Solid Freeform Fabrication 2018: Proceedings of the 29th Annual International Solid Freeform Fabrication Symposium – An Additive Manufacturing Conference
Reviewed Paper

A digitally-driven Hybrid Manufacturing process for the flexible production of engineering ceramic components

J. Hinton[1*], D, Basu, D. F. Flynn[2], R. A. Harris[1], R. W. Kay[1]

1. Future Manufacturing Processes, Institute of Design, Robotics and Optimisation Engineering, University of Leeds, West Yorkshire, LS2 9JT, UK
2. Herriot-Watt University, Edinburgh Campus, Edinburgh EH14 4AS, UK

Abstract

Ceramic materials are a versatile class of materials with numerous applications across a range of industrial sectors. Predominant methods of manufacturing ceramic components use template-driven methods, which hampers responsiveness and impose significant design constraints. This has driven significant interest towards digitally-driven manufacturing approaches, primarily, additive manufacturing. Additive manufacturing has demonstrated the rapid production of bespoke and highly complex geometries and designs direct from digital data without the need for component specific tooling. Yet, when used in isolation these techniques are restricted by uncontrollable porosity, high shrinkages during firing plus a lack of process-compatible materials. This paper presents the research and development of a new hybrid manufacturing process chain for the agile production of engineering grade ceramics components. The combination of high viscosity ceramic paste extrusion, sacrificial support deposition and subtractive micro-machining has yielded complex monolithic ceramic components with feature sizes of 100µm, part densities of ~99.7%, surface roughness down to ~1µm Ra and 3-point bend strength of 218MPa. Since a wide range of materials can be formulated into visco-elastic pastes they can be readily deposited using this approach.

Introduction

Engineering ceramics are broadly defined as inorganic, non-metallic materials [1] and are extensively used across broad and varied spectrum of applications and industries including; healthcare [2], oil and gas [3], power generation, automotive [4] and aerospace [5]. Ceramics exhibit a range of desirable material characteristics including high hardness, excellent thermal and electrical insulation, high resistance to wear, erosion and corrosion [6]. However, these characteristics present a number of challenges during subsequent formative and manufacturing processes.

Conventional methods of manufacturing precision-engineered ceramic components often involve multiple formative and finishing processes that are segregated by a profiled thermal cycle. The hard and brittle nature of ceramic materials results in bulk ceramic being unsuitable for formative and large-scale material removal processes. Therefore, ceramic powder with a narrow particle-size distribution, is dispersed within an organic binder and additional additives to improve processability and component performance. The subsequent matrix exhibits suitable fluidity and characteristics to undergo formative processing such as injection moulding [7], extrusion [8] and machining [9][10]; yielding a near-net-shape green state ceramic part. Subsequent thermal processing decomposes the organic elements and induces sintering of the remaining elements. The thermal processes causes an inherent degree of shrinkage between the green and sintered components, typically 16 -18% [11], [12]. Additional finishing procedures such as grinding, lapping and polishing can be used to improve the geometric tolerances and appearance of the component [13]. However, reliance on component specific templates and tooling place significant limitation on the flexibility and responsiveness of the manufacturing process. Moreover, tooling based production methods require volume production to be economically viable, whilst placing restrictive design constraints on the components that can be manufactured.

Digital-driven manufacturing approaches such as additive manufacturing (AM) is an emerging field of manufacturing approaches that seek to mitigate the short-comings associated with traditional manufacturing approaches. Additive manufacturing produces components directly from digital data in a layer-wise manner, mitigating the need for component specific templates and tooling [14]. Additive manufacturing was originally developed using polymers and waxes but have since been adapted to process materials such as engineering ceramics. Typically, this involves using conventional materials as a binding medium that is subsequently removed during thermal processing.

Vat photopolymerization has received significant research attention owing to the fine features, low surface roughness and densities that can be achieved using this approach [15]. The process involves the homogeneous dispersion of ceramic material within the photocurable monomer that is selectively cured to fabricate the green ceramic part. Thermal processing of the part, decomposes the polymer and sinters the remaining material, yielding a monolithic ceramic component. However, the additional material dispersed within the resin results in an alteration of the rheology and processing characteristics of the monomer. Consequently, the volume of ceramic that can be dispersed is limited, typically <41vol% [16] and shrinkages up to 30% can be experienced during thermal processing [17]–[19]. Moreover, the ceramic material modifies the interaction of the curing radiation and the monomer due to the different absorption characteristics of darker materials, thus restricting the range of materials that can be processed [20].

