Fabrication of High Quality Ceramic Parts Using Mold SDM

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Abstract

Mold Shape Deposition Manufacturing (Mold SDM) is being developed in order to fabricate complex structural ceramic parts such as components for miniature turbine engines. For this application, the ceramic parts must not only be strong, but they also must have high dimensional accuracy and superior surface quality. This paper presents current progress with process development and characterizes fabricated silicon nitride (Si_3N_4) parts including their surface quality, part density, isotropic shrinkage and build rate. The microstructure of the sintered parts has been characterized. Mechanical testing gave flexural strength values ranging from 400 to 800 MPa. The isotropic linear shrinkage of 18±0.5% and best RMS surface roughness of 0.45 μm was observed. A number of process improvements that lead to better quality parts will also be described.

Introduction

Micro-turbine engines (Figure 1) are attractive because their power density is expected to increase with decreasing size. Power density is proportional to the 'thrust to weight' ratio, a key performance parameter of any thrust producing engine. However, as engine size decreases efficiency may decrease as well. This is caused by additional friction losses stemming from an enlarged surface to volume ratio at smaller scales. To retain engine efficiency at acceptable levels operational temperatures can be elevated. However, the latter is limited by the high temperature properties of engine materials such as nickel based alloys. To further increase engine efficiency ceramic materials have been considered.

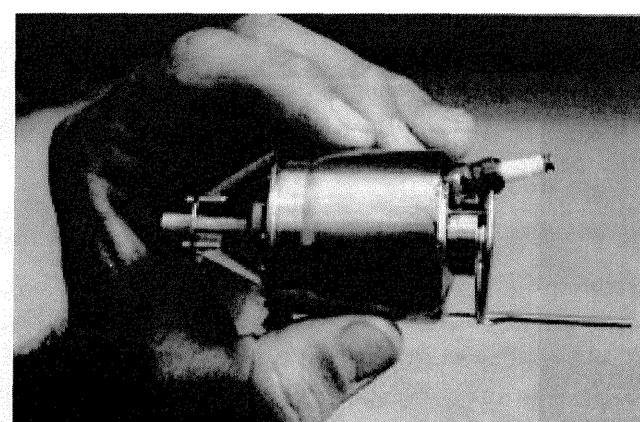
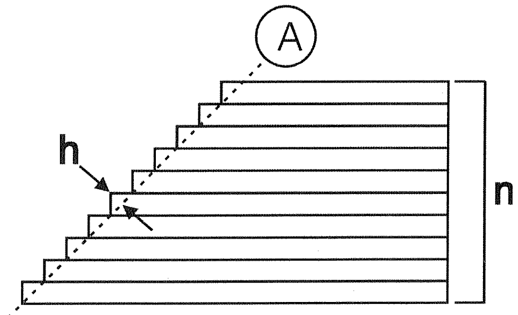


Figure 1. M-DOT micro-turbine engine

From the range of available engineering ceramic materials silicon nitride (Si_3N_4) is an excellent candidate for the small turbine engine application because of the lower density in comparison to super alloys and superior high temperature properties such as high strength, good creep, and chemical resistance. Low CTE and moderate thermal conductivity of Si_3N_4 deliver acceptable thermal shock resistance and enable maintaining adequate temperature gradients for steady state engine operation.

