PRACTICAL GUIDELINES FOR CERAMIC VAT PHOTOPOLYMERIZATION DEVELOPMENTS

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Abstract. *Ceramic materials are known for their remarkable properties, such as high-temperature resistance, hardness, and chemical inertia. Additive manufacturing (AM) or 3D printing is an emerging technology that fabricates parts by adding material layer-by-layer. Applied to ceramics, AM allows fabricating small series and customized components with greater geometric freedom, lower cost, and reduced delivery time. Among the AM technologies, vat photopolymerization stands out for its ability to produce complex ceramic parts with excellent dimensional accuracy, surface finish, and mechanical properties. However, commercial feedstocks for producing ceramics by vat photopolymerization are scarce. Consequently, the development of ceramic photosensitive suspensions has been in the spotlight in recent years. Although each application is unique, some common factors are essential for successful ceramic manufacturing by vat photopolymerization. However, such important technical details are often omitted in publications or are dispersed in the literature. Thus, this study proposes a general guideline for future developments related to ceramic vat photopolymerization. It considers the selection and concentration of monomers, photoinitiators, and dispersants; the adjustment of the printing parameters; and the definition of debinding protocol. This guideline, resulting from the authors' extensive experience with different materials, is believed to be valuable to other researchers in the area.*

Keywords: *3D printing, additive manufacturing, ceramics, digital light processing, vat photopolymerization*

1. INTRODUCTION

Ceramic materials are known for their remarkable properties such as high-temperature resistance, hardness, and chemical inertia (Jr. Callister and Rethwisch, 2013). Advanced ceramics are special ceramics that exhibit superior properties and are often produced in small quantities at higher prices (Carter and Norton, 2013), having applications in several areas such as solid-oxide fuel cells (Wei et al., 2019), (Xing et al., 2020) automotive sensors (Carter and Norton, 2013), prostheses (Manicone et al., 2007), dental applications (Dehurtevent et al., 2017, 2021), etc. There is a growing demand for customized (or small series) ceramic parts with complex geometries. In this context, additive manufacturing (AM) stands out for being able to produce these parts without the high costs of molds (Schwentenwein and Homa, 2015), (Galante et al., 2019), (Zhang et al., 2020), reducing costs and lead times (Lakhdar et al., 2021).

Additive manufacturing or 3D printing is a set of processes that fabricate parts by adding materials layer by layer. After the great development of additive manufacturing of polymers and metals, developments of this technique applied to ceramic materials have gained prominence in recent years (Chen et al., 2019b), (Shahzad and Lazoglu, 2021).

Although several AM processes can manufacture ceramic parts (Galante et al., 2019), the vat photopolymerization process (VP) stands out for its ability to produce tiny structures with excellent dimensional precision and good surface quality (Lian et al., 2017), (Santoliquido et al., 2019). In this type of AM, a photosensitive liquid in a vat is selectively cured by light-activated polymerization (ISO, 2021). It is a multi-step (ISO, 2021) (or indirect (Deckers et al., 2014)) process, in which additives and binders are used to create a green body that goes through debinding (to eliminate the organics) and sintering (to increase density) (Galante et al., 2019), (Camargo et al., 2020), (Pelz et al., 2021), as shown in Figure 1.
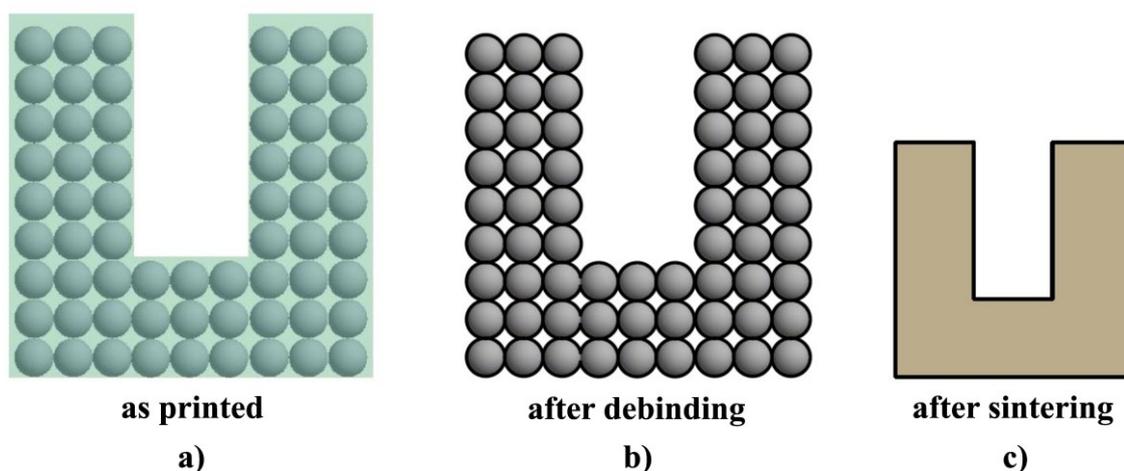
Figure 1. Schematic of the post-processing of a ceramic part fabricated by vat photopolymerization (a multi-step process). a) Green part after 3D printing containing organic binders and ceramic particles. b) Part after debinding (binder removal). c) Final ceramic part after sintering.