Powder-based processes have also received significant research attention due to the potential applications for these types of processes including medical tissue scaffolds and process catalysts. Powder-based processes encompass Selective Laser Sintering/Melting (SLS/SLM) and Binder Jetting. Whilst SLS/SLM has shown to be capable of directly processing ceramic material, issues with porosity, thermal shock and component integrity results in indirect processing methods being used predominantly [21]. Indirect fabrication techniques require the powder feedstock particles to be coated with a polymer coating to decrease sintering temperatures and reduce thermally induced stresses. Consequently, irregularities caused by the polymer coating reduces the flowability of the feedstock reducing density and minimum feature size [22], [23]. Alternatively, binder jetting doesn't require modification of the powder feedstock as binder is selectively deposited onto the powder bed. The absence of heat during the fabrication of components mitigates thermally induced stresses whilst retaining flowability of the powder. Despite this, all dry powder-based processes suffer from variation in the particle size distribution, preventing fully dense components from being fabricated and whilst components exhibit a porous and pitted appearance. Alternative, feedstock preparations have been investigated to address the issues with conventional feedstocks [24].

Material extrusion is one most common AM processes owing to its relatively low cost and the numerous configurations available. The process involves the extrusion of material through a nozzle or orifice to form a continuous bead of material [25]. Material feedstocks can include thermoplastic filaments that are highly loaded with ceramic materials [26], [27], ceramic slurries [28] or sol-gels [29]. The high viscosity of the feedstock materials facilitates higher loadings of ceramic material up to 75vol% [30]. Consequently, components experience smaller shrinkages compared to vat photopolymerization processes and components exhibit higher densities compared to powder-based processes. Moreover, the nature of the process offers greater multi-material compatibility compared to the aforementioned processes [31], [32]. Despite this, material extrusion processes are limited by manufacturing tolerances and poor surface finishes.

Despite the relative merits of AM processes capable of processing ceramic materials, the inherent limitations of AM are compounded by the additional processing stages and associated shrinkage, resolution, density, material selection and compatibility. Hybrid manufacturing is an emerging class of digitally-driven manufacturing approaches in which multiple manufacturing approaches are amalgamated into a single, integrated system [33], [34]. Hybrid manufacturing approaches that combine additive and subtractive manufacturing elements have been demonstrated in the production of customised large size ceramic moulds and templates using gypsum-based materials [35]. This paper presents the development of a digitally-driven hybrid manufacturing system for the direct production of components using engineering ceramics. Interleaving additive and subtractive elements into a single integrated system supported by auxiliary elements will facilitate the production of precision engineered components that exhibit low shrinkages and excellent multi-material compatibility. Figure 1 shows the system that has been developed as part of this work.

