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CHARACTERIZATION OF PRINTING CONDITIONS OF POWDER-REINFORCED RESIN BY LCD TECHNOLOGY

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Abstract: LCD technology has been known for decades and used in the manufacture of components with photosensitive material (6K, 8K, polyurethane-based elastics resin, eco- and bioresins). The functional characteristics and aesthetics of components can be improved by adding reinforcing materials in the form of powders, pigments, or other additives. The paper presents the effects on the printing process with such a modified resin through a systematic approach. The modifications consisted in adding metallic powders with two different grain size (aluminum and copper). The results showed not only the possibility of successful printing but also problems that can occur during the processes. Several tests were carried out on a desktop LCD 3D printer, which makes the proposed approach applicable to any type of equipment.

Key words: LCD printing, photosensitive resin, metallic powder, polymeric powder, resins.

1. INTRODUCTION

LCD technology is part of the Vat-photopolymerisation group which together with [1] Material Extrusion (ME), Binder Jetting (BJ), Material Jetting (MJ), Powder Bed Fusion (PBF), Directed Energy Deposition (DED) and Sheet Lamination (SL) cumulates most of the additive technologies available on the market. LCD technology along with Digital Light Processing (DLP), Stereolithography (SLA), Continuous Liquid Interface Production (CLIP) and Carbon Digital Light Synthesis™ (DLS™) use various systems (e.g., galvanometers, dynamic digital micro-mirror device, LCD which act as a mask [2]) for controlled exposure or directing UV light from various sources (e.g., LCD display, projector, laser) to solidify a liquid monomer (also known as photosensitive resin) in the printing equipment vat (reservoir or tank).

Initially, most common stereolithographic polymeric resins were free radical systems (based on acrylate and methacrylate monomers, polyesters), declining in popularity (due to the potential hazards which can appear during the curing reactions such as unpleasant odor, relatively volatile behavior and reaction inhibition by oxygen) after the emergence of

cationic polymerized mechanism (epoxides and vinyl ethers) [3]. Alongside this standard resins, over time other structural, hard and tough, elastic and flexible, bio-compatible, biodegradable, bioink and ceramic resins have been introduced [4].

Ceramic photopolymer resins were originally developed in 1992 by W.R. Grace and Co. Their ideas were adopted and further developed two years later by Ceramic Composites Inc. [5]. Nowadays, there is a multitude of companies (e.g., Formlabs, Formfutura, Tethon3D, 3Dresyns, Envisiontec, etc.) offering ready-to-use or customized services dedicated to the development and production of custom materials (resins with nano ceramic/metallic/polymeric powders) for multiple UV based additive manufacturing platforms.

UV curable metallic suspension can be also prepared by combining polymers that act as a binder/based material (e.g., unsaturated polyester, epoxy resin, vinyl ether or ester compounds, acrylate based monomers and oligomers), photoinitiators, additives and filler powder. A variety of approaches can be considered for powders, such as [3- 12]: Al₂O₃ with 0.4 μm average particle size (APS) or

10.34 μm APS, nitride powders such as Si_3N_4 E3 with 0.7 μm APS, Si_3N_4 E10 with 0.3 μm APS, AlN with 600 nm APS, 316L stainless steel with particle size $<16 \mu\text{m}$, tungsten with 0.3 μm APS or tungsten carbide with 6/2.5/0.8 μm APS, cobalt with 1.5 μm APS, AlSi10Mg particles coated by styrene with a total APS of 12.96 μm , high-speed steel with size range of 2~45 μm , different types of yttria-stabilized zirconia (3YSZ) with dimensions between 207 nm and 488.4 nm, TiO_2 with 10 nm APS.

Zimbeck et al. [5] stated that in the case of photopolymer-based rapid manufacturing of "green state" metallic and ceramic objects, a high concentration of solid material must be achieved (volume added material must be at least 50 % [3]) while maintaining low viscosity (between 2 and 5 Pa·s [3]) and stable particle suspension, also a good interlayer adhesion, minimal shrinkage during curing, nondestructive binder burnout, high density and desirable properties. Also, [3,5] in the case of materials with high relative density, it is recommended to use smaller particle size powder because of their lower sedimentation rates, diluent and dispersants or coupling agents for steric and electrostatic stabilization (thereby enabling stable suspensions and less particle agglomeration). Zhang et al. [6] stated that ultrafine powder (less than 30 μm) facilitates the formation of agglomeration between particles, leading to poor dispersion in the resin and consequently poor penetration of UV light. To improve the cure behaviors (reduce the high surface energy, refractive index difference between the particles surface and resin), it is recommended [6, 11] a modification of surface characteristics (of the solid powder) by chemical or physical methods.

In conclusion, in the specialized literature, studies present the possibility of utilizing ceramic/metallic slurry (with a high content of solid powders, requiring debinding and sintering) on different equipments (e.g., manual laminate building apparatus with an UV flood/photomask system [5], SLA system [3, 8, 12], DLP systems [6, 10, 11], customized 3D printer based on Fused Deposition Modeling system with an auxiliary device [7]). Unfortunately, these do not present the

capability of using a material obtained by simply combining a photosensitive resin (available on the market) with metallic powders on a dedicated LCD equipment.

Due to the possibility of controlling the entire UV pixel matrix, LCD 3D printing ensures the exposure of the whole layer at once, which enables fast print speeds and adequate details. Previous tests [2], carried out on the same printer as the one used for the current experimental research, have proved the possibility of successfully printing parts starting as from 10 μm Layer Height (Lh), features with a minimum thickness of 0.05 mm (which resemble to the XY pixel size of 47.25 x 47.2 μm) and 6 s Exposure Time (Et).

