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Boron Nitride Nanotube (BNNT) Reinforced Non-Oxide Ceramic Matrix Composites

by

Troy Y. Ansell

October 2023

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I. INTRODUCTION

A. BACKGROUND

High temperature materials, specifically ultra-high temperature ceramics (UHTC) are increasingly being investigated for use in applications like atmospheric reentry and hypersonic vehicles. The high temperatures experienced by, for example, the leading edge of a platform exceeding Mach 5 (the threshold for hypersonic speeds) requires materials that can continue to function at temperatures around 1100 °C or higher [1]. Common engineering materials used for structural applications cannot withstand such extreme temperatures without either active (e.g., transpiration cooling) or passive cooling (e.g., film or ablative cooling) [2]. For passive cooling routes, UHTCs are used to withstand high temperatures protecting the underlying substrate. Thermal protection, however, is not the only application of UHTCs.

Components of communication systems onboard hypersonic vehicles require UHTCs. An example includes apertures used in microwave-based communication since this component will need to be unshielded from the local environment to work properly. In this case, non-oxide dielectric ceramics like silicon nitride (Si_3N_4) or aluminum nitride (AlN) are needed. Unfortunately, the dielectric permittivity[1] in UHTCs like Si_3N_4 or AlN are dependent on temperature. Ceramics see an increase in permittivity with an increase in temperature; however, this permittivity must be kept low in communications. A possible solution is to integrate boron nitride nanotubes (BNNT) into the ceramic.

It has been demonstrated that BNNTs exhibit high thermal conductivities that are comparable to the more familiar carbon nanotubes (CNT) along the axis of the tubes (in-plane) in a temperature range spanning near absolute zero up to room temperature [3], [4]. Perpendicular to the axis (out-of-plane), the material is thermally insulating presenting a unique opportunity to route heat by patterning the BNNT in specific geometries. By routing heat away from ceramic phase, its temperature could be maintained allowing for control of permittivity. Unlike CNT, BNNT exhibit higher thermal stability and oxidation resistance making BNNTs an ideal composite filler

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[1] How much a material polarizes when encountering an electric field is its permittivity.

candidate for high temperature applications [5]. Successful integration of BNNTs into non-oxide ceramic matrix composites (CMC) has the potential to impact numerous platforms across the DoD.

B. RESEARCH OBJECTIVES

The goal of this effort was to incorporate BNNTs into a non-oxide CMC based on Si_3N_4 and to characterize its thermal and dielectric properties at elevated temperatures (> 1000 °C). The original plan was to integrate BNNTs into printable Si_3N_4 slurries. These BNNT-CMC slurries would then be 3D-printed into complex geometries. It was thought that printing would provide adequate shear stress to align the BNNTs normal to the broad surfaces of samples. Binder burn-out and sintering of the printed BNNT-CMCs would have occurred next. Binder burn-out would have (and still will) occurred at the Naval Postgraduate School (NPS). Plans for sintering included use of a vacuum furnace in Spanagel Hall as well as collaborating with the Naval Research Lab (NRL) to use a specialized furnace for difficult-to-sinter materials. Once binder burn-out and sintering was complete, BNNT-CMCs would be characterized. This would have involved the measure of high temperature thermal conductivity and dielectric permittivity (with the latter being measured at terahertz frequencies). Unfortunately, several issues hampered progress during printing of the BNNT-CMC. Printed BNNT-CMC was accomplished but not until the end of July and so the goals of the research had to be modified.

Dielectric testing of BNNT-CMC was one of the original tasks, but a requirement for the application was a test piece that had a diameter of 76.2 mm (3”). This requirement itself presented a problem for printing of Si_3N_4 discs, as 50.8 mm (2”) was the largest attempted and came with significant settling issues, which led to geometric inaccuracies in the final part. Further, issues with viscosity of the slurry caused delay in printing BNNT-CMC discs of standard sizes (25.4 mm or 1”). These complications led to the decision to attempt low temperature fabrication of BNNT-CMCs. Si_3N_4 and other UHTCs are typically fabricated by first compacting powders into a simple shape and then sticking it into a vacuum furnace and heating to around 1800 °C. The part to be sintered will be kept at this temperature for some time in a low-oxygen, inert environment, hence the vacuum. As can be imagined, this method is difficult and requires expensive equipment. Existing alternative methods allow for a reduction in temperature to as low as 1500 °C but still require expensive equipment with low throughput. The method proposed here is to sinter the BNNT-CMC at 1000 °C in a standard muffle furnace using a technique known as the molten salt synthesis (MSS) method [6]. This method is far cheaper than typical fabrication methods and could see higher throughput. An added potential benefit is that the lower temperatures may avoid thermal shock issues experienced by larger ceramics at higher processing temperatures. The first step in this method was to show that pure Si_3N_4 could be fabricated by MSS.