The widespread of ceramic AM depends on technological availability (Travitzky et al., 2014) and proper feedstock (Pelz et al., 2021), which accessibility has been an issue. A photosensitive suspension for the VP must fulfill several process requirements (Komissarenko et al., 2018), (Camargo et al., 2021c). For example, a slurry must have solid loading of not less than 40 vol% to avoid cracks and delamination during the post-processing (Griffith and Halloran, 1996), (Jang et al., 2019), (Camargo et al., 2021c); proper rheological behavior and stability to allow uniform and homogeneous micrometric layers to be formed (Schwarzer et al., 2017), (Santoliquido et al., 2019), (Zhang et al., 2019), (Ding et al., 2020) and adequate photosensitive parameters to ensure geometrical accuracy (Komissarenko et al., 2018), (Wei et al., 2019), (Camargo et al., 2021c). Thus, specifications of the ceramic particles, monomers, and dispersants should be considered (Camargo et al., 2021c). Also, a suitable photoinitiator, compatible with the chosen monomers and light source, must be selected (Park et al., 2018), (Gonzalez et al., 2019), as well as refractive index matching of the components to guarantee an adequate cured depth relative to the thickness of the layer, improving overall printability (Griffith and Halloran, 1996), (Wozniak et al., 2011).

Another important step in the manufacture of ceramic parts by DLP is the post-processing of the green printed parts. For example, the thermal debinding must have adequate heating rates to avoid cracks caused by the gases generated during organic burnout (Johansson et al., 2017), (Komissarenko et al., 2018). Thus, it is very challenging process since, in general, the slurries have at least 50 vol% of organic material.

The research and development of ceramic photosensitive suspensions have been in the spotlight in the last few years. Although each application is unique, some common factors are essential for successful ceramic manufacturing. However, some important technical details are often omitted in publications or are dispersed in the literature. The authors have extensive experience (Camargo et al., 2021b, 2021a, 2021c, 2022a), (Erbereli et al., 2022), (Morais et al., 2023) related to developments related to this process using different ceramic materials. Thus, this study proposes a general guideline for future developments related to the vat photopolymerization of ceramic materials.

2. FORMULATION

One of the main challenges in the vat photopolymerization of ceramic materials is the development of ceramic photosensitive suspension. These feedstocks are indispensably composed of monomers or oligomers, photoinitiators, and ceramic powder. A dispersant to help in the stability and viscosity of the suspension is usually also necessary. Also, diluents (Johansson et al., 2017), (Xing et al., 2018), (Lee et al., 2019), (Wang et al., 2019), defoamers (Chen et al., 2019a), (Zeng et al., 2019), plasticizers (Xing et al., 2018), (Gonzalez et al., 2019), and light absorbers (Halloran, 2016), (Pfaffinger et al., 2017), (Hu et al., 2018), (Gonzalez et al., 2019), (Schmidt et al., 2019), (Song et al., 2019) may be used in photocurable suspension. All the components must be carefully selected to create a proper feedstock (Camargo et al., 2021c). There is also an alternative emerging route to produce ceramics using pre-ceramics polymers, which can be converted into ceramics by pyrolysis. Such materials do not require ceramic particles. This type of material is outside the scope of this paper, and the reader is suggested to look after some reviews covering the topic (Camargo et al., 2021c), (Rasaki et al., 2021) if he wants to know more.

