Micro-Scale Ceramic Additive Manufacturing for Aerospace Applications

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Primary CNF Tools Used: Nanoscribe Photonic Professional GT2, Zeiss Ultra Scanning Electron Microscope

Abstract:

This paper suggests mechanisms for producing silica glass microscale electrospray emitters for spacecraft propulsion systems and prefaces the potential of these methods for producing microscale ceramics. The following research is conducted in an effort to replace tungsten needles currently used as emitters and explore micro-scale additive manufacturing. This work relies on two-photon photolithography for additive manufacturing green bodies that undergo thermal processing to produce silica glass, silicon carbide (SiC), or silicon oxycarbide (SiOC). GP Silica, a polymer-based resin containing glass nanoparticles, is converted into emitter-shaped green bodies using the Nanoscribe Photonic Professional GT2. Conversion of SiC and SiOC precursor resins into green bodies using the Nanoscribe is also attempted as in previous works [1-3], and the SiOC precursor exhibits success. Thermally processing green bodies is completed in an air furnace to produce glass and a microwave furnace to produce SiC and SiOC. Characterization of resulting structures suggests high potentials for additively manufacturing glass and thermally processing ceramics in the microwave furnace, although further optimizations remain necessary.

Summary of Research:

Additive manufacturing demonstrates increasing promise for device fabrication, making identifying technological limitations of interest. The Nanoscribe Photonic Professional GT2, a micro-additive manufacturing technology utilizing two-photon photolithography, fires a 780 nm femtosecond laser that is re-emitted at 390 nm after striking a molecule within the resin it is printing with. This provides sufficient energy to cure the resin. This paper explores the Nanoscribe's ability to micro-additively manufacture green bodies for thermal processing to make silica glass, silicon carbide (SiC), and silicon oxycarbide (SiOC).

GP Silica is a polymer-based resin containing glass nanoparticles. Developing emitter green bodies with this resin requires optimizing print parameters using the 10X large feature objective. This was completed by printing a 4 x 4 array of 150 μm cubes on a silicon substrate. Scan speed varied along one axis and laser power along the other. 60% laser power and 80,000 μm/s scan speed produced the smoothest edges and fewest bubbles.

The array was placed in a Nabertherm air furnace for thermal processing according to NanoGuide's standard curve [4], which peaks at 1300°C with 3 hour holds at 90°C, 150°C, 230°C, and 280°C. Following sintering, the array was imaged under the scanning electron microscope (SEM), revealing that 14/16 cubes survived, 12 of which were smooth without cracks or bubbles (Figure 1).

Due to this success, solid glass emitters with a 1000 μm base, 700 μm height, and 20° cone angle were attempted using the same process (Figure 2). Many emitters survived, although deformed, as signified by an estimated 55% shrinkage instead of the expected 30%. Next, a new batch of solid emitters and emitters with porous exteriors were printed and heated for 20 hours in the air furnace according to NanoGuide's fast curve4, which ramps to 1300°C at 180°C/hr, saving approximately 40 hours compared to the standard curve. These emitters were more successful, as demonstrated by an estimated 24% shrinkage (Figure 3).

To determine whether a similar process is possible for producing SiC and SiOC emitters, SiC and SiOC precursor resins were developed for printing on the Nanoscribe with 63X oil immersion on glass substrates. Starfire SMP-10 and Starfire SPR-688 were homogenized into resins by mixing in 1%/wt 2-Isopropylthioxanthone and 9%/wt 1,6-Hexanediol diacrylate. Attempting to print with the SiC precursor Starfire SMP-10 based resin using a 10,000 μm/s scan

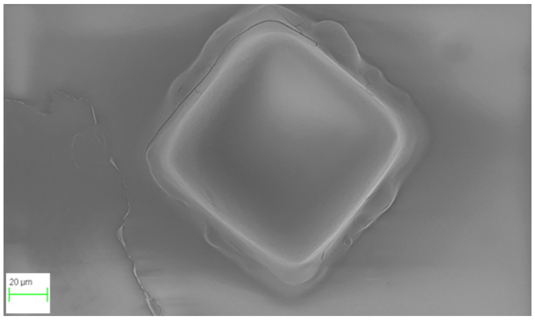
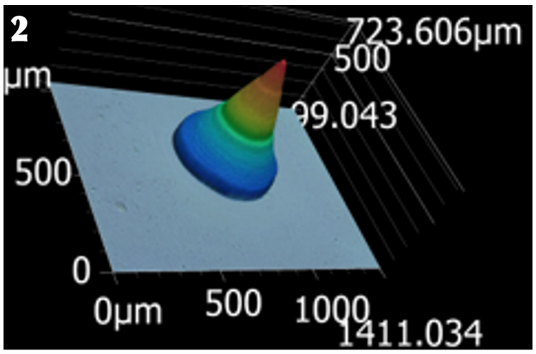


Figure 1: A successful glass cube has a smooth surface without cracks and bubbles.



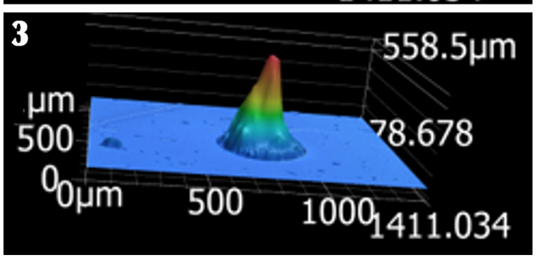


Figure 2: Keyence image of a GP Silica solid emitter green body. Figure 3: Keyence image of a solid glass emitter.

Figure 4: The alumina crucible is lined with SiC and goes inside of a 1 kW microwave.

speed with laser power ranging from 30% to 80%, as well as a 20,000 µm/s scan speed with 70% and 80% laser power produced no parts due to the resin's low viscosity. Significant motion during printing prevented proper curing and substrate adhesion. When printing with the SiOC precursor Starfire SPR-688 based resin, bubbling occurred at laser powers above 50% with a 10,000 µm/s scan speed, suggesting that printing is possible with the SiOC precursor but not the SiC precursor for the formulations and settings tested.

Lastly, the microwave furnace, an alumina crucible lined with SiC within a 1 kW microwave that ramps at 150°C/min, was evaluated for thermal processing ceramics. SiC precursor Starfire SMP-10 cured at 200°C and SiOC precursor Starfire SPR-688 cured at 445°C were placed into the microwave furnace independently for 2, 3, 4, and 5 minutes. Examining 4-minute thermally converted SiC precursor optically and using the SEM revealed consistency with SiC and an amorphous silicate phase. Similarly, 3-minute thermally converted SiOC precursor appeared consistent with SiOC after optical examination.

Conclusions and Future Steps:

In conclusion, GP Silica is useful for micro-additively manufacturing glass emitters with the Nanoscribe. The fast heating curve [4] proves effective as demonstrated by the emitters' 24% shrinkage. Additionally, printing was achieved with the SiOC precursor resin, but not the SiC precursor resin.

Finally, the microwave furnace may present an option for sintering ceramics, but further research is required. In the future, optimizing thermal processing glass emitters and developing emitters using the SiOC precursor resin is necessary, along with testing this process with a more viscous SiC precursor resin.

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