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II. EXPERIMENTAL METHODS

A. 3D-PRINTING CERAMIC COMPOSITES

The composite parts were printed by Lithoz, an Austria-based company that specializes in printing ceramics. Multi-walled BNNTs were procured from Nanointegris and had purities of between 80 to 90% BNNT. Impurities were primarily carbon and oxygen from the fabrication process. The BNNTs were sent to Lithoz who mixed the nanotubes into their proprietary Si_3N_4 slurry (LithaNit 782, Lithoz). Once the BNNTs were properly mixed in the LithaNit 782 slurry, samples were printed in a Lithoz printer, which uses Lithoz's lithography based ceramic manufacturing (LCM) process seen in Figure 1. Much like the familiar polymer-based stereolithography, the slurry is placed in a vat that is transparent to high frequency visible light (i.e., blue light). A build plate is lowered into the slurry and printing begins. The blue light moves along a path following a digital pattern and interacting with the slurry along this described path. The light-slurry interaction causes curing of a part layer-by-layer. Once the full digital pattern had been scanned into the slurry, the build plate is raised, and the "green" printed part is removed from the build plate.

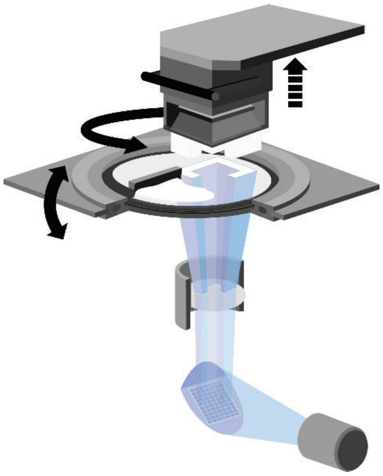

Figure 1. Lithoz's LCM Printing Diagram. Source: [7].

Following printing, binder burn-out is required to remove any residual polymer. This is done by placing the green part into a furnace, typically in air, and increasing the temperature up to 1000 °C. Because of the BNNT, however, the CMC samples in this case would have to see burnout in a low oxygen environment. So, BNNT-CMC samples would be placed in tube furnace (max temperature is 1150 °C) with a vacuum pump. Air is pumped out to a lower oxygen partial pressure and nitrogen gas backfilled into the tube. The sample is kept at 1000 °C for one minute and then allowed to cool in the furnace (known as furnace cooling) back down to room temperature while in a nitrogen rich atmosphere. Once complete, samples would be placed in a vacuum furnace and sintered at between 1700 and 1850 °C in an inert environment for some amount of time. Before heating, air is pumped out such that the partial pressure of oxygen is reduced. To prevent possible vaporization of a material, the chamber is usually backfilled with an inert gas like argon. NPS does not have a vacuum furnace that can go to the temperatures required to sinter Si_3N_4. The sintering times are variable and depend on the sample.

B. LOW TEMPERATURE FABRICATION

Because of the delay in receiving printed parts, the MSS method was pursued for fabricating Si_3N_4 ceramics. Some the printed samples, however, may be sintered by this method; this will be highlighted in future work. The first step of the MSS method is to compact powders. Si_3N_4 powders were obtained from US Research Nanomaterials, Inc. Two phases of the material are stable at room temperature, α-Si_3N_4 and β-Si_3N_4. Both were purchased, but for this work the α phase was used. About 1.0 g of powder was mixed with 2 wt% polyvinyl alcohol (PVA). Mixing was performed in an agate mortar. Powders of Si_3N_4 and PVA were mixed 98:2 ratio by weight in water and ground in the mortar with an agate pestle until a dry powder resulted. The PVA was used as a binder to allow for handling of compacted powders in later fabrication stages. The PVA is water soluble allowing for easy mixing with the silicon nitride powders. The mixed powders were then placed in a pressing die and pressed to between 20 MPa (uniaxial) for several minutes. The compacted powder was then sealed in a FoodSaver® vacuum bag using an edge vacuum sealer (VS1200 FoodSaver® vacuum sealer). The sealed sample was then placed in a cold isostatic press (CIP) die and further pressed with 20 to 40 MPa of hydrostatic pressure. Once pressure was reached, samples were held for several seconds before pressure was released.