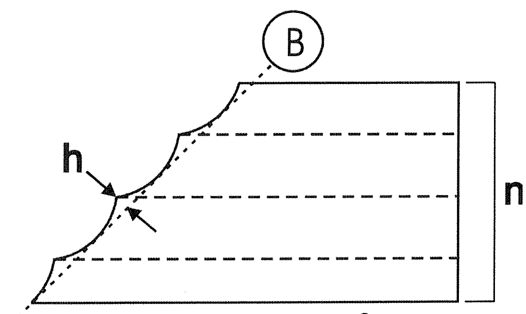
Requirements

Ceramic engine components must have high dimensional accuracy and exhibit high strength at elevated temperatures. The overall part strength is a function of not only inherent material properties, but also of surface finish. Today's Solid Freeform Fabrication (SFF) methods can produce ceramic parts with complex shapes, but limited surface quality. In particular, SFF processes generate 'stair-steps' on inclined surfaces. Stair-steps reduce the benefits of ceramic materials in two ways. First, they make the inclined surfaces rough. Surface discontinuities give rise to stress concentrations and hence may reduce overall part strength. Second, traditional SFF processes are accompanied by a loss of dimensional accuracy. Inclined surfaces formed with stair-steps represent only an approximation of desired geometry, within a certain deviation h (Figure 2). Some stair-steps can be removed by post processing of the ceramic parts such as machining of green or sintered parts. However, this may not always be feasible due to limited tool accessiblity and challenges associated with accurately fixturing and indexing complex part shapes.



$$h = O(n^{-1})$$

Figure 2. Inclined surface formed with layers of constant thickness converges to surface A with the rate of n^1 (n: number of layers)



$$h = O(n^{-2})$$

Figure 3. Inclined surface shaped with ball endmill converges to surface B with the rate of n^2 (n: number of machining path)

An inclined surface shaped by conventional CNC machining using a ball endmill (Figure 3) has advantages in strength and dimensional accuracy over the one formed by layers of constant thickness (Figure 2). In Figure 2 and 3, h is the deviation from the designated surface, which can be considered as measure for surface roughness and dimensional accuracy. Assuming surfaces A and B have the same h (i.e., have similar surface roughness), surface A is formed with sharp concave corners that induce high stress concentrations, while surface B is formed with smoother surfaces that exhibit lower stress concentrations. Surface B is less susceptible to failure at a given stress level and can preserve the inherent strength of the ceramics material. Also, the convergence rate of h to the designated surface is faster for surface B (n^2) than for surface A (n^1). Therefore, CNC machining can improve dimensional accuracy more efficiently than layered manufacturing.

While traditional ceramics fabrication processes, such as machining of green ceramic blanks, can produce parts with good surface quality, they have limitations in achievable shape complexity. Mold SDM is an additive-subtractive process that combines the advantages of layered manufacturing and CNC milling process in order to produce parts of high shape complexity with high surface quality. This paper presents current efforts made in Mold SDM to build quality ceramic parts.

Mold SDM Process

Shape Deposition Manufacturing (SDM) is a layered manufacturing process involving an iterative combination of material addition and material removal. Parts are built up within a sacrificial support material that encases each layer to provide a platform for deposition of the next layer and to support overhanging part geometry features. Unlike most other SFF techniques which decompose models into thin 2-1/2 dimensional layers, SDM retains a three dimensional representation of the parts so that parts are built without stair-steps [1, 2].

