

A REVIEW OF ADDITIVE MANUFACTURING PROCESSES FOR FABRICATING CERAMICS AND COMPOSITES

Knowing which additive manufacturing techniques are advantageous for specific manufacturing applications leads to better results.

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Manufacturing is an energy and resource intensive process. For example, during the production of ceramic tiles, 3% and 2% of the losses can be due to breakdown of green tiles or finished products, respectively^[1]. The manufacturing uncertainties during tile manufacturing result in financial loss and generate 6.25×10^8 kg of solid waste, which ends up in landfills^[1]. Further downstream, the produced components often require machining, which involves material removal using tools in a lubricated condition (subtractive manufacturing). This machining step can lead to 10%-60% scrap production, generation of ecotoxic lubricants, and consumption of complex machining tools^[2].

Circular economy (CE) envisions a therapeutic process that is built on the following principles: “(a) design out waste and pollution, (b) keep products and materials in use, and (c) regenerate natural systems”^[3]. Additive manufacturing (AM) has emerged as a key technology, which can potentially fit into the CE model as it is possible to incorporate recycled and reclaimed materials during manufacturing^[4]. AM is defined as, “a process of joining

materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies”^[5]. AM terminology is used synonymously with three-dimensional (3D) printing especially with low capacity or price printers and is defined as, “the fabrication of objects through the deposition of a material using a print head, nozzle, or another printer technology” in the ASTM International standard for AM terminology^[5]. AM can also eliminate the need for machining during the manufacturing process. Ingarao et al.^[6] performed a case study on Ti-6Al-4V components in which they showed that AM via electron beam melting is an energy efficient process compared to machining raw materials and were more efficiently manufactured with AM. For realizing the full potential of the AM process, limitations that must be overcome include: (a) need for customized feedstock selection; (b) slow deposition kinetics; (c) product quality, which is partially dependent on surface finishing; (d) high-priced equipment, which is a barrier of entry for entrepreneurs and small business^[6,7]; and (e) anisotropic properties due to layered deposition^[5]. In this

brief review, some of the current stand-out AM technologies that can be used for manufacturing ceramics and ceramic-based composites are presented.

CURRENT STATUS OF AM FOR MANUFACTURING CERAMICS AND THEIR COMPOSITES

Chen et al.^[8] have classified AM technologies according to the morphology of the pre-processed feedstock, for example, slurry, powder, and bulk feedstock^[8]. In this brief review, the classification proposed by Chen et al.^[8] for understanding different types of AM technologies will be utilized and diversified with focus mainly on powder and slurry-based processes (Fig. 1).

AM Based on Slurry Design.

Chaudhury et al.^[9] defined vat photopolymerization as a family of AM manufacturing processes where a vat of photosensitive slurry with controlled viscosity was cured by scanning light of a suitable wavelength^[9]. Stereolithography (SL) is an important member of this family of AM techniques. SL is defined as, a “photopolymerization process used to produce parts from photopolymer materials in

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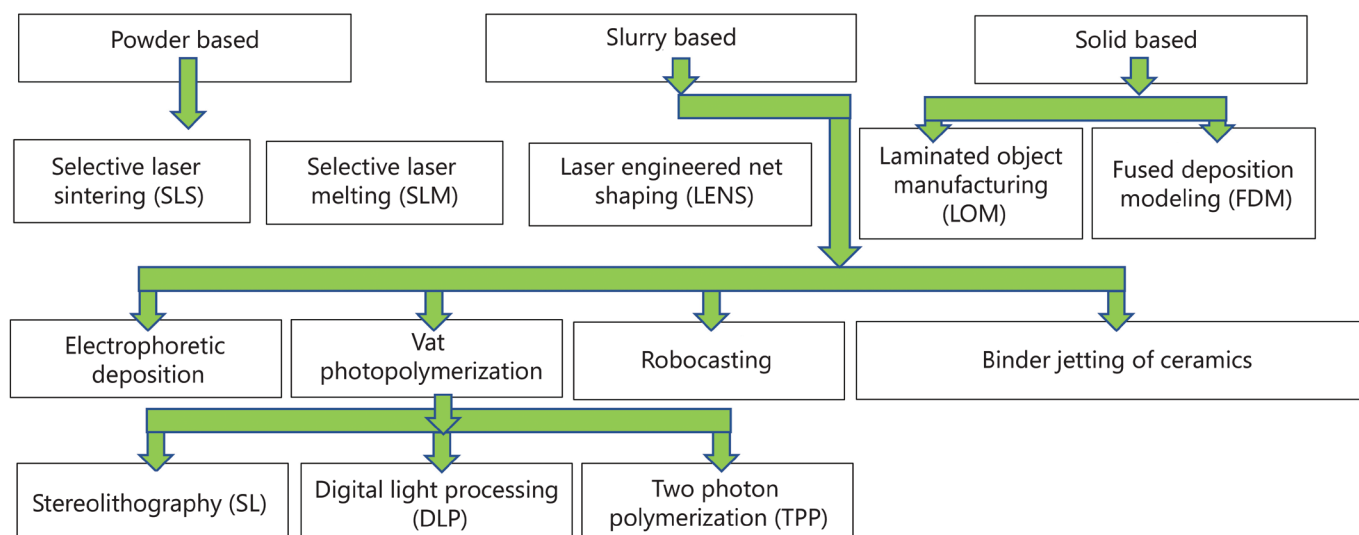