At this point, one of two sintering routes was pursued. Either the samples were placed in a larger pressing die and encased in NaCl before being embedded in a bed of loose NaCl powder (schematic seen in Figure 2) or the CIP'd sample was placed directly in the bed of NaCl powder. The crucible with the salt bed and green ceramic (encased or not) would then be heated in air to 1000 °C and held there for three hours. Since many salts including table salt melt between 700 and 900 °C, the sample would be in molten salt at 1000 °C. The molten salt would provide an oxygen barrier to the sample [8]. Once cooled, the salt would crystallize around the sample. To remove the sample, distilled water would be poured over the salt. The salt would dissolve and allow removal of the sample.

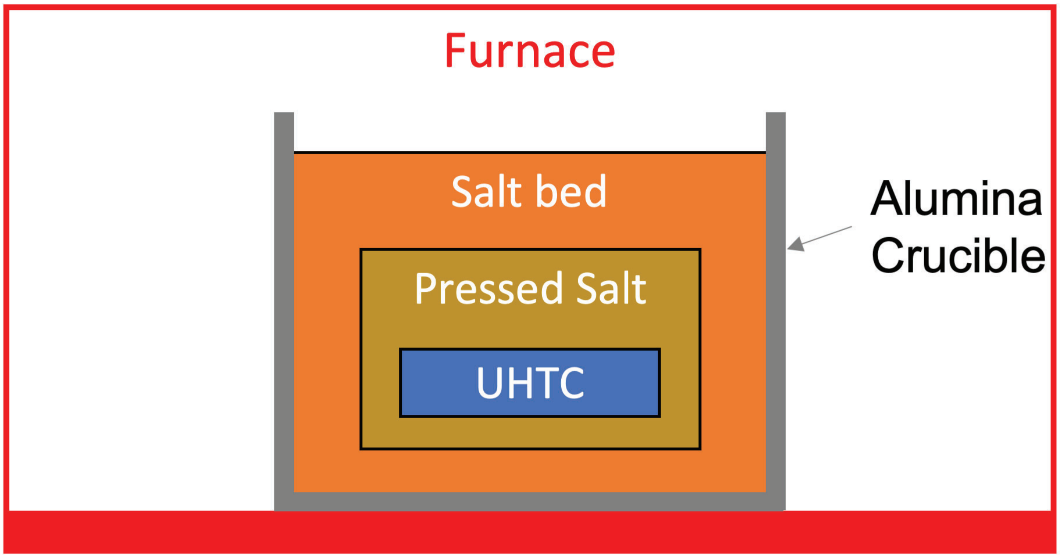

Figure 2. Schematic of the MSS method to sinter UHTC like Si_3N4.

C. SAMPLE CHARACTERIZATION

Once samples were removed from the salt bed, the sample density was measured. A QUINTIX224-1S laboratory scale (Sartorius) was used to measure density. A beaker was filled with distilled water and then placed on the scale. An empty sample holder resembling a holey tray was inserted into the water and the scale was zeroed. The sample was then placed on the tray and the weight was taken. The density was calculated with the Eq. 1,

$$\rho = \frac{\rho_{Water} W_{in\ air}}{W_{in\ air} - W_{in\ water}} \quad (1)$$

where w is the sample weight either in air or in immersed in water and ρ_{water} is the density of water at room temperature. After density measurements, samples were placed in a vacuum furnace for 24 hours to remove any moisture.

Starting powders were imaged in a scanning electron microscope (SEM). Powders were spread thinly across a sticky carbon tape, which was then placed on a SEM stub. The SEM used for imaging of starting powders, fracture surfaces, and sample cross-sections was a Helios 5-UX SEM (Thermo-Fisher Scientific). For imaging of powders, the typical settings used were a beam current of between 25 and 100 pA, a beam voltage of 5 to 20 kV, and a working distance of 4 mm. Samples that had density measurements performed were fractured and the fracture surface placed on clean carbon tape, which again had been placed on a SEM stub. The same SEM settings were used for imaging of fracture surfaces.