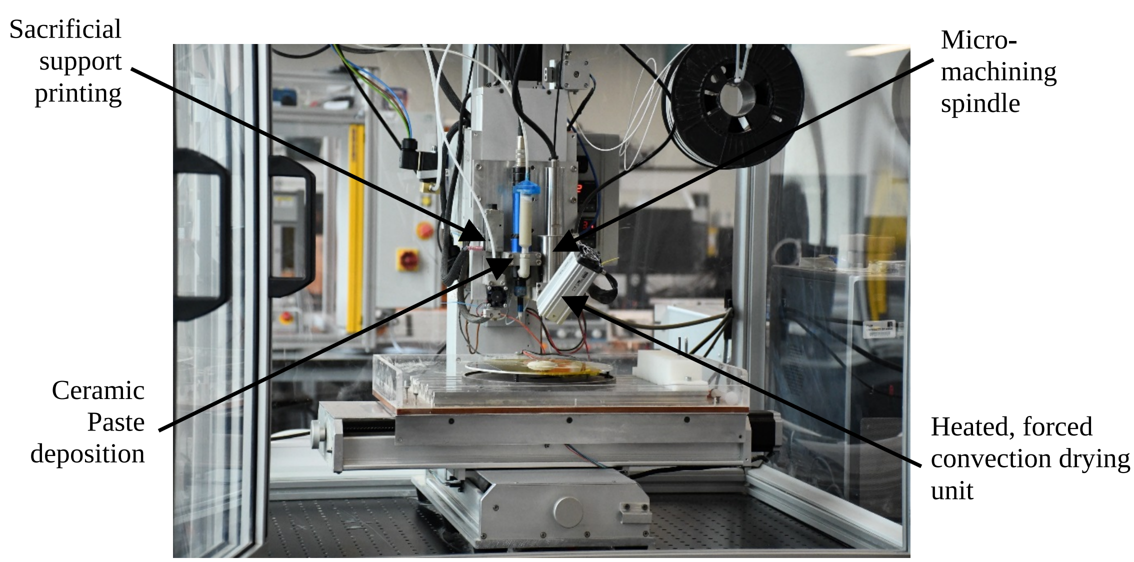


Figure 1 – Bespoke, digitally-driven manufacturing platform using a hybrid manufacturing approach

Method

The bespoke system has been developed for this application by interleaving a number of standalone subsystems into a single integrated manufacturing platform. The process is centred around an auger screw, high viscosity paste extruder (Eco-pen 300, Germany) using interchangeable luer lock nozzles. Subtractive machining is achieved using a precision micro-machining spindle with automatic tool changer (Nakanishi, Japan) used in conjunction with fluted, CVD diamond machining tools. Auxiliary elements include a commercially available hot end (E3D, UK) with a 0.4mm nozzle and Bowden extruder (Bulldog Lite, UK) used to deposit PLA polymer. Additionally, a heated, forced convection drying unit increases throughput of the process. The various elements have been mounted to a 3 axis CNC stage. The stage and the various elements of the process are actuated and controlled using commercially available control software that has been modified for this bespoke application (Newfangled Solutions, USA).

The process was developed using 96wt% alumina with an average primary particle size of 2-12µm that had been formulated into a high viscosity, aqueous paste with a measured moisture content of between 18-22%, using a binding medium developed by Morgan Advanced Materials using a proprietary composition. Characterisation of the paste confirmed the pastes non-Newtonian properties and thixotropic response. This material was selected due to its broad application scope. However, any ceramic material that can be formulated into a high viscosity paste with similar rheological characteristics can be processed. The rheology of the formulated material was investigated using an MCR 102 parallel plate rheometer (Anton Paar, Austria). Sheer values between 50-600s^{-1} were used. Figure 2 shows the results of the rheological tests which confirms the shear thinning rheological response of the precursor material, verifying its suitability for use in an extrusion type process.

