FAST MANUFACTURE OF SILICON NITRIDE FOR AEOROSPACE, DEFENCE, AND AUTOMOTIVE INDUSTRIES

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ABSTRACT

Rapid prototyping combined with microwave sintering can provide a fast manufacturing route, from virtual to finished part in less than one day. This combination can also be used for rapid qualification of materials and design development. Silicon nitride compositions were fabricated by rapid prototype using a layering method. The challenge was to develop a thermal package and microwave sintering parameters for achieving high density test specimens with an inexpensive 1.3 kilowatt microwave. This work is part of a larger project to qualify a material for an aerospace application. Preliminary results are presented on the microwave densification of rapid prototype silicon nitride.

INTRODUCTION

Rapid Prototyping

Rapid prototyping (RP) technologies have proven to be valuable tools in the design and engineering of new products. Current RP technologies can rapidly prepare models, form-and-fit prototypes and masters for molds directly from computer-generated models, sometimes in a matter of hours[1]. The materials used in RP have slowly expanded to include materials, such as ceramics, to fabricate parts that can be used in functional applications.

Javelin has demonstrated success in this area with their CerLAM™ process for monolithic ceramics[2]. (CerLAM™ is a Javelin 3D, Salt Lake City, UT trademark). Javelin's CerLAM™ process is based on a general laminated object manufacturing process. Laminated object manufacturing is one of several rapid prototyping techniques that have been developed recently. While most of the RP techniques have been developed to prepare plastic, wax, or resin parts, the LOM™ system is unique in that it builds models by layering and laminating paper. Paper LOM parts are primarily used in form-and-fit functions or as visual models. While RP processes differ somewhat, they all produce a physical part directly from a computer-generated solid model, without the use of any hard tooling or dies. These techniques allow great flexibility in the design of the part without incurring large tooling costs. This flexibility translates into time-and-cost savings, which can be a significant advantage by reducing the time-to-market.

The initial steps in each of the different rapid prototyping techniques are similar. First, the part to be manufactured is designed using an advanced CAD program. A three-dimensional solid model is created from the CAD. The model is converted into a de facto file format called an .STL file. The .STL file is sliced into thin cross sections by a separate computer program. These slices are used by the RP equipment to build the part(s), layer-by-layer. While there are a variety of systems that build paper, plastic, resin and wax parts, the CerLAM system has been designed specifically for monolithic, composite and laminar ceramics.

High-density ceramic models, containing complex geometry, have been produced by Javelin using the CerLAM process. In this process, a green flexible ceramic tape is used to create the part instead of the LOM paper. The feedstock material, consisting of tape cast sheets is interfaced with the LOM build platform by Javelin's automated SteamRoller™ (SR) system. To realize a fully-automated process required the design and fabrication by Javelin of a single-sheet feed system, called the SteamRoller, for use with a conventional laminated object manufacturing LOM machine. The feedstock is processed by the LOM in the same fashion as the more traditional paper material. The green LOM ceramic parts undergo decubing (removal of build support material). The parts can then be debound and sintered to dense complex monoliths for use in specific applications.

Javelin's SR/LOM process for ceramics has offered the possibility of producing ceramic engineering models in a matter of days as opposed to months required by more conventional methods. Javelin has been able to demonstrate that it is a flexible process suitable for most engineered ceramic materials, in virtually any configuration. Javelin's sintered ceramic parts produced on the LOM system have a similar appearance and strength to parts formed using metal or ceramic injection molding techniques.

Microwave Sintering

A logical step for the rapid prototyping of ceramic parts, is a rapid firing method, using microwaves. Microwave heating occurs by direct coupling of microwave energy with the ceramic, causing volumetric heating.[3] This avoids thermal stresses and allows rapid heating rates. The firing cycle can typically be decreased by a factor of 10 or more, providing significant time and energy savings. Using 2.45 GHz magnetrons, an inexpensive microwave source, many ceramics require a secondary source of heat before they have high enough dielectric losses to couple directly in the field. For this reason, susceptors are often used to start the firing process. As the temperature of the load increases initially by radiation, the load begins to couple, and then absorbs microwave energy preferentially over the susceptors.