Fig. 1 — Classification of different types of AM processes.

a liquid state using one or more lasers to selectively cure to a predetermined thickness and harden the material into shape layer upon layer”^[5].

The simple schematics of the SL process are shown in Fig. 2a^[8,10]. SL can be visualized as a green body forming process where pre-forms require pyrolysis to form dense bodies. For fabricating ceramic structures, the ceramic powder content of the photopolymerizable slurry should be > 50 vol%^[10]. Some of the critical design challenges of this process are: (a) maintaining adequate solid content and uniformly distributing ceramic particles, with particle size determining the layer thickness, as an unstable slurry can lead to inhomogeneous parts; (b) light scattering by ceramic particles that can affect the curing area; and (c) viscosity engineering of the slurry, which can resist perturbations during the deposition process (typically each layer has a thickness of 100 μm) and can be influenced by a shear stress differential in different regions^[8,10]. Once the process is optimized, SL can be used to fabricate polymer-matrix composites^[11] and ceramics, such as 99.3% dense alumina (Al₂O₃)^[12]. This process can be accomplished using commercial printers,* including the Lithoz CeraFab 7500 (Lithoz GmbH,

Vienna), 3DCeram FCP (3DCeram, Limoge), and CPT6060 (DDM Systems, Atlanta)^[10]. The process can be adopted for manufacturing composites by using tabletop SL printers from Formlabs^[11] and other commercially available printers^[10].

SL can be further fine-tuned by processes, including by digital light processing (DLP) and two photon polymerization (TPP). In DLP, the light source passes through an engineered mask like a digital micromirror device (DMD), which bestows further control over the SL process where the x-y pattern is governed by pixel size, and the z-direction is dependent on the thickness of individual layers^[8,13].

In TPP, an ultrashort pulsed beam is used to initiate simultaneous absorption of two photons in the photosensitive chromophore that initiates the chain reaction polymerization reaction^[14]. This process allows a resolution of 100 nm, which can be adapted for complex designs^[14].

Derby^[15,16] classified inkjet printing (IJP) into three categories: continuous inkjet printing (CIJ); drop-on-demand inkjet printing (DOD); and electrostatic inkjet printing (EIJ). CIJ typically utilizes a drop size of 100 μm , drop generation between 20 kHz and 60 kHz, and drop velocity $> 10 \text{ ms}^{-1}$, whereas DOD uses a

drop size 20 mm–50 μm , drop generation 1 kHz–20 kHz, and a drop velocity that can be controlled depending on use case. Both CIF and DOD have been studied for printing ceramics. In both cases, creating a ceramic suspension with a desired solid content is a critical and necessary step. In the CIJ process, drops are created by Rayleigh instability and consequently placed by charging. The drops have inherent problems such as accuracy of placement, wastage of ink during recirculation, and limited applicability of fluids due to dispersion issues (Fig. 2b). Comparatively, DOD has more control on the drop content and placement by controlling the pressure pulse using piezoelectric or heat sources (Fig. 2c). Piezoelectric actuation, which creates drops by mechanical actuation, is preferred for DOD, as the use of a heat source puts constraints on the volatility of the fluid medium in the inkjet. The efficacy of printing kinetics by DOD is dependent on the droplet size and drying kinetics. For example, drying of isolated drops can lead to inhomogeneous drying (e.g., coffee stain effect). Readers are encouraged to consult references 15 and 16 for detailed information about this process.