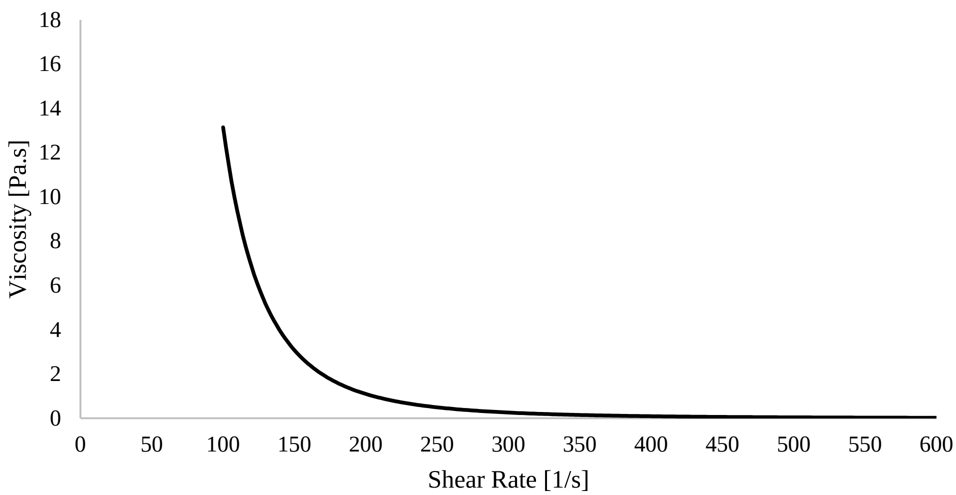


Figure 2 – Graph showing the results of the rheological tests on the paste

Optimisation of the deposition characteristics was undertaken using, 25 gauge (437µm) tapered nozzles with extrusion rates between 80 to 120µL/min and nozzle speeds between 380 and 470mm/min as these were found to produce the most consistent results. These parameters were identified as providing reasonable resolution and feature size with satisfactory production rates. Further optimisation of the processing parameters identified a nozzle speed of 410 mm/min with an extrusion rate of 104µL/min as being optimum for this process. The machining parameters were determined through the machining of components produced during these initial tests. Ø4mm CVD diamond end mill was investigated using side and end milling operations with feedrates between 200-750mm/min with spindle speeds ranging from 6,000-18,000 RPM. It was determined that a feedrate of 330mm/min and spindle speeds of 16,500 RPM achieved the best machining characteristics and lowest surface roughness. Figure 3 shows the process flow for the fabrication of a green state ceramic component.

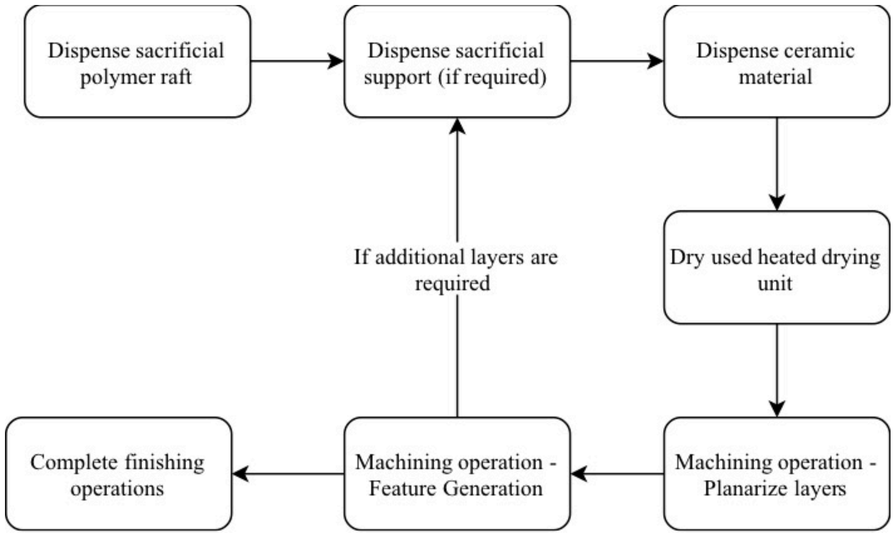


Figure 3 - Process flow demonstrating the production of a green state ceramic component

The fabrication of a green ceramic component involves the selective deposition of ceramic material with a controlled excess of around 2-5%, providing enough material to planarize the top surface and remove defects during post-process machining. A concentric infill pattern is used to prevent excess material within the perimeter regions of the component. Deposition of material is proceeded by accelerated drying using the in-situ heated, forced convection drying unit, drying is undertaken until the component has a sufficiently low moisture content to facilitate machining. This is proceeded by a planarizing, machining operation to ensure flatness and consistency of the deposited layer. If required, this machining stage can also be used to improve the tolerance and fidelity of features, prior to the deposition of subsequent layers. Additionally, PLA can be deposited directly onto the machined ceramic which exhibits satisfactory adhesion to provide sacrificial support for overhanging features. During this work, investigation of enclosed sacrificial PLA supports was undertaken using structures of varying densities. Additional post process machining operations can be undertaken using a variety of fluted machining tools to improve tolerances, remove defects and reduce the appearance of stair-stepping. Figure 4 shows the planarizing machining of a conformal geometry using the optimised machining parameters.