Some samples were cross-sectioned for SEM imaging as well as optical imaging, micro- or nano-indentation hardness testing. These samples were cut using a diamond saw set to a low speed. The cut samples were then mounted in an epoxy. The epoxy used was Epofix (Struers). The sample was placed in a silicone mold with the cross-section placed face-down in the mold. The resin was mixed with a hardener to a 15:1 ratio by volume for two minutes and then allowed to rest for two minutes before being poured into the mold with the sample. Once poured the epoxy was allowed to cure for 12 hours before removal from the mold. The mounted sample was then ground using 320-, 500- or 600-, and 1200-grit SiC paper in that order until scratches on the surface were no longer visible to the naked eye. Optical imaging was performed on a Nikon Epiphot 200 optical microscope. Nano-indentation hardness was performed using a G200 nanoindenter (KLA). A modified continuous stiffness method was used for measuring load-unload cycles.

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III. RESULTS

A. PRINTED COMPOSITE SAMPLES

Composite BNNT-CMC samples were printed with Lithoz's LCM technology. Pure Si_3N_4 (0BNNT-CMC) samples were first printed. Then samples with 2 (2BNNT-CMC) and 4 vol% BNNTs (4BNNT-CMC) were printed. The shapes printed included a hemisphere with a base diameter of 25 mm, height of 13 mm, and a 1.0 mm wall thickness or a hexagon, 20 mm at the widest, and a thickness of 2.0 mm. The 2 vol% samples suffered issues with the sample removal and with the slurry, so only strips could be printed. The strips were roughly 15 mm by 2.0 mm. All samples are imaged in Figure 3.

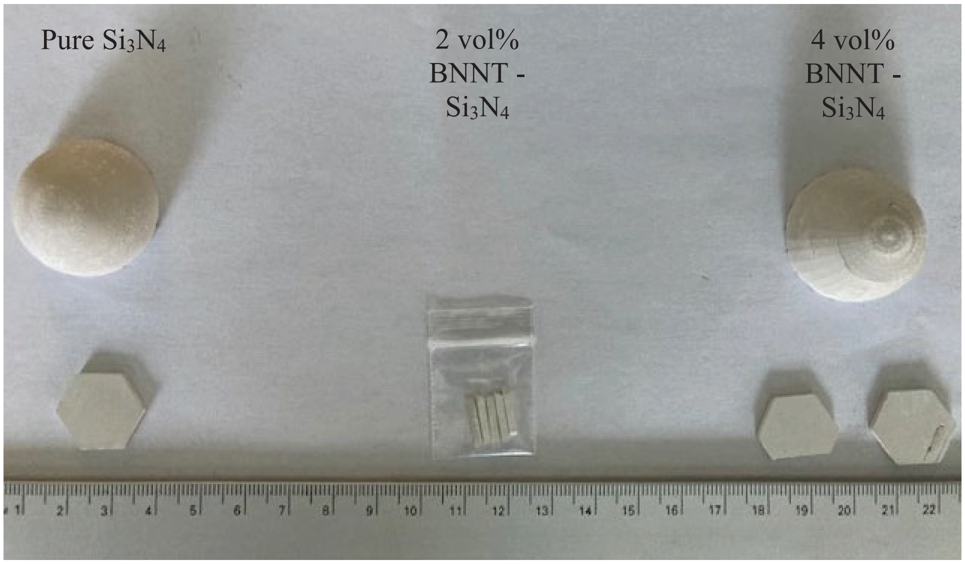

Figure 3. Images of printed, cleaned, and preconditioned Si_3N_4 and Si_3N_4 composite samples. Scale is in centimeters.

Close-up images of the hemisphere samples are seen in Figure 4. In Figs. 4a and 4b are images of the top and undersides of the hemisphere made of pure Si_3N_4, respectively. A complete support structure is seen in the underside of this hemisphere. In Figs. 4c and 4d are images of the top and undersides of the hemisphere made of 4BNNT-CMC, respectively. A large fracture is seen in the topside of this composite sample while on the underside, much of the support structure is missing. Strut width may have been too small to support the overlying material in the hemisphere, so the width was increased for the 0BNNT-CMC hemisphere. Due to breakdown of the support structure in the 4BNNT-CMC hemisphere, inclusions were observed in completed print surface, which led to decreases in fracture toughness and as a result, extensive cracking as seen in Fig. 4c.