Ceramic robocasting is a promising subset of AM processes where tailored

* Specific vendor and manufacturer names are explicitly mentioned only to accurately describe the hardware used in this study. The use of vendor and manufacturer names does not imply an endorsement by the authors or the U.S. Government nor does it imply that the specified equipment is the best available.

ceramic suspension with engineered rheological behavior is extruded via a small orifice. The process is controlled with computer aided design, which helps in forming 3D materials. This process is not a single step process, as the 3D printed product needs an intermediate step to remove binder and other volatiles before the final sintering step. This process is relatively low cost, but some of the challenges include fabrication of a dispersed ceramic slurry with high loading, resulting in the need for special care in selection of organic additives, dispersant, plasticizer, and binder, as well as limitations with hygroscopic materials and stair-step finishing due to layer-by-layer manufacturing^[17].

AM Based on Powder Technology.

Sach et al.^[18] pioneered the binder jetting technique where initially a layer of powder bed is sprayed with binder then subsequent layers are built by repeating this layer-by-layer process. This method needs minimal feedstock preparation as powders alone can be used for this process (Fig. 3a). The five key factors of binder jet printing ceramics are powder selection, binders, printing parameters, equipment, and

post-treatment process. Some of the critical challenges in this process are (a) milling of ceramic powders, (b) low binder strength, (c) and powder layer consistency. In addition, this process requires binder removal and sintering for densification after the printing process where green body strength, binder removal, etc. constitute some of the critical challenges during the post-processing step^[19].

Deckard and Beaman pioneered selective laser sintering (SLS)^[20]. During the SLS process, a laser with the desired power is used to sinter a bed of powders. Thereafter, the process is repeated to sinter the material in a layer-by-layer sequence (Fig. 3b). There has been limited success in directly sintering ceramics using this method due to the long sintering times typically required for densification due to a combination of factors, including solid state sintering process limitations, thermal shock resistance of ceramics, and thermal stresses, which can lead to cracking and high porosity^[8,21]. This process is referred to as direct-SLS (d-SLS)^[21]. A compromise is to bond

ceramic particles with low melting inorganic or polymeric binders, which is also referred to as indirect-SLS (shown in Figs. 3a and d)^[21], to overcome some of the issues with d-SLS.

Meiner et al.^[22] pioneered the selective laser melting (SLM) process in 2001. In this method, the bed of powder is melted layer-by-layer to build the 3D component by using a high-power laser. This promising process can be potentially used to manufacture high performance ceramic components with complicated designs in a single step^[8]. Like d-SLS, SLM also needs to overcome limitations, such as poor thermal shock resistance of ceramics, residual stresses, and thermal gradients, during production. Gahler et al.^[23] utilized a 100- μm beam by using a Rofin Sinar (Hamburg, Germany) SC 10 carbon dioxide (CO_2) laser tube (100 W) and a galvano-scanner (type: hurrySCAN, Scanlab AG, Puchheim, Germany) to print the Al_2O_3 -silica (SiO_2) system. Gahler et al.^[23] used

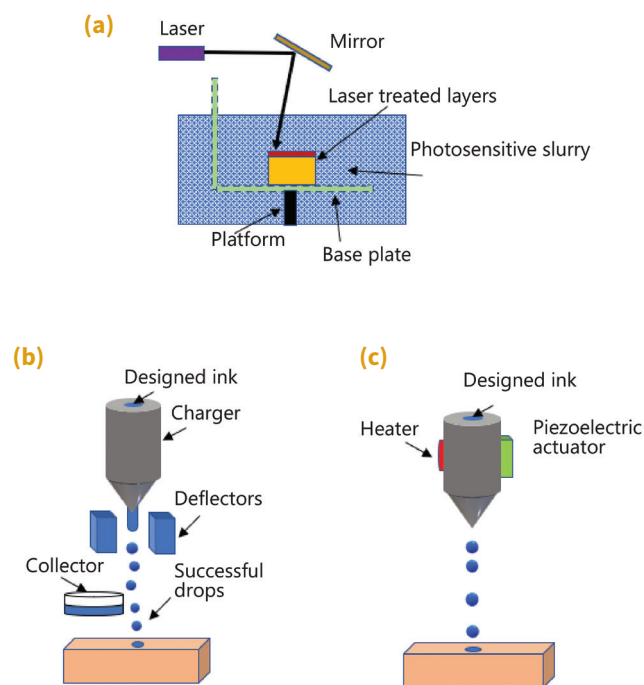


Fig. 2 — (a) Schematics of SL^[8,9], (b) IJP process^[16], and (c) DOD inkjet printer with either thermal (left side) or piezoelectric actuation^[16,17].

