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Effects of Electric Field on Selective Laser Sintering of Yttria-Stabilized Zirconia Ceramic Powder

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Abstract

Selective laser sintering (SLS) of ceramic material is particularly challenging. High sintering temperatures and slow sintering kinetics of ceramic material combined with poor thermal shock resistance have resulted in cracking when ceramics are sintered to full density one layer at a time. This work investigates the use of an electric field applied simultaneously with laser scanning to accelerate the kinetics of sintering to produce a multi-layer SLS ceramic part. Ceramic sintering rates have been shown to increase by orders of magnitude during conventional furnace-based flash sintering, in which electric field applied simultaneously with furnace heating. In this work, we investigate the effects of an electric field applied during SLS processing of yttria-stabilized zirconia ceramic.

Additive Manufacturing

Additive manufacturing (AM) is a layer-based manufacturing method that is capable of making a part directly from a computer-aided-design (CAD) file. Additive manufacturing does not require expensive part-specific tooling, which drastically shortens the time between design and the build of a part as well as reducing the cost due to tooling. Most AM methods also can be utilized to build intricate and complex-shaped parts which are difficult or impossible to build with traditional manufacturing methods.

Selective laser sintering (SLS) is a type of AM in which a high-powered laser fuses powdered materials. Typically, a layer of powder is applied to the top of the build chamber. A laser is then focused onto a layer of powder that sits on top of the build chamber with a set of optics. A pair of mirrors rotate so that the reflected laser beam rasters across the top layer of powder in a pattern that melts or sinters only the powder that will become part of the solid formed shape. After the layer is lased, the piston drops the build chamber slightly and another layer of powder is applied to the top surface and this layer is then scanned with the laser. The layer application and selective laser scanning steps are repeated until the entire part is completed.[1]

Complex shapes and detailed parts are achievable using SLS processes. Supports are not necessary for overhanging or sloped surfaces as they are for stereolithography (SLA)[2] and fused deposition modelling AM methods because the powder bed is self-supporting. Low temperature polymers, high temperature polymers, and metals are being commercially produced using a direct SLS process.

Additive Manufacturing of Ceramics

Ceramic additive manufacturing has been primarily focused on indirect sintering methods that utilize a binder or adhesive to fuse ceramic powder particles together and create a solid part. In contrast, direct sintering does not form the shape of a part using adhesives or binders. Instead, direct sintering uses energy source(s) to fuse powder particles directly together when forming the shape of the part. SLA[2], binder jet printing[3], and extrusion-based ceramic AM processes[4] all use polymers to temporarily fuse ceramic powders together into the desired shapes. The polymers that are used for shape formation for all of these ceramic AM processes is subsequently removed by pyrolysis, which can fracture the part unless it is performed very slowly. The time required for crack-free pyrolysis increases exponentially with part thickness, essentially limiting the size of ceramic parts that can be economically produced.[5] After the pyrolysis step, the ceramic parts are densified by a furnace firing.

Attempts at direct SLS sintering processes of ceramics with little or no binder have had limited success.[6-8] These processes aim to provide enough energy to the process to completely sinter together ceramic powder particles one layer at a time. The intent is to fully densify each layer either by melting or sintering the ceramic. The direct sintering approach is particularly challenging because the laser energy densities required to melt or sinter ceramics in the time it takes the laser to scan can be also be high enough to thermally shock the densified ceramic. In addition, shrinkage of approximately 50% by volume is required to densify a layer and such large shrinkages cannot be easily accommodated by the underlying layers of the brittle ceramic material.

Laser Flash Sintering Hypothesis

We propose an alternative approach to AM of ceramic parts in which a laser is used in conjunction with an electric field to induce initial stage sintering of the part to a sufficient degree that necks are produced between the particles such that the part will hold together wherever the laser was scanned over the powder bed. This process is repeated one layer at a time until a bulk part is fabricated using this layer-by-layer approach. Final densification is accomplished in a post-processing firing.

Flash Sintering: Effects of Electric Fields on Sintering of Ceramics in Furnace Environments

Electric-field assisted sintering in conventional furnace-fired ceramic processing has experienced a rapid increase in interest as the result of its ability to dramatically lower sintering temperatures and increase sintering rates compared to conventional sintering. Two types of electric field-assisted sintering experiments have been demonstrated 1) spark plasma sintering where sintering occurs within a die at high pressure[9], and 2) flash sintering where the field is applied to an otherwise bare specimen using electrodes. Although the precise mechanisms that cause the electric field to increase sintering kinetics during flash sintering remains controversial, it has been demonstrated that sintering temperatures can be hundreds of degrees lower than for conventional sintering and sintering times can be reduced from hours to seconds.[10-15]

Laser Flash Sintering Process

Laser flash sintering is an approach to AM of ceramic parts in which a laser is first used in conjunction with an electric field to induce initial stage sintering of the part to a sufficient degree that necks are produced between the particles such that the part will hold together wherever the laser was scanned over the powder bed. The sample is then over coated with fresh powder and the laser is scanned over the surface again. This process is repeated until a bulk part is fabricated using this layer-by-layer approach. In the second step, the partially sintered part is removed from the powder bed, the loose powder is gently removed to expose the partially sintered part, and a post-process, conventional sintering treatment in a furnace or a conventional flash sintering treatment is performed to complete densification.