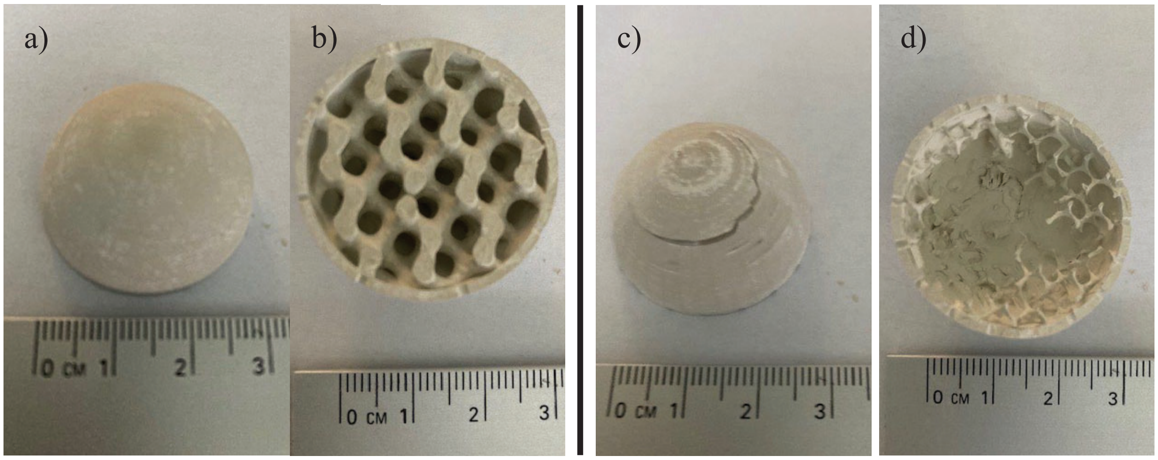

Figure 4. Images of the printed hemisphere samples. In a) the topside of the pure silicon nitride sample, and in b) the underside. In c) the top of the 4BNNT-CMC sample, and in d) the underside.

B. SILICON NITRIDE SAMPLES FABRICATED VIA MSS METHOD

Seen in Figure 5 are SEM images of the Si_3N_4 powder before and after mixing with the PVA binder. Seen in Fig. 5a, the majority of the Si_3N_4 powders are small tubular shaped particles on the order of 1.0 µm. This dimension is just slightly above the 800 nm particle size quoted by the vendor. A bimodal particle size distribution is observed with large particles interspersed with smaller particles (Fig. 5b, and one large particle seen in Fig. 5a). After mixing with PVA, particle agglomerations were observed (Fig. 5c). On the nanoscale, however, no noticeable changes were observed after mixing: the same bimodal size distribution persisted, with smaller tubular particles sitting among larger particles.