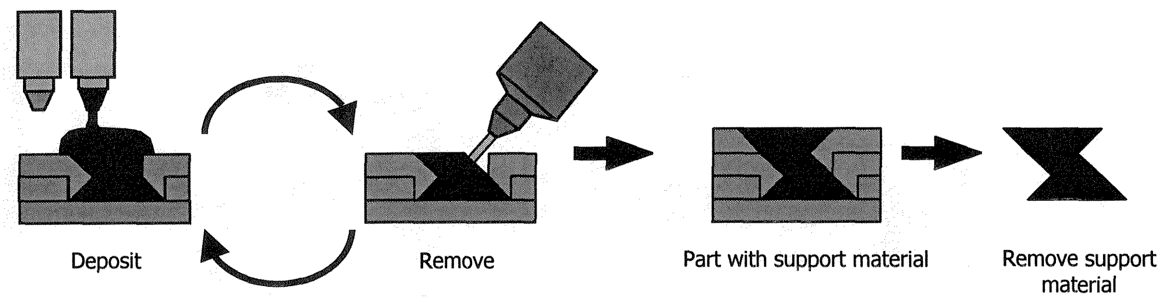


Figure 4. Schematic of SDM process

Mold SDM, illustrated in Figure 5, is a technique in which an intermediate, fugitive mold is fabricated using SDM. The mold is fabricated using SDM with a sacrificial support material occupying the mold cavity. Once the mold is complete, the support material is removed, exposing the mold cavity into which the final part material is cast. The final part material can be any compatible castable material. Green ceramic parts are formed by gelcasting ceramic slurry into the mold and subsequently curing of the slurry. Then, the mold is removed by melting and the remaining green ceramic material is sintered to produce the final ceramic part.

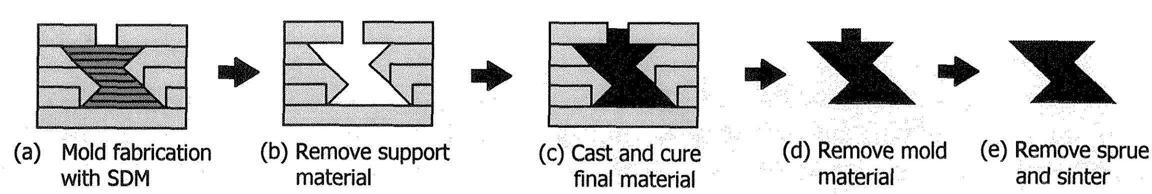


Figure 5. Schematic of Mold SDM process

While this process adds an additional step in the production of green ceramic parts, it does have significant advantages. First, since the mold itself is built using the SDM technique, high geometric complexity can be achieved. Also, the mold design is significantly simplified because the mold is sacrificed, which means no parting surfaces are necessary. Second, the molding approach produces monolithically cast parts, so there will be no layer boundaries or interlayer voids. Third, every surface of the mold is formed by direct machining or by replication of machined surface. In this way, the merits of machined surfaces are transferred to the final ceramic parts. This enables Mold SDM to efficiently achieve dimensional accuracy [3].

Current Process Issues

While Mold SDM has a number of benefits for the fabrication of parts for small turbine engines, there several issues regarding surface quality and part build rate. The choice of the support material is critical for addressing these issues. One support material for Mold SDM is a commercially available, ultraviolet-photopolymerizable, water-soluble soldermask. It is cast as a liquid, then transformed into a machinable solid by curing it with UV light. The water-solubility of cured soldermask provides a convenient way to remove the support material from the mold to expose the cavity for casting the final material.

Soldermask, however, has undesirable properties that lead to lower building rates and that degrade the surface quality of the final parts. The cured soldermask tends to be brittle and prone to cracking, which severely limits the speeds at which it can be machined. This material also has a maximum cure depth of only around 1 mm, so multiple steps of deposition and curing are required for building thick part layers as depicted in Figure 5 (a). Also, the soldermask does not cure evenly. In many cases, there is a thin poorly cured region at the boundaries between layers of soldermask. During the wax deposition step, the incompletely cured regions can deform which results in surface ripples (see Figure 6).

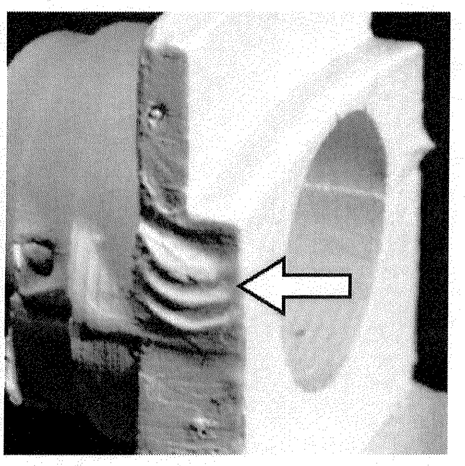


Figure 6. An example of surface ripples.

