

## Extrusion of Materials for Additive Ceramic Fabrication Using a Polymer-Free Binder for Ceramic Precursors

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### Abstract

The high polymeric concentration of the ceramic feedstock materials has made additive manufacturing of thick ceramic items difficult. However, the binder and additives have remained in the polymeric composition despite numerous studies to enhance the ceramic percentage in the feedstock. The use of a sol-gel-based ceramic slurry without the addition of polymeric additives is described as a revolutionary method for highly dense additive manufacturing [1-2]. The most popular fine ceramic substance, alumina, was used as a stand-in for ceramics. The proposed sol-gel solution met the requirements for material extrusion process, such as extrudable viscosity (100 Pa.s) and self-sustainable yield stress, while enabling a high solid loading of roughly 50% vol% without any polymeric dispersion. During the sintering process, this brand-new sol-gel binder system transforms into aluminium oxide nanoparticles that finally fuse to the alumina particles. The as-printed green body had a higher alumina content and a theoretical density of 66%, compared to the conventional moulding methods' results, which were lower. Additionally, a high density of 99.5% of the theoretical value and a reduced linear shrinkage of less than 16% were attained. This study would outline a workable plan for ceramic additive manufacturing as a developing fabrication technique.

### Introduction

It is anticipated that additive manufacturing (AM), which has received considerable attention over the years in both the academic and industrial sectors, will play a crucial role in the production process and spur innovations across a range of industries [3-7]. The capacity of the AM process to materialise modern ceramics with complex structures, despite their inherent low processability and high processing costs, makes the ceramic industry one of the most attractive sectors for their adoption. Ceramic AM mostly uses two feedstock types: suspension and powder. Binder jetting and selective laser melting/sintering methods for powder-based ceramic additive manufacturing are gaining popularity because they allow for the use of a variety of ceramic powders and the construction of ceramic components with intricate shapes. A light-induced densification mechanism (i.e., vat polymerization process) is used in the case of suspension used as a feedstock for ceramic AM to create a polymeric network for the formation of green bodies. Vat In terms of dimensional accuracy, polymerization techniques for ceramic additive manufacturing show remarkable results, with good resolutions in the micrometre range guaranteeing surface quality. However, the powder-based ceramic AM process's low packing density (50 vol%) results in inherent porosity in the printed object. The density of ceramic objects created using powder-based AM has been improved in a number of ways, including by the combination of different sizes composition of different materials and infiltration of ceramic particles. nanoparticles or precursors. The production of dense, highly pure, technological ceramic structures from the powder-based feedstock is still difficult [8-12]. The phase transition of the polymer to the gaseous species must be carefully managed during vat polymerization, necessitating a slow and challenging debinding method which could take over a week. Due of the high gas pressure inside the structure, fracture and delamination happen if the phase change happens too quickly. Consequently, although polymers and metals use additive manufacturing (AM) techniques for both prototype and production the AM technology for ceramics is still in its infancy, especially for dense, monolithic constructions.

### Subjective Heading

Its high shrinkage ratio is one of the main issues that prevents

ceramic AM from being used for dense, monolithic constructions. Regardless of the kind of ceramic material used, earlier investigations on ceramic components made using the AM process found substantial linear shrinkage after sintering: over 33% with 20% with alumina (Al<sub>2</sub>O<sub>3</sub>) and 29.7% with silicon-based preceramic polymers. The low ceramic content of the raw materials causes these high shrinkage ratios. Ceramic powder suspensions with organic species for polymeric networks are typically employed as feedstock materials in the majority of ceramic additive manufacturing (AM) processes. The suspensions must have a high organic content in order to create a polymeric network for the green body hence needing a low ceramic concentration (less than 50% vol%), which causes severe post-sintering shrinkage. This extreme shrinkage can result in significant deformation, serious flaws like cracks, and even failure of the finished product, which is a major issue in industrial applications.

The unresolved debinding procedure, which removes the polymers and organic components from the green body through heat phase transitions and leaves only the ceramic substance, is another obstacle. When the pressure inside the remaining ceramic structure reaches a critical level, it causes the structure to crack and delaminate. In this process, the organic components of the decomposing gaseous species apply pressure inside the remaining polymer-based ceramic structure [13-15]. This is comparable to how traditional injection moulding is done. In order to limit the design variability of the AM process, similar

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design constraints, such as a maximum volume or wall thickness, should be used as a design requirement for suspension-based AM. As a result, two prerequisites for the creation of acceptable feedstock for dense, monolithic ceramic can be deduced.