Conventional controlled atmosphere furnaces are fairly expensive as well as slow compared to microwaves. An inexpensive, versatile microwave furnace, the ThermWAVE was developed by Research Microwave Systems (RMS). The RMS ThermWAVE has been used successfully to sinter oxide ceramics from several rapid prototyping methods, including epoxy casting and selective gelation printing. In this work, the RMS ThermWAVE was incorporated into the CerLAM process to sinter silicon nitride in inert atmosphere. The complete system from design through fabrication and firing, is especially valuable for new composition development and monitoring changes in the fabrication parameters. One goal is to have a feedback loop so fast that process and materials improvements can be made on a daily basis.

Silicon nitride

Javelin is currently involved with projects sponsored by NASA and the Air Force to demonstrate the feasibility of rapidly fabricating silicon nitride components with Javelin's CerLAM method.[4] Silicon nitride is an engineering ceramic material that has a variety of practical uses in many high-stress and high-temperature applications. It is one of the most important of the bulk monolithic materials for high-temperature structural applications. High temperature strength retention in an oxidizing atmosphere and resistance to thermal shock make it a good material choice for applications such as turbopumps. These properties and the fact that it is a fairly light-weight material, has generated interest for space-based components applications, as well as in the defense and automotive industries.

PROCEDURES

The LOM equipment is a commercial product, and paper is normally used to rapidly fabricate wood-like parts, primarily used in the automotive industry. During the normal LOM build process, a paper sheet that is coated with a heat-sensitive adhesive is loaded onto an indexing build platform. The LOM's laser, controlled by the machine's software that interprets an STL file into very fine slices, first cuts the part's cross-section into the paper layer. A new layer of material is then placed on top of the previous layer. A heated roller is passed over the two layers to laminate the new layer to the previously cut layer. The computer-directed laser then cuts the next layer based on the next computer slice and the process is repeated until the part is finished. The excess material around the part area remains with the part to support it as it is built. The laser cuts tiles into the support material to facilitate the removal of the part at the end of the build cycle. After the prototype has been built, the support material is removed.

The SteamRoller offers an important materials options for the overall LOM process. Since the SR system bypasses the roller material feed mechanism that is provided as standard equipment by Cubic Technologies, it allows the LOM process to use a much wider range of build materials. The SR system was designed and built by Javelin to allow advanced metal or ceramic feed materials to be employed by the LOM machine. To operate in the sheet materials mode, the SR/LOM is first pre-loaded with a stack of sheets fabricated by Javelin. The system is then configured for the part-specific geometry. The ceramic parts build in a manner virtually identical to the standard paper build with increased speed and quality control for each layer. After de-cubing, the green (unfired) ceramic parts undergo tape-cast binder removal and sintering in a conventional or microwave furnace. A microwave sintering procedure was developed using the RMS ThermWAVE, discussed in the following section.

During these studies, silicon nitride polymer blend tapes of final sintered composition: 92 % wt Si_3N_4, 6 % wt Y_2O_3, and 2% wt Al_2O_3 were cast into 0.010" thick feedstock for the SteamRoller. Various geometries were laser cut out of this feedstock using 40% laser power and 0.9 inches per second cutting speed settings for the LOM system.

DISCUSSION OF RESULTS

Rapid Prototyping Geometry

Turbine blades as shown in Figure 1, were fabricated using the CerLAM method to demonstrate the ability to fabricate silicon nitride compositions into complex geometries. This is one of the candidate parts for rapid prototype plus microwave firing.

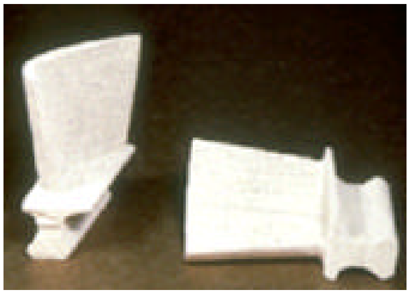

Figure 1. Green Silicon Nitride Turbine Blade Fabricated by the CerLAM Method

Conventional Firing

Green parts were first debound and then sintered to 1820^o C in a controlled atmosphere conventional furnace at 200 psi N_2, with an annealing step. Densification of the silicon nitride was typically 98% of theoretical density. Average flexural strength at 25^o C was 557 MPa based on 4-point bend testing and 585 MPa at 1000^o C.