Water-soluble wax has been investigated as an alternative support material. However, unlike soldermask, a thick layer of water-soluble wax can be deposited by a single casting. Since a thick layer is built-up in one step, the chance of surface ripple is eliminated. Also, the water-soluble wax has better machinability than soldermask. Therefore higher machining feed rates can be used with the water-soluble wax and the build rate can be significantly increased.

The selection of the melting point of the water-soluble wax is critical. If the melting points of the water-soluble wax and the mold wax are very different, then features created in the lower melting point material will be damaged during casting of the higher melting point material. Features in the higher melting point material will not be damaged much, if at all, during casting of the lower melting point material. To minimize the risk of feature damage the best compromise is to use materials with similar melting points. With the materials used to fabricate parts, it was found that casting at 10°C above the melting point produced negligible remelting and feature damage. However, because of the low casting temperatures, the cast material tends to solidify very rapidly when it contacts the surface and does not always have sufficient time in the liquid state to fully and accurately replicate the surface details. Mild preheating of the surfaces prior to casting overcomes this effect. Excessive preheating can cause feature damage either during preheating by melting or during casting if the material has softened too much.

Part Characterization

The slurry used to cast the parts shown in this paper is a proprietary formulation developed by Advanced Ceramics Research (Tucson, AZ) based on Si_3N_4 powders from UBE or Starck. Alumina and yttria serve as sintering aids. The solid loading of the slurry is 50-53%. Before casting the slurry into the wax mold, an initiator is added which helps polymerizing the monomers dissolved in the slurry. The slurry gels in 1 to 2 hours at elevated temperatures.

After melting off the wax mold, the green part is dried and debinded. Depending on the maximum section thickness in the part this process takes 2 - 4 days. Debinding is done at temperatures between 200°C and 600°C in air. In a first stage the binder begins to decompose and in a second stage the remaining carbon residue is burnt out by the oxygen in the debinding atmosphere. Sintering has been done at temperatures between 1650°C and 1800°C.

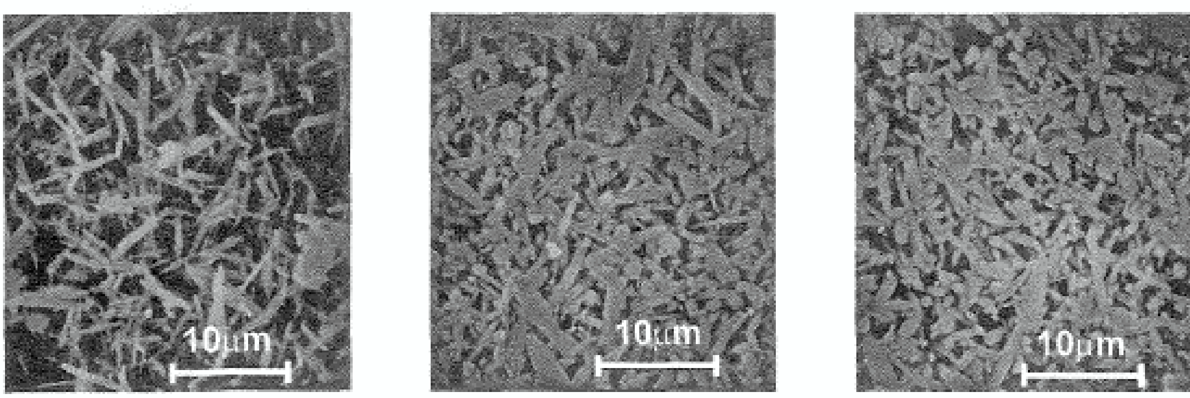


Figure 7. Microstructure of silicon nitride. From left, UBE powder sintered at 1700°C, at 1750°C and Starck powder sintered at 1750°C.

The microstructure of three samples sintered between 1700°C and 1750°C are shown in Figure 7. All samples were etched in molten NaOH at 400°C between 30 seconds and 2 minutes. The microstructure shows the typical needle-shape of the β-Si_3N_4 grains which toughen the material. The density of the samples sintered at 1750°C was 97% of full density.

