to ceramic conversion process. A micrograph showing the transition between carbonized wood and SiC ceramic is shown in Figure 4. Changes in the thickness of the cell walls can be seen with the SiC to the left of the image and carbon to the right. The micrograph in Figure 5 is a higher magnification showing SiC crystallite morphology at the surface of a cell wall.

CONCLUSIONS

The method for producing porous carbon materials from wood precursors has been presented and is found to have many uses. The carbon bodies produced can be used as means to producing net shape ceramic bodies. Results presented here demonstrate that SiC ceramic can readily be produced by heat treatment of the porous carbon body with silicon or silica while maintaining the unique microstructural characteristics of the precursor wood. Considering the great diversity of cell sizes and distributions in woods of different species it is possible to choose beforehand the type of microstructure desired. Carbonization which avoids the formation of macrocracks in the porous carbon body has been performed and described. Many other applications for monolithic porous carbons produced by this method are currently being studied.

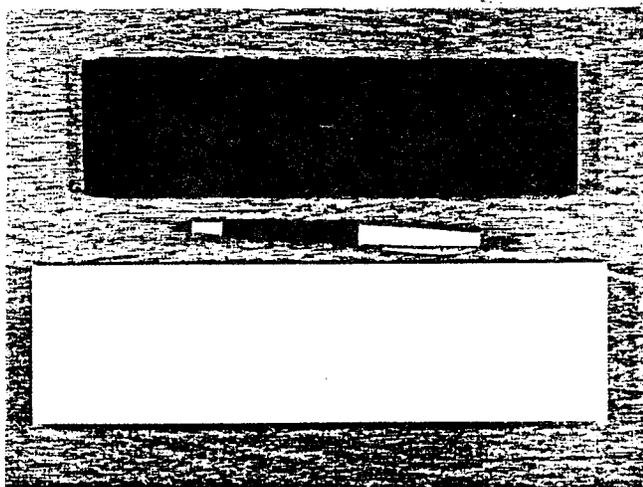


Figure 1. Carbonized Balsa wood compared with wood.

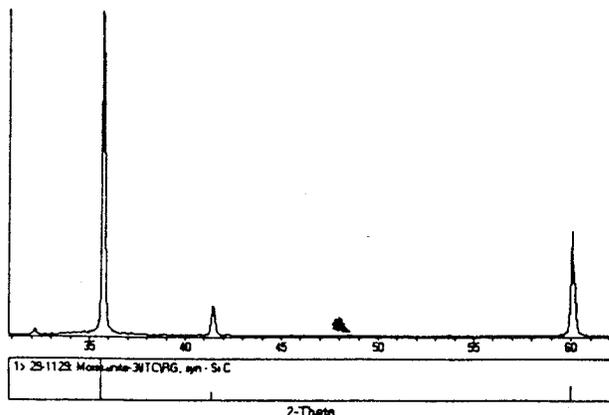


Figure 2. X-ray diffraction scan of SiC produced from wood.

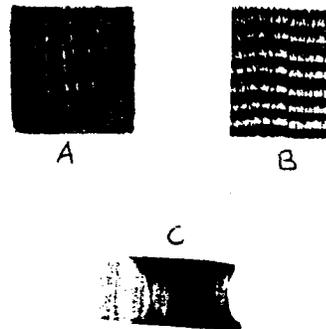


Figure 3. Radiographic image of samples converted to ceramic, A) Red Oak-SiC, B) carbonized Red Oak, C) Tulip Poplar-SiC (full scale).

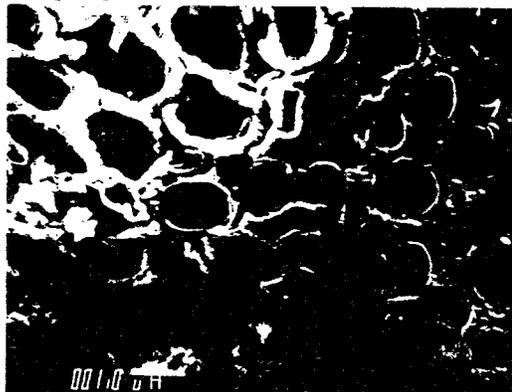


Figure 4. Micrograph of transition between carbonized wood (on right) and SiC ceramic.



Figure 5. Micrograph of wood derived SiC showing crystallite morphology.