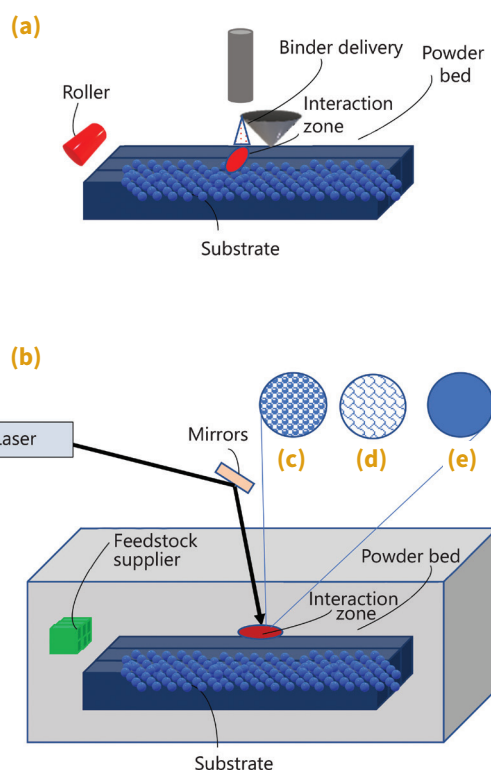


Fig.3 — Schematics of (a) binder jetting of ceramics^[19], (b) powder-based laser assisted manufacturing approach which will result in (c) partially sintered microstructure formed by direct-SLS, (d) ceramics bonded with inorganic or organic binder by indirect-SLS, and (e) melted structure during SL^[8,21,24].

a combination of layer deposition (~100 μm thickness) by using ceramic slurries and a low melting SiO_2 phase that further reacted with Al_2O_3 to fabricate products in the porosity range of 86% to 92%. The samples showed anisotropic (10% axial and 2% lateral shrinkage), and dense regions had an arithmetic average surface roughness, R_a , of ~1.0 μm . Christian et al.^[24] have fabricated 100% dense zirconia (ZrO_2)/ Al_2O_3 samples by preheating the ceramic to at least 1600°C. The authors used a laser with 60 W power, layer thickness of 50 μm , and a scanning velocity of 200 mm/s. Due to rapid melting, this process was hard to control and resulted in uneven crystal growth and coarse surface. Comparatively, laser engineered net shaping (LENS) is a laser beam deposition process where gas feed deposits powder, thus eliminating the need for a powder bed like SLS or SLM^[25]. Application of ultrasonic vibrations during the LENS process has also ameliorated the cracking and inhomogeneity problem observed during laser processing of ceramics^[26].

A CASE STUDY ON SLM OF MoAlB

Experimental Details. SLM of MoAlB was explored as a case study to evaluate the applicability of SLM to a novel ceramic system never before printed using a laser-based AM approach. MoAlB is a ternary ceramic boride, which is a class of lightweight ceramics that possess exceptional thermal conductivity, thermal shock and corrosion resistance, and high toughness and hardness compared to traditional structural ceramics and metals, making it an attractive candidate for a myriad of applications ranging from aerospace to energy. A configurable additive testbed (CAT) was used. The term configurable implies that both the hardware and software can be modified to accommodate specific experimental designs. The CAT consists of an environmental chamber housing a laser powder bed fusion additive manufacturing build platform. The build environment was

kept at < 10 ppm oxygen (O_2), measured using a PureAir trace oxygen analyzer. The laser source was an IPG Model YLR-1000-WC-Y14, with a modulated continuous emission wavelength of 1070 nm and a maximum power of 1 kW. The scan system was a SCANLAB GmbH IntelliSCAN III 20 with a LINOS F-Theta-Ronar lens with a 255 mm focal length.

The layer-wise powder depositions, in the SLM manner^[27], were achieved by lowering the build platen by 50 μm , placing 20 grams of powder atop the build platen, then flattening and spreading the pile of powder using a 22-mm diameter titanium rolling spreader. The process of manufacturing MoAlB powders can be found elsewhere^[28].

Description of Results. The results of a trial run to densify MoAlB particulates using SLM and corresponding energy dispersive spectroscopy (EDS) compositional data for points are shown in scanning electron microscopy (SEM) micrographs collected in backscatter electron (BSE) mode in Fig. 4, highlighting the variety of microstructures and compositions attainable by the SLM process. Some issues that were observed included problems with spreadability and stability of MoAlB particles under laser. Currently, work on optimizing the processing parameters for designing 3D printed ternary metal boride structures is underway.

