The effects of Yttrium Oxide nanoparticles in processing and mechanical performance of Nicalon™/KiON CERASET® continuous fiber ceramic composites by preceramic polymer pyrolysis method have been investigated. Ceramic fiber reinforcements (Nicalon™) and preceramic polymer (KiON CERASET®) was mixed with nano size fillers in the presence of a surfactant agent which gives a good dispersion of the particles during the process. Three different types of nanoparticle filled composites were manufactured in this work; the test samples consisted of 0%, 5%, and 10% of Nanoparticles with respect to the matrix volume. A characterization analysis of the samples using scanning electron microscopy revealed good quality of the parts. Four-point bending test was also conducted to evaluate the mechanical performance of the ceramic composites samples at room temperature. The three types of samples were manufactured and tested accordingly to ASTM 1341-97 standards. The test results exhibited an increase in flexure strength for the 5% composition by approximately 4.55% and a decrease of flexure strength for the 10% composition by approximately 7.56% with respect to the 0% composition specimens.

INTRODUCTION

Continuous fiber ceramic composites (CFCC) are novel, advanced class of materials in which there is an increased interest in their elevated-temperature applications such as aerospace and automotive structural members, hot gas nozzles, and jet engine components. CFCCs are able to withstand very high temperatures, (>1200°C) with the capability to preserve high strength and modulus properties [1-3]. Ceramic materials however, possess many unsolved problems such as low fracture toughness and strength, and poor resistance to creep and fatigue [4, 5]. In this work, an attempt has been made to solve these problems by incorporating Yttrium Oxide Nanoparticles in CFCC.

MATERIAL SYSTEM

The three main constituents in the manufactured CFCC are: 1) Plain weave Nicalon™ Ceramic fiber reinforcement, 2) KiON CERASET® preceramic polymer, and 3) Yttrium Oxide (Y2O3) Nanoparticles. Nicalon™ is a silicon carbide (SiC) type fiber and has a desirable combination of modulus, strength, density, and electrical properties with retention of these properties at elevated temperatures up to 1400°C. The primary function of fibers or
reinforcements is to carry load along the length of the fiber to provide strength and stiffness in the longitudinal direction. Reinforcements can be oriented to provide tailored properties in the direction of the loads imparted on the end product.

KiON CERASET® preceramic polymer was used in this research study. The low molecular weight, thermosetting Polyureasilazane combines the processing flexibility of liquids with the high pyrolytic yields previously demonstrated by higher molecular weight thermoplastic precursors. This polymer contains repeat units in which silicon and nitrogen atoms are bonded in an alternating sequence. Once heated to temperatures in excess of 1,000°C, the polymer converts to Silicon Carbide-containing ceramic materials [6] in presence of Nitrogen gas.

Yttrium Oxide Nanoparticles was used to reinforce the CFCC specimens. This particular type of Nanoparticle was chosen because of their desirable mechanical properties, i.e., small average particle size (29 nm), high melting point, and small bulk density. The density and surface area are 1 g/cc and 42 sq.m/gm, respectively [7].

**MANUFACTURING PROCESS**

The manufacturing process for the test samples had to go through the following procedure: wet lay-up, curing using autoclave, and polymer pyrolysis/reinfiltration cycles. In the wet lay-up process, the preceramic polymer is mixed with the surfactant agent and nanoparticles using a high-speed stirrer. Surfactant agent is a denser, viscous liquid used for proper dispersion of nanoparticles. Sheets of the woven fiber are cut in desired dimensions and are stacked on top of each other as the matrix is impregnated in between and onto the layers. The desired thickness of the test specimen was achieved by using 14 layers of the fabric. The wet lay-up laminate is then vacuum bagged as shown in Figure 1 for curing and consolidation inside an autoclave.

![Figure 1. Schematic diagram of the vacuum bag/autoclave technique.](image)

Figure 1 shows the typical curing process used for all of the test samples. For the final step of pyrolysis/reinfiltration cycle, the edges of the laminated composite plates are trimmed and samples of required dimensions (ASTM C 1341-97) are cut using a diamond blade cutter. The cut samples are then pyrolyzed in a high temperature tube furnace in an inert nitrogen gas environment to convert the preceramic polymer into ceramic.
The pyrolysis process, see Fig. 3, converts the polymer into ceramic. During this process, micro cracks and voids form throughout the structure due to the processing of the material at high temperature. Thus, subsequent reinfiltration of the preceramic polymer is necessary to reach a weight convergence factor within 1% of a previous measurement.