## Discussion

To achieve dense and monolithic ceramic printing, the intricate design of ceramic feedstock for AM is crucial. Despite numerous studies to increase the ceramic content, binder systems still only include polymeric components. One of the forming agent candidates for ceramic AM is a ceramic precursor sol-gel solution, which might take the place of a polymeric binding system. A popular technique for creating metal-oxide ceramics, the sol-gel process has benefits like homogeneity, high-purity synthesis, a low reaction temperature, and solution processability. The metal precursors in solution form an inorganic hydrogel network and are transformed into metal-oxide ceramics during the sol-gel process. It was recently reported that by sculpting the green body with an inorganic gel made using the sol-gel method, sol-gel technologies have been directly used to ceramic additive manufacturing. However, because the inorganic precursor gel contains a significant amount of water, the sol-gel-applied ceramic AM had the same restrictions as the traditional ceramic AM, most notably significant shrinkage. For instance, over a 40% green body shrinkage was recorded in the direct ink writing (DIW) method using Ti-containing sol-inks, while a 56% green body shrinkage was reported in the vat polymerization process using a photocurable silica sol-ink.

In order to create optimum feedstocks without polymeric binders for the dense ceramic three-dimensional (3D) printing with a material extrusion (ME) technique, an Al<sub>2</sub>O<sub>3</sub> sol-gel composite slurry (SGC) was carefully constructed based on the analysis of its physicochemical properties. With the benefits of broad material selectivity and great process control variability, ME AM for ceramics has a lot of industrial use. For instance, ME AM was used to print yttrium oxide-stabilized zirconia the 120 mm high zirconia structure and lithium-ion battery components. However, due to the inescapable usage of polymeric components in feedstocks, the adoption of ME AM methods has been constrained. Here, a polymer-free slurry was created to address the drawbacks noted above (low ceramic content and challenging debinding).

SGC is a slurry in which a sol-gel solution of an inorganic ceramic precursor is used to disseminate the fine ceramic powder. In addition to being "debound" out during post-processing, the sol-gel solution in SGC also causes the development of ceramic nanoparticles that are identical to the target ceramic during the calcination and sintering processes. Additionally, the SGC can be produced with a high percentage of ceramic powder (greater than 50% vol%), which is a crucial component for minimising green body shrinkage (less than 16.1%) and producing materials with good mechanical properties (hardness 17.5–0.9 GPa; flexural strength 397.0–16.8 MPa). A schematic of the complete procedure is shown in Scheme 1. Numerous analytical experiments have been performed to verify the properties and processes of Al<sub>2</sub>O<sub>3</sub> SGC. It has been determined how to generate an optimum Al<sub>2</sub>O<sub>3</sub> SGC composition that satisfies the ME 3D printing process' criteria. A specialised layer-wise curing print (LCP) technique based on the ME process was used to 3D print the Al<sub>2</sub>O<sub>3</sub> SGC. To ensure effective printing of the green body and minimise printing flaws such layer segregation and delamination, the deposition and curing conditions were examined. After being sintered at a temperature of over 1500 °C, the printed green body was evaluated for its fundamental properties as 3D-printed Al<sub>2</sub>O<sub>3</sub>.

## Conclusion

On the surfaces of corundum powders, it is thought that alumina nanoparticles emerged from the AlO-gel, lowering the nucleation energy by offering crystallographically similar sites. As a result, using the Al<sub>2</sub>O<sub>3</sub> SGC method, consistent nano/sub-micro bimodal alumina particles were obtained. In order to lower the surface energy, the -alumina nanoparticles from the AlO-gel aggregate and form relatively big particles (100 nm) at 800 °C (Fig. 3(e) and (f)). The phase transition from the phase to the phase is also connected to this rise in particle size. In order to fill the gaps between the corundum powders, these aggregated particles act as conjunction points. This enhances the sintering of the powders and raises the sintered density of the finished product. At 1250 °C, the alumina nanoparticles that had been combined had completely transitioned to -alumina and fused with the corundum powders, causing particle growth and densification.

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## Conflict of Interest

The authors declare that there are no conflicts of interest.

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