CONCLUSIONS

There are a variety of types of

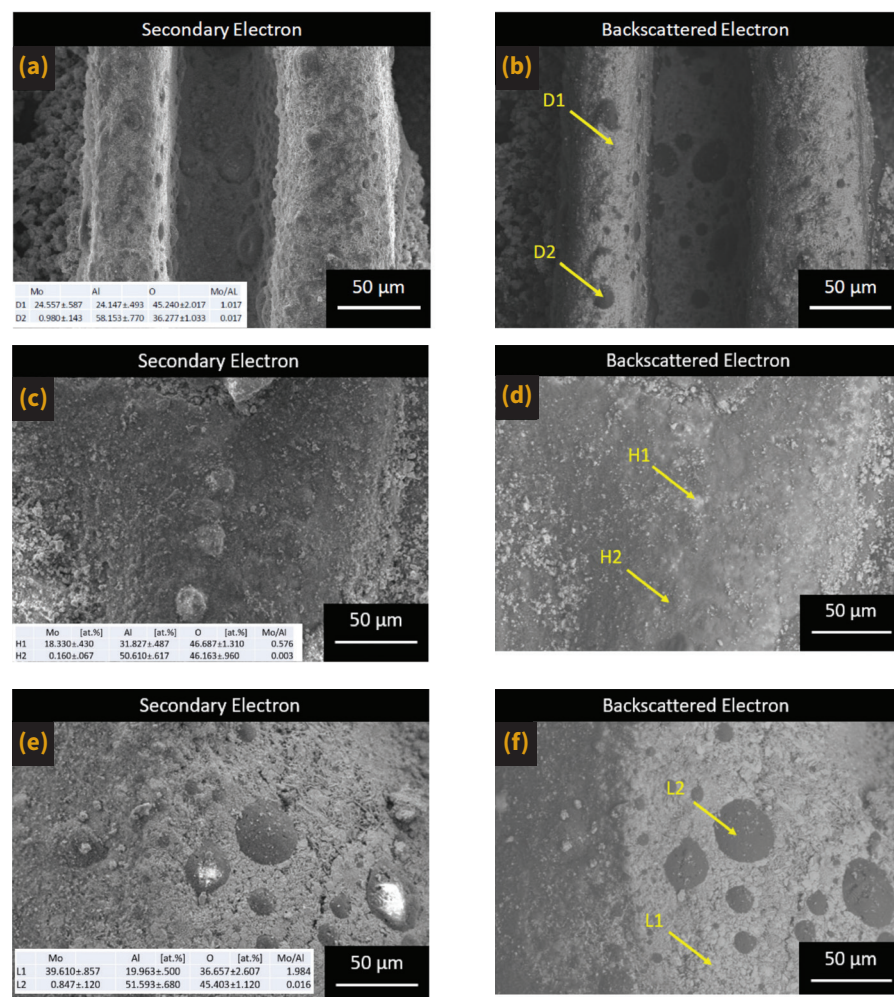


Fig.4 — SEM micrographs of 3D printed MoAlB in (a) SE and (b) BSE at 100 W at 25 mm/s, (c) SE and (d) BSE at 200 W at 75 mm/s, (e) SE and (f) BSE at 400 W at 225 mm/s. The EDS compositional data for points D1 and D2, H1 and H2, and L1 and L2 labeled in (b), (d), and (f), respectively, are shown in tables located on the corresponding secondary electron images^[28].

AM practices that can be used for manufacturing ceramics and ceramic-based composites. SL and different associated techniques offer the possibility of producing ceramic components with finer resolution. Powder-based techniques like binder jetting and indirect SLS can be used for designing porous ceramics. SLM offers practitioners the option of manufacturing ceramics with minimal post-processing requirements, though further research is needed to decrease the cost of materials processing by SLM. ~AM&P

Note: PureAir is a registered trademark of Pure Air Filtration LLC.