Two types of samples were tested for their mechanical properties: polished bars gelcast with the Starck powder and unpolished bars made with the UBE powder. An average strength (4-point bending) of 414 MPa with standard deviation of 70 MPa was measured for the as cast bars. The corresponding value for the polished samples was 650 MPa with standard deviation of 200 MPa. The Weibull modulus for the as cast bars was n = 9.6. The maximum strength values were 600 MPa for the as cast beams and 800 MPa for the polished samples.

Table 1. Shrinkage of sintered silicon nitride parts. The small differences implies isotropy of sintered parts

Parts	X-Y plane	Along height	Difference
Center seal	17.7%	18.0%	0.3%
1st inlet nozzle	17.6%	17.7%	0.1%
2nd inlet nozzle	18.2%	17.7%	0.5%

The sintering shrinkage of Mold SDM parts are summarized in Table 1. The results show that the linear shrinkage is about 18±0.5%. The difference between the shrinkage in X-Y plane and Z direction (along the height) is less than 0.5%, which indicates the isotropic nature of sintering shrinkage and density of the final parts.

The surface roughness of the faces of center seal components, which are turbine components described in the next section, were measured using a profilometer over distances of approximately 6 mm. The bottom surfaces, where the geometry was replicated from a machined surface, had root mean square (RMS) roughness of 0.5 - 0.7 μm. The upper surfaces, where the casting features were cut off by manual machining of the green part, had RMS roughness of 1.3 - 1.8 μm. These values compare favorably with values of 4 μm reported for ceramic parts produced by stereolithography [4].

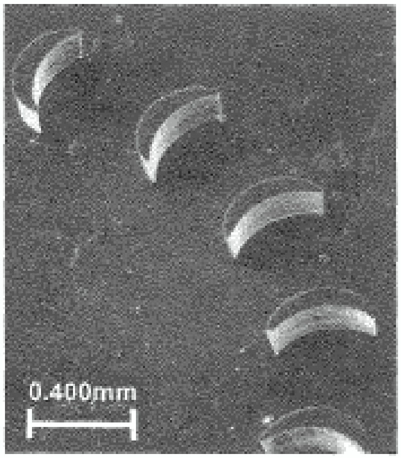


Figure 8: Gelcasting can replicate feature sizes of 100 μm and less as can be seen in this Si_3N_4 green part of a small turbine.

One of the advantages of using gelcasting for the fabrication of ceramic parts is the small feature size that can be achieved with this technique. As an example the green part of a small turbine is shown in Figure 8. The mold for this turbine has been manufactured with micromachining techniques [5]. The blade thickness is about 100 μm. The mold has been replicated very well, and even the gray-shades originating from the triangulation of the blade curvature can be seen.

Example Silicon Nitride Parts

Center Seal

Figure 9 shows a pair of center seals for a miniature turbine engine design. Each piece is a semi-circular shape that has a tongue on one end and slot on the other end such that two of them can fit together to form a complete center seal. The seal prevents gas leakage between the hot turbine side of the engine and the cold compressor side. They also work as thermal barrier between the two sections. It's 26 mm in diameter and 1.8 mm in thickness. The slot and tongue are about 0.5 mm thick. They fit together with 40 μm clearance. The RMS roughness of bottom surfaces is 0.5 μm. It took 6 hours to build the mold and 4 hours to dissolve out the water-soluble wax support material.