2.1 Selecting the monomer(s) and photoinitiator

1,6-Hexanediol diacrylate (HDDA), 1,4-Butanediol diacrylate (BDDA), Poly(ethylene glycol) diacrylate (PEGDA), Trimethylolpropane triacrylate (TMPTA), and ethoxylated pentaerythritol tetraacrylate (EPTTA) are the monomers most used in related developments (Camargo et al., 2021c). Viscosity and functionality must be considered to select a monomer (or a mixture of them). The number of reactive functional groups of a monomer is a major factor in polymerization. Multifunctional monomers promote cross-linking during polymerization, increasing the green 3D printed part strength compared to monofunctional monomers (Griffith and Halloran, 1996), (Goswami et al., 2014), (Komissarenko et al., 2018) and also have less polymerization shrinkage. On the other hand, in general, the greater the number of functional groups, the greater the viscosity of the monomer, which tends to generate more viscous suspensions. A complete analysis of the monomers used in the development of ceramic suspensions for vat photopolymerization was reported in a review article of our research group (Camargo et al., 2021c).

An appropriate photoinitiator must be selected to initiate the photopolymerization reactions, considering mainly its compatibility with the adopted monomers and the relationship between its light absorption spectrum and the wavelength emitted by the light source (Park et al., 2018), (Gonzalez et al., 2019), (Camargo et al., 2021b). Photoinitiators can be divided into two groups: free radical and cationic. Photoinitiators used in research related to ceramic vat photopolymerization are based on free radical chemistry. The most used photoinitiators are Diphenyl(2, 4, 6-trimethylbenzoyl) phosphine oxide (TPO), 1-Hydroxycyclohexyl phenyl ketone (HCPK or Irgacure 184), and phenylbis(2,4,6-trimethyl benzoyl)phosphine oxide (BAPO or Irgacure 819). They all produce radicals from the energy of a light source, being common for applications where acrylate monomers are used. It is important to notice that BAPO and TPO are suitable for the most common VP printers (with wavelength centered at 405 nm), while the longest absorbed wavelength of HCPK is 380 nm (Dai et al., 2015), which requires a different light source.

2.2 Selecting the dispersant

Dispersants are needed to make the ceramic powder compatible with the selected monomer, preventing agglomeration, maintaining suspension stability, and reducing viscosity (Sun et al., 2019). Several dispersants have already been used in ceramic suspensions for vat photopolymerization (see the complete list in our review article (Camargo et al., 2021c)) among which Ammonium polyacrylate, Oleic acid, DisperBYK-111 (BYK-Chemie), KOS110 (Guangzhou Kangoushuang Trade), and Triton X-100 (Sigma-Aldrich) stand out. The correct selection depends on the ceramic material in process and on previously applied surface functionalization techniques.

To select a suitable dispersant, one should pick a few used with similar materials and compare them concerning rheological behavior (especially viscosity at a shear rate of 30 s[-1]) and sedimentation. For that, prepare suspensions with a predefined solid loading (e.g., 40 vol%) and a fixed amount of the tested dispersants (e.g., 3 wt% of the ceramic powder mass).

2.3 Selecting the ratio of the components

Once the monomer(s) and dispersant have been selected, prepare suspensions with a predefined solid loading (e.g., 40 vol%) and different amounts of dispersant (e.g., from 0 to 5 wt% of the ceramic powder mass). Then, compare their sedimentation and rheological behavior (especially viscosity at a shear rate of 30 s[-1]) to determine the dispersant optimum concentration.

Next, prepare suspensions increasing the solid loading using the optimized amount of dispersant and characterize their rheological behavior. In general, the highest possible ceramic loading is desired. However, the viscosity limits of the 3D printer to be used must be considered. If the equipment is not dedicated to ceramics, such limits should be discovered empirically.

3. MANUFACTURING

As it uses a raw material quite different from polymeric resins, usually used in non-dedicated commercial 3D printers, ceramic vat photopolymerization requires some extra care regarding the process specifications and equipment preparation. Also, heating post-processing is needed as described next.