![Figure 2. Autoclave cure cycle.](image2)

![Figure 3. Pyrolysis cycle.](image3)

**EXPERIMENTAL SETUP**

Five test samples of each composition (i.e. 0%, 5%, and 10% Yttrium Oxide nanoparticles) were cut to specified dimensions as per ASTM 1341-97. The nominal width, thickness, and length for all of the test specimens were approximately 12.85mm, 3.63mm, and 180mm, respectively. A four point bending fixture was used with each loading point positioned uniformly along the test specimen. The distance between each loading points was 1/3 of the outer support load span. The four point test is favored over a three point bending test due to a larger portion of the test specimen that is subjected to maximum stresses, therefore allowing more accurate statistical data [8]. The experimental setup is shown below in Figure 4 where the test specimen is shown mounted between the four point bending.

![Figure 4. Experimental setup for the four point bending test fixture. Test geometry II-B (4 Point) was used per ASTM 1341-97.](image4)
An XY plane recorder was used to simultaneously plot the respective load and extension during the testing process. The test rate was determined to be approximately 1.18 mm/sec [8]. The maximum stress in the outer surface at the point of maximum stress, i.e., flexural strength ($S_U$), can be calculated using the specimen dimensions and maximum load obtained from the four-point bending test, and is given in Equation (1) [8].

\[ S_U = \frac{P_U L}{b d^2} \]  

(1)

where $P_U$ is the maximum load in the flexure test in N, $b$ is the specimen width in mm, $d$ is the specimen thickness at the point of break in mm, and $L$ is the length of the outer support span.

## RESULTS AND DISCUSSION

All of the test specimens fractured in between the inner loading points, which was required in order for the test results to be valid. This insured that failure was not due to faulty manufacturing procedures or a failure due to shear. Table 1 below shows the results for the average maximum load ($P_U$ in N) and deflection ($D$ in mm) of the test specimen midpoint. Note that Ni/CE-n represents the CFCC specimen composed of Nicalon™ fibers/KiON CERASET® with n% of Y$_2$O$_3$ nanoparticles.

Table 1. Flexural strength for Ni/CE-n% test specimens. (Parentheses indicate standard deviation values).

<table>
<thead>
<tr>
<th>Specimen</th>
<th>d, mm</th>
<th>Pu, N</th>
<th>D, mm</th>
<th>Strength, MPa</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni/CE-0%</td>
<td>3.86</td>
<td>37.37</td>
<td>1.02</td>
<td>31.85 (1.06)</td>
</tr>
<tr>
<td>Ni/CE-5%</td>
<td>3.40</td>
<td>30.65</td>
<td>1.04</td>
<td>33.30 (4.05)</td>
</tr>
<tr>
<td>Ni/CE-10%</td>
<td>3.64</td>
<td>31.30</td>
<td>0.62</td>
<td>29.43 (3.45)</td>
</tr>
</tbody>
</table>

![Figure 5. SEM images of a CFCC specimen: a) sample cross-section, b) void content.](image-url)
The flexure strength for the 5% composition increased approximately by 4.55% compared to the 0% composition. However, the 10% composition decreased in flexure strength by approximately 7.56%. The explanation for this low strength for the 10% composition is believed to be due to the excessive amount of nanoparticles in which the formation of agglomerates was highly feasible. These agglomerates may have acted as inclusions or high stress concentration areas. However, a 5% incorporation of Yttrium nanoparticles produced positive results in which the flexural strength did indeed increase. Future study would possibly be to optimize the percentage of nanoparticles so as to achieve an optimum flexural strength. Nanoparticle inclusions had no effect on the manufacturing process. Characterization of the manufactured CFCC specimens was performed using a SEM, which revealed good part quality and proper consolidation of the final specimen as shown in Figure 5.

CONCLUSION

Three different types of continuous fiber ceramic composites with varying % of Yttrium oxide nanoparticles were manufactured following preceramic polymer pyrolysis route. Yttrium Oxide with an average particle size of 29 nm was used as the nanoparticle reinforcement. Woven ceramic fiber reinforcements (Nicalon™) fabric was used here. Preceramic polymer (KiON CERASET®) was mixed with the nano size fillers in the presence of a surfactant agent, which gives a good dispersion of the particles during the process. A characterization analysis of the samples using scanning electron microscopy revealed excellent quality of the parts. All the samples tested fractured inside the uniformly stressed region of flexure specimen (i.e., between the upper loading points of the fixture). The test results indicated a +4.55% increase and a -7.56% decrease in flexural strength for the 5% and 10% composition test specimens, respectively, when compared with test specimens without nanoparticles. Nanoparticle inclusions had no effect on the manufacturing process. Future work improvements would be to devise a method to optimize the amount of nanoparticles to include into the specimens in order to achieve optimum mechanical properties of this novel class of advanced composite materials.

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