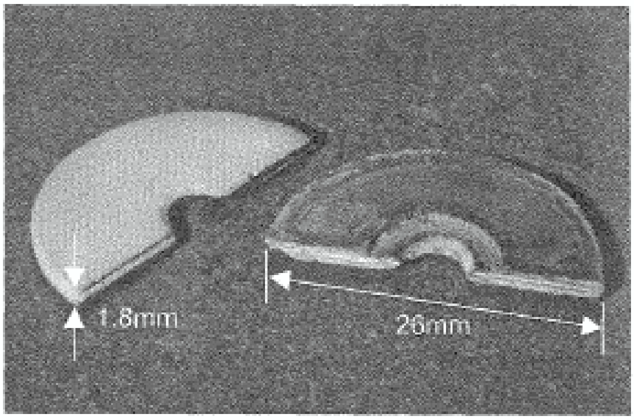


Figure 9. A pair of center seals will form a complete center seal. Left one shows bottom, right one shows top surface. The bottom surface gives 0.5 μm RMS roughness.

Inlet Nozzle

The turbine inlet nozzles shown in Figure 10 and 11 are two design versions for a non-rotating part that directs the hot gasses from the combustion chamber into the turbine. The first design, in Figure 10, has sharp corner features, while every edge of the second design, in Figure 11, is round with a radius of about 0.3 mm. The parts are about 35 mm in diameter. The height is around 9 mm for the 1st version and 16 mm for the 2nd version. It experiences the highest static temperatures in the engine. Estimates indicate that a ceramic inlet nozzle could increase engine performance by 7%, in terms of thrust/weight ratio, compared with a metal nozzle. The first design of the inlet nozzle was tested on a jet engine test rig and survived under the flow of 1250°C, high pressure gas expected in the turbine engine application. This shows the capability of mold SDM to build functional ceramic parts.

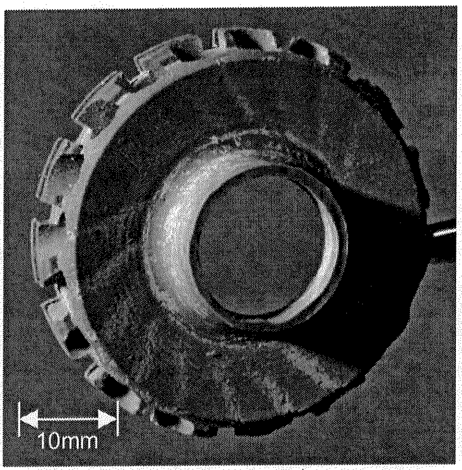


Figure 10: Top view of 1st version inlet nozzle. It was tested and survived under flow of 1250°C gas.

Conclusion

Mold SDM is a suitable process for building small ceramic engine parts. Mold SDM combines the accuracy of CNC machining with the shape complexity which is frequently associated with pure additive SFF processing techniques. Wax molds were used to fabricate green silicon nitride parts with high geometric complexity. The sintered components showed β-Si_3N_4 grain structure which is characteristic of high performance Si_3N_4 materials. The maximum flexural strength ranges between 600 MPa for unpolished samples and 800 MPa for polished samples. The isotropic linear shrinkage is 18±0.5% and the best achievable RMS surface roughness was determined to be better than 0.5 μm.

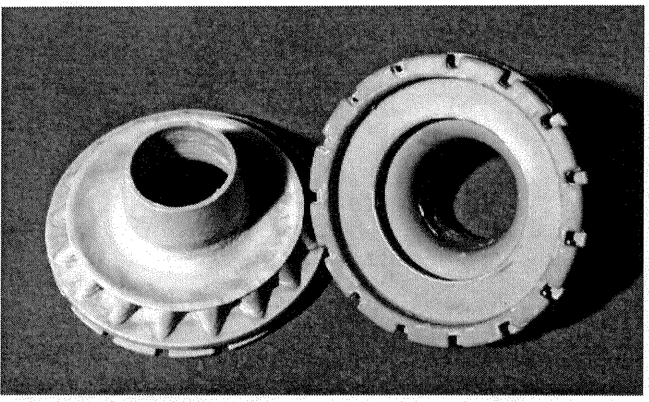


Figure 11: 2nd version inlet nozzles. The surface roughness is 0.45 μm (RMS) for bottom and 1.75 μm (RMS) for top surface.

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