Figure 4 - Interlayer machining of a conformal geometry using a Ø4mm CVD diamond end mill

Once fabricated, the components were thermally processed following a standard production thermal cycle in which temperatures exceed 1400°C. Testing of the manufactured substrates was undertaken to determine the density of the manufactured substrates, surface roughness and mechanical bend strength. Density and associated porosity was calculated using the Archimedean approach using a 0.1mg accuracy micro-balance (Sartorius, Germany) [36]. Surface roughness measurements were obtained using 3-dimensional focus-variation measurements (InfiniteFocus, Alicona, Austria). Mechanical bend strength was determined by 3-point bend testing of samples measuring 5mm x 5mm x 50mm. The test samples were produced in a single batch to facilitate the direct comparison of results. The test parameters were derived from the standard tests used by Morgan Advanced Materials (Z005, Zwick/Roell, Germany).

Results and Discussion

During the course of this work a bespoke, digitally-driven hybrid manufacturing platform has been developed for the production of high density ceramic components. A range of geometries have been fabricated including hemispheres, pyramids and conformal geometries. Additionally, components demonstrating internal cavities and voids have also been produced using PLA sacrificial support that was deposited directly onto the ceramic. Figure 5 shows some of the geometries that have been produced as part of this work.

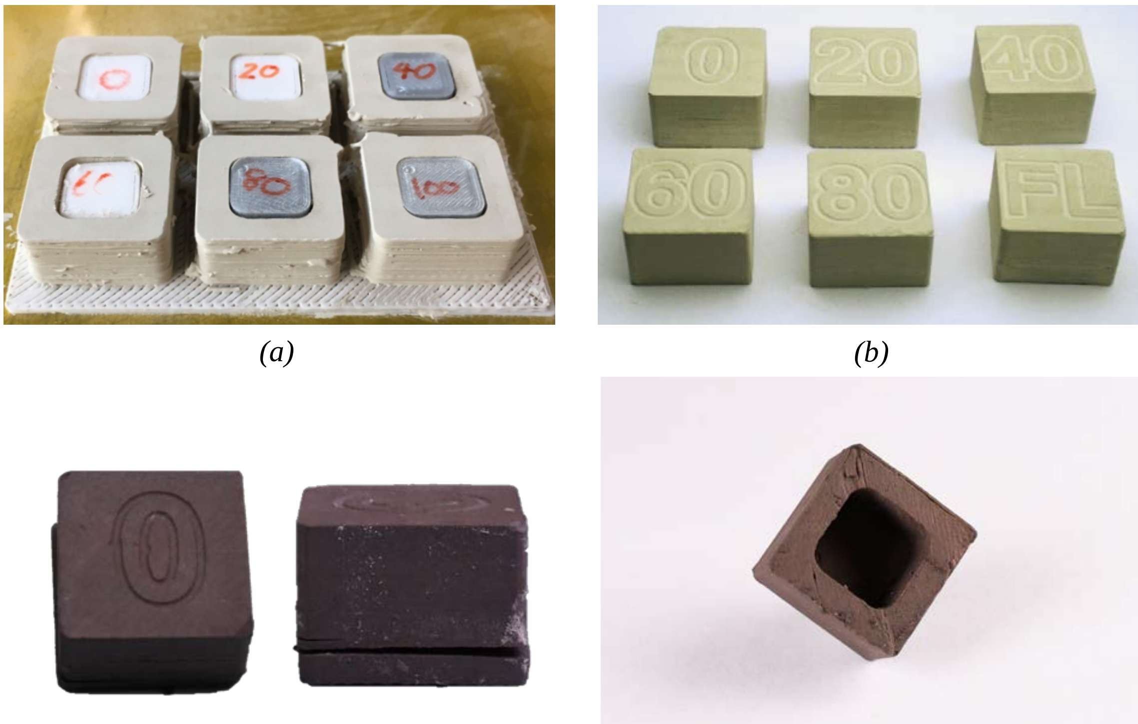
(a)

(b)

(c)

Figure 5 – a: Green state Ø40mm conformal geometry having undergone post process machining operation. b: Conformal geometry post-thermal processing with a measured diameter of Ø33.2mm. c: a sintered pyramid, hollow hemisphere and tile that have been fabricated using the hybrid approach but have not been post processed using machining.