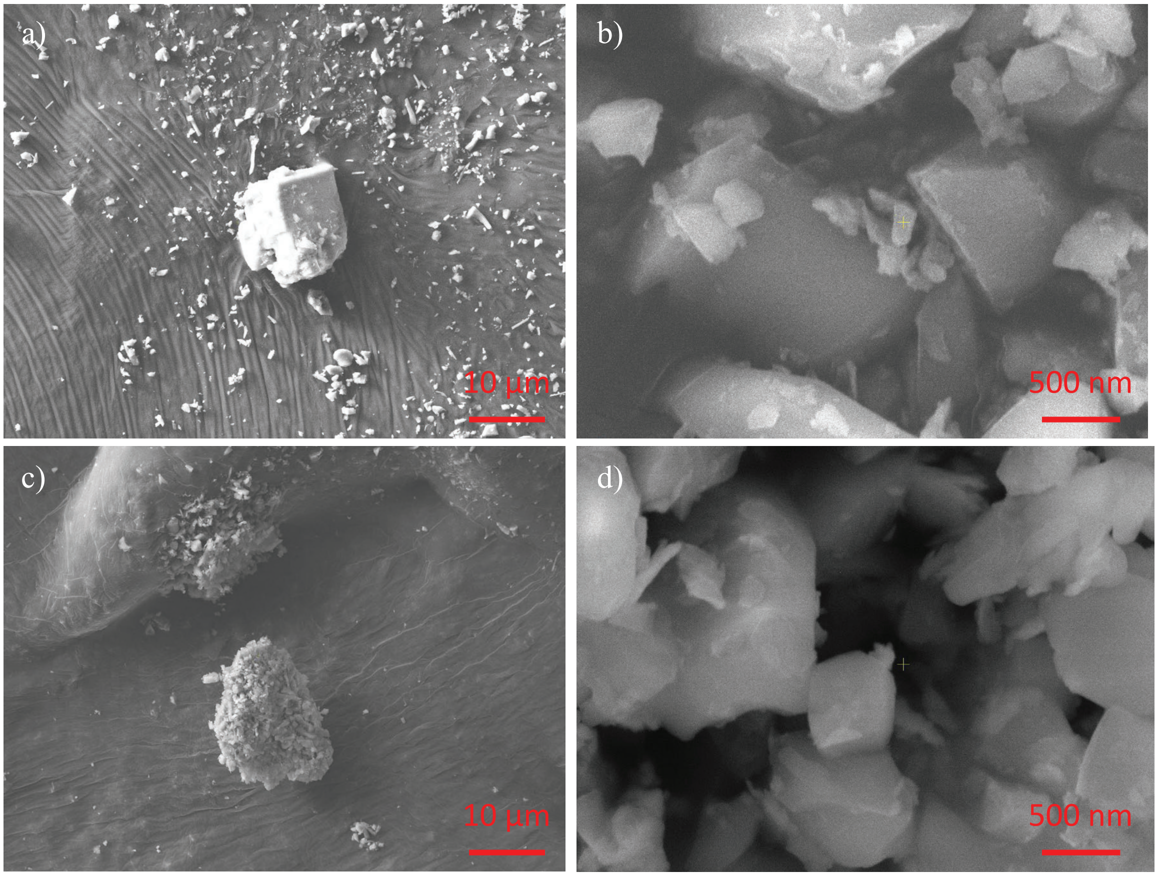

Figure 5. SEM images of unmixed Si_3N_4 powders at a) low and b) high magnification. SEM images of Si_3N_4 powders mixed with PVA at c) low and d) high magnification.

The accepted density of Si_3N_4 is 3.17 g/cm^3 [9]. Once sintered, density was measured and the results of some of the samples are listed in Table 1. As a reminder, samples were pressed in the cold isostatic press (CIP) with pressures of 20, 30, or 40 MPa. What is missing are samples pressed, isostatically, at 30 MPa. These samples consistently fractured during fabrication and processing and so results were not obtained. The average density was found to be 2.48 g/cm^3 ± 0.12 g/cm^3. This corresponds to a theoretical density of 78% (the ratio of measured to accepted density). When splitting results by applied isostatic pressure, and when samples are CIP'd at 20 MPa, the average density was found to be 2.43 g/cm^3 ± 0.09 g/cm^3. When pressing at 40 MPa, the density was 2.63 g/cm^3 ± 0.02 g/cm^3 albeit with a sample size of two. So, the theoretical densities for Si_3N_4 fabricated via the MSS method were 77% and 83% for samples CIP'd at 20 and

40 MPa, respectively. Theoretical density reached up to 84%. It does appear that an increase in the isostatic pressure means an increase in the theoretical density.

Table 1. List of results of sintered samples.

Sample	Cold Isostatic Pressure (MPa)	Mass (g)	Diameter (mm)	Thickness (mm)	Density (g/cm^3)
1	20	1.14	13.5	3.6	2.42
2	20	1.10	13.7	3.4	2.36
3	20	1.24	13.6	3.8	2.37
4	20	1.11	13.1	3.9	2.60
5	20	1.38	13.3	4.5	2.38
6	40	1.23	13.1	4.0	2.65
7	40	1.24	13.1	3.9	2.61

Samples that underwent sintering and survived density measurements were then fractured. Figure 6 shows an example of the fracture surface of a sample CIP'd at 40 MPa. The grain structure appears to indicate some level of sintering occurred. Larger grains coexist with smaller grains, and both are not uniformly distributed across the sample. Further, little porosity is seen at least in the center of the sample. If sintering did not occur, then a higher number of pores would be expected. In fact, a more porous structure was observed in samples that had been pressed at lower pressures. The high volume of contact between grains when pressed at a sufficiently high pressure would promote self-diffusion of Si and N from grain to grain, leading to higher densification and thus improved sintering behavior.