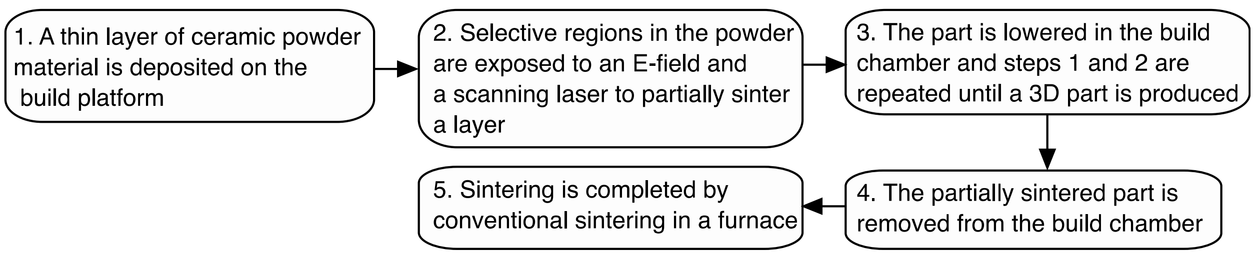

Figure 1: Laser flash sintering process flow

A DC electric field is applied to the powder bed using two electrodes placed on opposite sides of the build chamber. Powder bed layers can be applied as a slurry consisting of powder, a solvent, and a binder. After evaporation of the solvent, the remaining binder is used to temporarily hold the nano-scale ceramic powder in a dense layer, and a dispersant keeps the powder from forming pore-inducing agglomerates. The binder breaks down thermally several hundred degrees lower than the ceramic sintering temperature and pyrolyses during the lasing process. A carbon dioxide laser beam is focused through zinc selenide optics and rastered across the powder bed with a pair of mirrors whose motion is controlled with precision galvanometers.

Laser Flash Sintering Results

The literature on flash sintering reports experimental results performed in a furnace environment; there are no previous reports of inducing flash sintering using a scanning laser heating rather than uniform furnace heating. Experiments with 8% yttria stabilized zirconia ceramic reported here demonstrate that flash sintering effects also occur during laser heating.

Pressed green ceramic pellets of 8% yttria stabilized zirconia (Tosoh TZ-8YS), one inch in diameter and approximately 0.12 inches thick, were lased with and without the application of an electric field. A DC voltage of 315 V was applied across electrodes painted onto the surface of the pellet with colloidal graphite paint. The gaps between electrodes was varied to give field strengths up to 900 V/cm by varying the distance between electrodes. The laser beam was rastered in a serpentine patterned on the powder bed, with the edges of the serpentine touching both electrodes. Beam speed was 1 m/s, with at least three repetitions at various beam powers. The scan was done only on the surface of the pressed green pellet.

The samples lased with electric field showed increased grain growth and more rapid densification as compared to the samples lased in the absence of an electric field. Very bright light was emitted during lasing with an applied electric field, while a dull red glow was the only visible effect on the pellets lased without an electric field. An electric current was also measured during lasing when an electric field was applied. Electrical current is reported in the literature for flash sintering where electrodes are connected directly to the sample. The bright light emissions, the rapid densification (and subsequent over-sintering), and the electrical current demonstrate a flash effect when ceramics that are directly connected to a DC power supply are simultaneously lased. Figure 2 shows differences between the field and no-field conditions.

During LFS Microstructure of Surface

With Electric Field

Without Electric Field

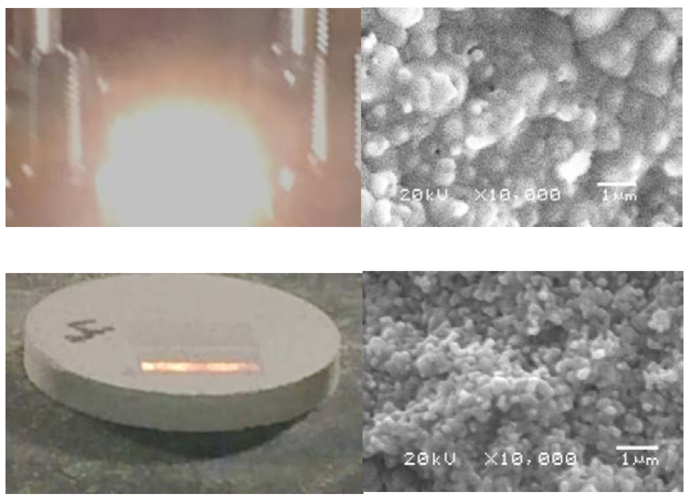

Figure 2: Comparison of SLS of yttria-stabilized zirconia pellets with and without electric field. Visual appearance during scanning is shown on the left, and microstructure of the surface of the pellets in shown on the right.

Future Work and Conclusions

Bright light was observed during lasing of ceramic powder under an electric field that were not visible in the absence of an electric field, confirming that a scanning laser can be used to initiate flash sintering. Future work will investigate the effects of electric field on the processing of ceramic multi-layer parts with SLS process. Application of multiple ceramic powder layers and adhesion of these layers to one another through direct sintering will be investigated. The effects of lasing and electric field parameters on shape formation, microstructure, and defects will be examined. Future work will focus on a better understanding of the effects of electric field on sintering in the highly dynamic ceramic SLS process, and the ability to use these effects to build ceramic parts of better quality and larger sizes than is currently possible.

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