A number of overhanging features and enclosed cavities have been demonstrated as part of this work. Figure 6*a* show the creation of internal cavities through the placement of preformed support structures of varying densities. Figure 6*b* shows the components the components once the support structure has been fully encapsulated within the ceramic components. Post process machining has been used to engrave the corresponding density into the ceramic component using the Ø1mm ball nose cutter. The ability to utilise a multitude of fluted and non-fluted machining tools improves the systems capability to produce high accuracy features. Consequently, finer features, smaller tolerances and greater fidelity with the digital data can be achieved compared to standalone AM systems. Figure 6*c/d* shows the components after undergoing thermal processing and the resultant internal cavity.



(a) *(b)*

(c) *(d)*

Figure 6 - a: Varying infill densities of enclosed PLA support structures measuring 15mm × 15mm × 15mm. b: PLA supports that have been fully encapsulated within the ceramic material and use of post process machining to engrave the corresponding densities of the support structure. c: The sintered samples, measured 12.3mm × 12.3mm × 12.3mm showed no signs of collapse, although delamination was evident on all samples. d: The internal cavity shows no signs of collapse or residue from the encapsulated support material.

Analysis of manufactured components post thermal processing confirmed the production of monolithic ceramic parts that exhibited a measured shrinkage of ~18% between green and sintered parts. Visual inspection of the components revealed no visible residue attributable to the PLA support material. Samples with enclosed cavities containing PLA showed signs of delamination, possibly due to the expulsion of gases generated by the breakdown of the PLA support. Inspection of the cavity however, showed no signs of collapse with high fidelity to the cavity of the green state component. Additional testing of the manufactured components confirmed the production of full-density ceramic parts with part densities of ~99.8%. Surface roughness measurements of post machined parts exhibit a measured R_a of 1.11µm compared to measured R_a value of 8.09µm for non-machined parts. Mechanical bend strength was determined to be 218MPa. Minimum feature sizes were determined to be ±100µm. Figure 7 shows a SEM image of the interlayer region of a component, confirming the production of a monolithic ceramic component. The image confirms the complete sintering and adhesion between adjoining layers.

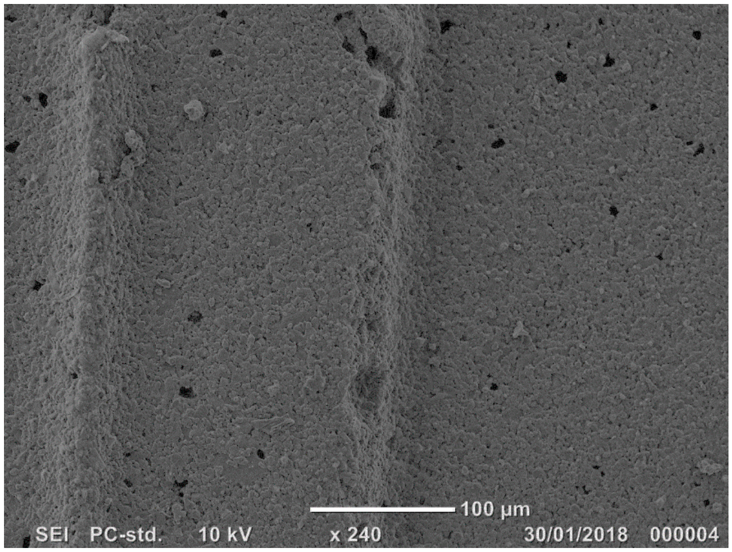


Figure 7 – SEM image of the interlayer region on a sintered part

Conclusion

In conclusion, this paper has presented the development of a digitally-driven, hybrid manufacturing platform for the production of precision engineered ceramic components of arbitrary complexity. The use of sacrificial PLA support structure has facilitated the production of overhanging geometries and enclosed cavities. The fabrication of components has been achieved using production methods comparable to conventional manufacturing approaches to yield monolithic components with densities in excess of 99.8% and 3-point bend strength of 218MPa and measured shrinkages of ~18%. The use of fluted cutting tools and good multi-material compatibility results in a broad and diverse application scope.

Acknowledgements

The author like to acknowledge of Morgan Advanced Materials for providing materials and technical guidance. The author would like to acknowledge the contributions of Chris Hampson, Marianne Sanderson and Heather O'Brian, Morgan Advanced Materials.