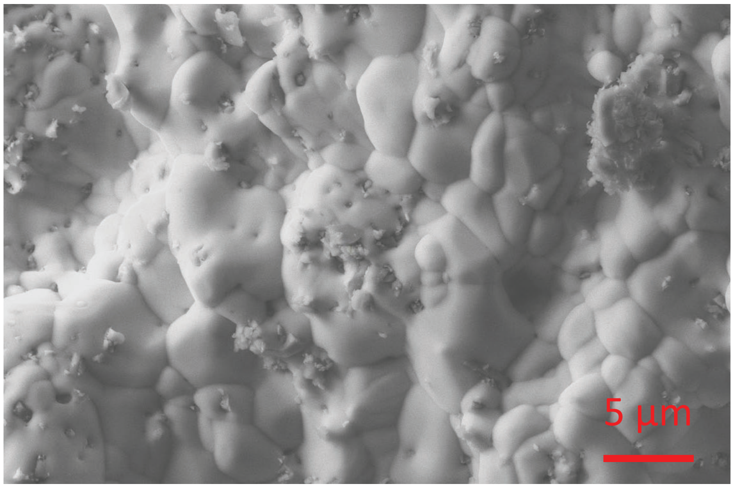

Figure 6. SEM image of the fracture surface of a sample CIP'd at 40 MPa.

Figure 7 shows an image of a mounted sintered sample. What can be seen on the surface is a series of cracks. Because of the low fracture toughness of ceramics in general, much effort is expended in reducing the presence of cracks and, should cracks be present, reducing the crack tip size. The presence of cracks on a ceramic this large would preclude the use of the material in most applications, especially those where thermal shock resistance is required. It was speculated that these cracks are formed when the salt surrounding the sample crystallized, penetrating the sample and initiating cracks. To reduce crack initiation in MSS Si_3N_4 samples, encasing samples in NaCl was avoided and instead samples were placed directly in a bed of loose NaCl powder. It was expected that crystallization would still occur, but most penetration events would be avoided. This did seem to be the result as ceramic pellets where salt encasement did not occur had a reduced amount of cracking and a lower frequency of failure before characterization.

Figure 7. Image of a sintered sample after mounting in an epoxy. The corner of a standard mouse pad is seen for reference.

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IV. CONCLUSION AND FUTURE WORK

A. CONCLUSION

Fabrication of Si_3N_4 based composites where BNNTs were the fillers (BNNT-CMC) was attempted. To orient the tubes in a direction parallel with the top and bottom surfaces of the composites, printing via a lithography based ceramic printing technique was attempted. Issues with the printing progress, however, slowed progress down and prevented electrical and thermal characterization of samples. Printing of composite samples was achieved, but with poor fidelity to a digital model and with significant defects even before binder burnout and sintering. Instead, the molten salt synthesis (MSS) method was attempted first on pure Si_3N_4. It was shown that densities of nearly 84% theoretically can be reached when powders are uniaxially pressed at 20 MPa, cold isostatically pressed (CIP) at 40 MPa, and then sintered in a bed of NaCl at 1000 °C for one hour. Crack initiation was also shown to decrease when pressed samples are directly placed in a bed of loose powder as opposed to first being encased in NaCl pellets.

B. FUTURE WORK

Characterization of the printed samples will be performed to validate (or not) the printing of BNNT-CMCs as a viable method for fabricating these composites. Because of the difficulty in sintering, printed samples will be subjected to the MSS process at least at the later stages. Some samples will be sent to NRL for sintering. Before any sintering takes place, however, binder burnout will be performed in a tube furnace with a nitrogen rich atmosphere at 1000 °C. Thermal and electrical characterization of samples will follow.

For the fabrication of BNNT-CMC samples via the MSS process, the first step is to make changes to the process parameters. Changes to the pressing die, sintering temperature, sintering hold time, and isostatic pressure and the effect to density will be explored. Should the theoretical density improve above 90%, the next step would be to integrate BNNTs into the powders, likely via high energy ball mill or acetone agitation. The MSS process would be repeated for composite powders. One problem with this method is poor control of BNNT orientation. A further investigation would involve looking at ways of orienting BNNTs. This might involve forming a slurry with the BNNTs, Si_3N_4 powders, and additional fluids like water or water with dispersants and surfactants added.

C. ACKNOWLEDGEMENTS

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