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References

1. J.L. Amoros, et al., Green Strength Testing of Pressed Compacts: An Analysis of the Different Methods, *J. Eur. Ceram. Soc.*, 28, p 701-710, 2008.
2. J.B. Dahmus, et al., An Environmental Analysis of Machining, *Proceedings of the ASME International Mechanical Engineering Congress and RD&D Expo*, Nov. 13-19, 2004.
3. What is a Circular Economy, *Ellen MacArthur Foundation*, Feb. 2023, <https://www.ellenmacarthurfoundation.org/circular-economy/concept>.
4. M. Despeisse, et al., Unlocking Value for a Circular Economy through 3D Printing: A Research Agenda, *Technological Forecasting and Social Change*, 115, p 75-84, Feb. 2017.
5. ASTM F2792-12a: Standard Terminology for Additive Manufacturing Technologies, ASTM International, West Conshohocken, PA, 2012.
6. G. Ingarao, et al., A Comparative Assessment of Energy Demand and Life Cycle Costs for Additive- and Subtractive-based Manufacturing Approaches, *J. of Mfg. Proc.*, 56, p 1219-1229, 2020.
7. T. Gutowski, et al., Note on the Rate and Energy Efficiency Limits for Additive Manufacturing, *J. Ind Ecol.*, 21 (S1), p S69-S79, 2017.
8. Z. Chen, et al., 3D Printing of Ceramics: A Review, *J. Eur. Cer. Soc.*, 39, p 661-687, 2019.
9. R.P. Chaudhary, et al., Additive Manufacturing of Polymer-derived Ceramics: Materials, Technologies, Properties and Potential Applications, *Prog. in Mat. Sci.*, 128, p 100969, 2022.
10. J.W. Halloran, Ceramic Stereolithography: Additive Manufacturing for Ceramics by Photopolymerization, *Annu. Rev. Mater. Res.*, 46, p 19-40, 2016.
11. R. Dunnigan, et al., Beneficial Usage of Recycled Polymer Particulates for Designing Novel 3D Printed Composites, *Prog. Addit. Manuf.*, 3, p 33-38, 2018.
12. M. Schwentenwein, et al., Additive Manufacturing of Dense Alumina Ceramics, *Int. J. Appl. Ceram. Technol.*, 12, p 1-7, 2015.
13. M. Lee, et al., Development of a 3D Printer using Scanning Projection Stereolithography, *Sci. Rep.*, 5, p 9875, 2015.
14. K.S. Lee, et al., Advances in 3D Nano/Microfabrication using Two-photon Initiated Polymerization, *Prog. Polym. Sci.*, 33, p 631-681, 2008.
15. B. Derby, Inkjet Printing of Functional and Structural Materials: Fluid Property Requirements, Feature Stability, and Resolution, *Annu. Rev. Mater. Res.*, 40, p 395-414, 2010.
16. B. Derby, Additive Manufacture of Ceramics Components by Inkjet Printing, *Eng.*, 1, p 113-123, 2015.
17. E. Peng, et al., Ceramic Robocasting: Recent Achievements, Potential, and Future Developments, *Adv. Mat.*, 30, 2018.
18. E.M. Sachs, Three-dimensional Printing Techniques, United States Patent No. 5,204,055, 1993.
19. X. Ly, Binder Jetting of Ceramics: Powders, Binders, Printing Parameters, Equipment, and Post-treatment, *Cer. Int.*, 45, p 12609-12624, 2019.
20. J.J. Beaman, et al., Selective Laser Sintering with Assisted Powder Handling, United States Patent No. 4,938,816, 1990.
21. X. Zhang, et al., Additive Manufacturing of Zirconia Ceramics: A State-of-the-Art Review, *J. of Mat. Res. Tech.*, 9, p 9029-9048, 2020.
22. W. Meiners, et al., Selective Laser Sintering at Melting Temperature, United States Patent No. 6,215,093 B1, 2001.
23. A. Gahler, et al., Direct Laser Sintering of Al_2O_3 - SiO_2 Dental Ceramic Components by Layer-wise Slurry Deposition, *J. Amer. Cer. Soc.*, 89, p 3076-3080, 2016.
24. H. Yves-Christian, et al., Net Shaped High Performance Oxide Ceramic Parts by Selective Laser Melting, *Phys. Procedia*, 5, p 587-594, 2010.
25. Y. Li, et al., Additive Manufacturing of Alumina using Laser Engineered Net Shaping: Effects of Deposition Variables, *Cer. Int.*, 43, p 7768-7775, 2017.
26. Y. Hu, et al., Ultrasonic Vibration-assisted Laser Engineering Net Shaping of ZrO_2 - Al_2O_3 Bulk Parts: Effects on Crack Suppression, Microstructure, and Mechanical Properties, *Cer. Int.*, 44, p 2752-2760, 2018.
27. W.J. Sames, et al., The Metallurgy and Processing Science of Metal Additive Manufacturing, *Int. Mat. Rev.*, 61, 5, p 315-60, 2016.
28. A.H. Hocker et al., The Printability of Ternary Metal Boride (MAB) Materials using Laser Powder Bed Fusion, *MS&T'21*, Oct. 17-20, 2021.