References

[1] H. Warlimont, "Ceramics BT - Springer Handbook of Condensed Matter and Materials Data," W. Martienssen and H. Warlimont, Eds. Berlin, Heidelberg : Springer Berlin Heidelberg, 2005, pp. 431–476.

[2] M. Vallet-Regí and F. Balas, "Silica materials for medical applications," *Open Biomed. Eng. J.*, vol. 2, p. 1, 2008.

[3] J. Cui, X. Zhang, H. Liu, S. Liu, and K. L. Yeung, "Preparation and application of zeolite/ceramic microfiltration membranes for treatment of oil contaminated water," *J. Memb. Sci.*, vol. 325, no. 1, pp. 420–426, 2008.

[4] A. Okada, "Automotive and industrial applications of structural ceramics in Japan," *J. Eur. Ceram. Soc.*, vol. 28, no. 5, pp. 1097–1104, 2008.

[5] N. P. Padture, "Advanced structural ceramics in aerospace propulsion," *Nat. Mater.*, vol. 15, no. 8, p. 804, 2016.

[6] J. B. Wachtman, W. R. Cannon, and M. J. Matthewson, *Mechanical*

properties of ceramics. John Wiley & Sons, 2009.
[7] M. J. Edirisinghe and J. R. G. Evans, “fabrication of engineering ceramics by injection moulding. II. Techniques,” *Int. J. high Technol. Ceram.*, vol. 2, no. 4, pp. 249–278, 1986.
[8] M. J. Ribeiro, J. M. Ferreira, and J. A. Labrincha, “Plastic behaviour of different ceramic pastes processed by extrusion,” *Ceram. Int.*, vol. 31, no. 4, pp. 515–519, 2005.
[9] I. P. Tuersley, A. Jawaid, and I. R. Pashby, “Various methods of machining advanced ceramic materials,” *J. Mater. Process. Technol.*, vol. 42, no. 4, pp. 377–390, 1994.
[10] K. P. Belous, “Machining of ceramic materials,” *Chem. Pet. Eng.*, vol. 8, no. 5, pp. 447–449, 1972.
[11] I. O. Ozer, E. Suvaci, B. Karademir, J. M. Missiaen, C. P. Carry, and D. Bouvard, “Anisotropic sintering shrinkage in alumina ceramics containing oriented platelets,” *J. Am. Ceram. Soc.*, vol. 89, no. 6, pp. 1972–1976, 2006.
[12] A. Shui, L. Zeng, and K. Uematsu, “Relationship between sintering shrinkage anisotropy and particle orientation for alumina powder compacts,” *Scr. Mater.*, vol. 55, no. 9, pp. 831–834, 2006.
[13] T. Doi, E. Uhlmann, and I. D. Marinescu, *Handbook of ceramics grinding and polishing*. William Andrew, 2015.
[14] B. Mueller, “Additive manufacturing technologies–Rapid prototyping to direct digital manufacturing,” *Assem. Autom.*, vol. 32, no. 2, 2012.
[15] S. Martin and H. Johannes, “Additive Manufacturing of Dense Alumina Ceramics,” *Int. J. Appl. Ceram. Technol.*, vol. 12, no. 1, pp. 1–7, Sep. 2014.
[16] Y. De Hazan, J. Heinecke, A. Weber, and T. Graule, “High solids loading ceramic colloidal dispersions in UV curable media via comb-polyelectrolyte surfactants,” *J. Colloid Interface Sci.*, vol. 337, no. 1, pp. 66–74, 2009.
[17] F. Doreau, C. Chaput, and T. Chartier, “Stereolithography for manufacturing ceramic parts,” *Adv. Eng. Mater.*, vol. 2, no. 8, pp. 493–496, 2000.
[18] C. Hinczewski, S. Corbel, and T. Chartier, “Stereolithography for the fabrication of ceramic three-dimensional parts,” *Rapid Prototyp. J.*, vol. 4, no. 3, pp. 104–111, 1998.
[19] J. W. Halloran, “Ceramic stereolithography: additive manufacturing for ceramics by photopolymerization,” *Annu. Rev. Mater. Res.*, vol. 46, pp. 19–40, 2016.
[20] S. P. Gentry and J. Halloran, *Depth and width of cured lines in photopolymerizable ceramic suspensions*, vol. 33. 2013.
[21] J. Wilkes, Y. Hagedorn, W. Meiners, and K. Wissenbach, “Additive manufacturing of ZrO_2-Al_2O_3 ceramic components by selective laser melting,” *Rapid Prototyp. J.*, vol. 19, no. 1, pp. 51–57, Jan. 2013.
[22] T. J. Gill and K. K. B. Hon, “Experimental investigation into the selective laser sintering of silicon carbide polyamide composites,” *Proc. Inst. Mech. Eng. Part B J. Eng. Manuf.*, vol. 218, no. 10, pp. 1249–1256, 2004.
[23] K. Shahzad, J. Deckers, Z. Zhang, J.-P. Kruth, and J. Vleugels, “Additive manufacturing of zirconia parts by indirect selective laser sintering,” *J. Eur. Ceram. Soc.*, vol. 34, no. 1, pp. 81–89, 2014.
[24] H.-H. Tang, M.-L. Chiu, and H.-C. Yen, “Slurry-based selective laser sintering of polymer-coated ceramic powders to fabricate high strength alumina parts,” *J. Eur. Ceram. Soc.*, vol. 31, no. 8, pp. 1383–1388, 2011.
[25] D. E. B., T. M., and L. J. A., “Sol-Gel Inks for Direct-Write Assembly of Functional Oxides,” *Adv. Mater.*, vol. 19, no. 21, pp. 3485–3489, Oct. 2007.