Microwave Firing

Preliminary work required the development of an appropriate thermal package for firing in the ThermWAVE. A refractory box (Zircar Eco 25B) was fitted with susceptors and alumina crucible as shown in Figure 2. A high temperature thermocouple with a gas introduction tube was developed by Research

Microwave Systems. Flowing argon or nitrogen gas was used to flush oxygen and provide a fairly inert atmosphere. Silicon nitride samples were further protected by submersion in high purity boron nitride powder contained in an alumina crucible.

Various size silicon carbide susceptors (RMS Thermcepts) were used to start heating. It was found that a total susceptors mass or 100-140 grams was sufficient to start fast heating without excessive competition for valuable microwave energy. Since the system has only 1.3 kW of power, it was found that minimizing the refractory volume and energy loss was essential.

a) b)

Figure 2. Thermal packages showing refractory box, alumina crucible filled with silicon nitride sample and boron nitride powder, and susceptors a) RMS Thermcept SiC-50 (curved) and b) RMS Thermcept SiC-70 (disc shape).

Figure 3 shows a typical heating curve for the thermal packages shown in figure 2. It can be seen that the heating rate was over 50 °C/min at the beginning of the run. As the temperature increased the ramp rate decreased because of the limited power. The spikes in heating rate correspond to adjustments of the gas flow. Nitrogen gas flow was decreased slightly during the run.

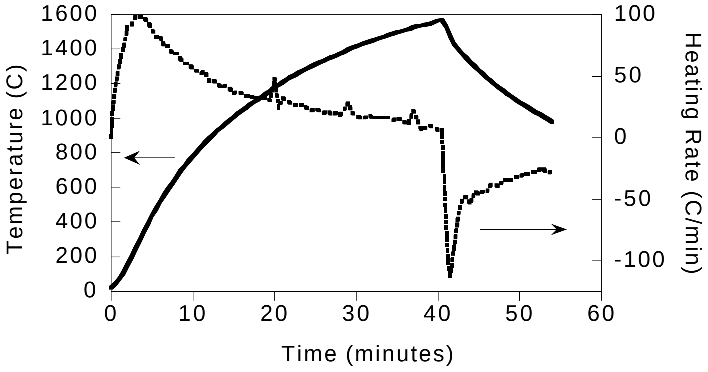

Figure 3. Heating curve in the ThermWAVE for silicon nitride.

The power was turned off at 40 minutes with the gas remaining on for several minutes. When the gas was turned off, a drastic decrease in heating rate occurred indicating that the thermocouple was being cooled by the gas stream. The temperature measurements were therefore lower than actually experienced by the silicon nitride due to the flow of cool gas close to the thermocouple. The design has since been upgraded to eliminate this problem.

A measured temperature of 1600 °C from the ThermWAVE runs similar to shown in Figure 2, resulted in a linear shrinkage of 15% on silicon nitride discs and densities of approximately 95% of theoretical. Flowing nitrogen of 4 l/min was sufficient to prevent oxidation in the silicon nitride samples. A complete sintering run including cool down required less than two hours.

The thermal package was modified to use a pre-sintered silicon nitride powder bed with the same composition as the samples in a porous alumina crucible. Two discs were sintered per run and the results were reproducible from run to run and between discs in the run.

Due to the laminar building up from the rapid prototype method, delamination occurred frequently in conventional firing. Up to 20% of samples were rejected due to warping, cracking, and delamination. In microwave firing, there have been no rejects due to these problems after dozens of runs. It appears that the volumetric heating causes less stress in the laminated structure.

CONCLUSIONS

A low cost process was developed for microwave sintering a rapid prototype silicon nitride composition. Silicon nitride was effectively sintered in a 1.3 kW microwave to 95% of theoretically density in less than one hour. The process was extremely reproducible and is being used for material qualification in an aerospace application. Microstructural and mechanical properties are currently under investigation. Microwave heating profile studies, including high temperature dwells are being conducted in order to increase the density further, and explore grain structure control.

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