3.1 Setting the process specifications and preparing the 3D printer

The main printing parameters for vat photopolymerization are thickness and the light exposure time per layer. Unless there is a specific reason, the thickness may be the equipment default (e.g., 50 μm). For defining the light exposure parameter, expose the suspension to different light times, rinse the created layers with isopropyl alcohol (IPA), and measure their thickness with the proper device (e.g., micrometer). The light exposure selected must provide a cure slightly thicker than the selected layer thickness (e.g., 1.3 times) to ensure layer integration without impairing the accuracy (Wei et al., 2019). Beer-Lambert law (Griffith and Halloran, 1996), described by Eq. (1), can be used to select the light exposure time required for a pre-defined thickness.

$$C_d = S_d \ln \left(\frac{E}{E_c} \right) \tag{1}$$

Where C_d is the cure depth, S_d is the sensitivity of the suspension, also called penetration depth, E is the incident light energy density, and E_c is the critical energy (the minimum energy density to initiate polymerization for a suspension).

In addition to the usual vat photopolymerization precautions, such as carefully leveling the building platform, using ceramic suspensions as feedstock may require additional steps to prevent printing failures. For example, sanding the building platform (with 180-grit sandpaper) may prevent failures related to part detachment from the platform (Roach et al., 2018). Also, a PTFE-based lubricant may be applied to the bottom of the vat to reduce the periodic detachment force between the part being manufactured and the bottom surface of the vat.

3.2 Defining the debinding protocol and evaluating the geometry of the parts to be manufactured

A piece of a green 3D printed part should be subjected to thermogravimetric analysis to study its thermal decomposition. Temperature ranges with higher thermal decomposition should have reduced heating rates ($\approx$0.1-0.5°C/min) or even holding times in the debinding protocol. The debinding protocol is also geometry-dependent and thicker parts may need lower heating rates. Certainly, more complex thermal treatment methods can be applied, including pressure control and different atmospheres.

Ceramic vat photopolymerization has limitations regarding maximum wall thickness. The major commercial suppliers specify 10 mm as the maximum wall thickness to be produced (ADMATEC, 2023), (Truxova et al., 2020), (Camargo et al., 2022b). Also, bottom-up 3D printers tend to fail when manufacturing parts with large cross-sections, which causes high adhesion of the part to the bottom of the vat (Huang and Jiang, 2005) and may cause the first layer to be stuck in the bottom of the vat or part of the piece to detach from the build platform during the manufacturing, as shown in related work (Camargo et al., 2022c). Also, abrupt changes in the cross-sectional area should be avoided, as they can be a source of defects because the regions with different wall-thickness can retract at different rates during debinding and sintering. Finally, the anisotropic shrinkage during the post-processing must be considered.

3.3 Health and safety considerations

With the expansion of this technology and the search for high performance of the final parts, many materials have been explored. However, some of them, especially monomers and dispersants, may promote allergy, chemical burns, and skin irritations, even using standard personal protective clothing and equipment. Their fumes and volatile content are also unhealthy and require special air purification. Furthermore, many of them also require particular disposal because they are toxic to the environment and cannot be thrown in regular trash containers.

During the debinding, a high volume of organic material is burnt, promoting local air pollution, which might require gas filtration. In addition, it is important to look at future possibilities of water-based resins, visible light photocuring, and solvent-assisted multi-step hybrid removal techniques.

4. CONCLUSIONS

In order to carve out a niche within the growing demand for customized (or small series) ceramic parts with complex geometries, vat photopolymerization has to increase the feedstock availability and increase knowledge about how to deal with their manufacturing difficulties. Although an increasing number of related works have been published, some important technical details are often omitted in publications or are dispersed in the literature. Thus, this study proposes a general guideline for future developments related to ceramic vat photopolymerization, considering formulation (components selection and determination of their concentration) and manufacturing (printing parameters, debinding protocol, etc.). This guideline may be valuable for future developments in the area and highlight a growing need for attention to health and environmental issues related to vat-photopolymerization.

5. ACKNOWLEDGEMENTS

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7. RESPONSIBILITY NOTICE

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