[26] S. Ashley, “Fused-deposition structural ceramics,” *Mech. Eng.*, vol. 119, no. 5, p. 32, 1997.
[27] A. Bellini, *Fused deposition of ceramics: a comprehensive experimental, analytical and computational study of material behavior, fabrication process and equipment design*. 2002.
[28] J. Cesarano III, “Robocasting of ceramics and composites using fine particle suspensions,” Sandia National Labs., Albuquerque, NM (US); Sandia National Labs., Livermore, CA (US), 1999.
[29] D. Owen, J. Hickey, A. Cusson, O. I. Ayeni, J. Rhoades, Y. Deng, Y. Zhang, L. Wu, H.-Y. Park, and N. Hawaldar, “3D printing of ceramic components using a customized 3D ceramic printer,” *Prog. Addit. Manuf.*, pp. 1–7, 2018.
[30] W. Gao, Y. Zhang, D. Ramanujan, K. Ramani, Y. Chen, C. B. Williams, C. C. L. Wang, Y. C. Shin, S. Zhang, and P. D. Zavattieri, “The status, challenges, and future of additive manufacturing in engineering,” *Comput. Des.*, vol. 69, pp. 65–89, 2015.
[31] J. Cesarano III, B. H. King, and H. B. Denham, “Recent developments in robocasting of ceramics and multimaterial deposition,” Sandia National Labs., Albuquerque, NM (United States), 1998.
[32] Ma. Jafari, W. Han, F. Mohammadi, A. Safari, S. C. Danforth, and N. Langrana, “A novel system for fused deposition of advanced multiple ceramics,” *Rapid Prototyp. J.*, vol. 6, no. 3, pp. 161–175, 2000.
[33] Z. Zhu, V. Dhokia, S. T. Newman, and A. Nassehi, “Application of a hybrid process for high precision manufacture of difficult to machine prismatic parts,” *Int. J. Adv. Manuf. Technol.*, vol. 74, no. 5–8, pp. 1115–1132, 2014.
[34] T. Yamazaki, “Development of A Hybrid Multi-tasking Machine Tool: Integration of Additive Manufacturing Technology with CNC Machining,” *Procedia CIRP*, vol. 42, pp. 81–86, 2016.
[35] R. Soares, L. C. Moreira, M. Machado, A. J. Cavaleiro, R. Neto, J. P. Pereira, A. Jesus, and A. Reis, “Large-area Ceramic Tooling Production through Hybrid Manufacturing,” 2018, no. March, pp. 3–8.
[36] H. Birol, D. Damjanovic, and N. Setter, “Preparation and characterization of (K0.5Na0.5)NbO3 ceramics,” *J. Eur. Ceram. Soc.*, vol. 26, no. 6, pp. 861–866, 2006.