This paper proposes to demonstrate the feasibility of fabrication with micro-metallic-particle filled resin by addressing several issues, such as achieving solids loading in the resin while maintaining a stable particle suspension, controlled level of the resin viscosity, adequate layers adhesion, homogeneous distribution of inorganic particles in the end-product and, of course, safe operation for both equipment and operator.

2. EXPERIMENTAL WORK

Further research starts with the material preparation and sample shapes determination, followed by a preliminary test to determine (both theoretically and experimentally) the implications of adding metal particles to the resin. Based on the collected information, the samples are then printed and analyzed using a digital microscope.

2.1. Sample preparation

The material used during the experimental study is the 3D Jake Color Mix Water Washable Transparent Photosensitive Resin, from Niceshops GmbH Company (Paldau, Austria) reinforced with different grain sizes metallic fillers. The considered metallic materials (from Laborlanden.de- Blumberg, Germany) were aluminum powder (99.8 % purity) $<45 \mu\text{m}$ (Figure 1. a), Pyro 5413H Super Al powder (99.5 % purity) $<5 \mu\text{m}$ (Figure 1. b) and copper powder (99.6 % purity) $<45 \mu\text{m}$ (Figure 1. c). The concentrations of metal powder considered

were 0.5 % respectively 1 % by volume. The metal powders were added to the liquid resin and the mixture was homogenized for 30 min at 800 rpm, using the overhead stirrer Ika® Nanostar 7.5 digital, from IKA®-Werke GmbH & Co. KG (Staufen, Germany). The mixture was also mechanically/manually stirred to achieve better homogeneity of the final material (to avoid agglomerations formation and powder sticking to the glass vessel walls).

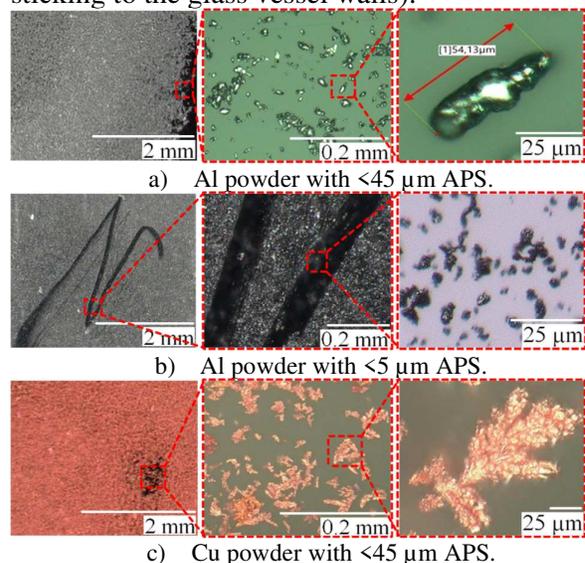


Fig.1. Metallic powder

The preparation process of the material for 3D printing was conducted in a ventilated chamber which also offers protection against UV light from the outside environment. Subsequently analysed samples (from Figure 2) of photosensitive resin with metallic fillers were printed on an Anycubic Photon S LCD 3D printer, from Anycubic Company (Shenzhen, China). The final considered printing conditions are 0.1 mm for Layer Height (Lh) and 25 s Normal Exposure Time (Et).

The process parameters were chosen based on preliminary experiments, the printing conditions of the resin (provided by the manufacturer) and information from literature (for similar applications). Anycubic Photon S and Anycubic Photon Workshop V3.1.3 slicing software were used, because it is an affordable combinations (desktop 3D printer and free dedicated CAM software) that meets all the requirements for experimental tests (possibility of using a wide range of materials, user-friendly and low-cost maintenance, dimensional accuracy, safety

operation and low vat volume, etc.). It is also possible to precisely adjust the movements of the printing platform and the LCD (e.g., control the height and speed of lifting and lowering the bed, off-time between layers, exposure time of the first layers to ensure a proper adhesion of the first layers of the plate and Et).

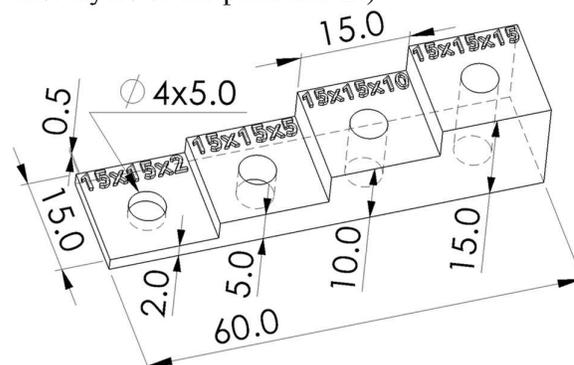


Fig.2. Shape and dimensions of the sample

After printing, the post-processing of the samples is essential for cleaning excess material (uncured resin) from the inside and outside of the part, achieving optimal optical and mechanical properties of the material, making the part safe to touch and achieving safety standards set by regulatory agencies (for biocompatible materials). During post-processing, safety and protection rules specific to the technology must be followed, such as wearing nitrile or neoprene gloves, safety glasses, protective clothing and covering of the work surfaces. All of this must be performed in a well-ventilated area and the part must be protected from UV light until the post-curing stage. In this paper, Wash & Cure Station (from Anycubic) was used including washing the samples in isopropanol 99.9 % (from Höfer Chemie® GmbH) for 6 min followed by post-curing for 6 min with the included 40 W and 365 nm/405 nm UV Lamp.

Before analysis, the samples were conditioned for 24 hours at 23 °C and 50 % humidity. These conditions also correspond to the environmental conditions during the preparation of the material for printing, during 3D printing and post-processing. Temporary storage for transporting the resin from the mixing station to the 3D printer was done using containers that do not allow UV light to pass through. To avoid any possible build-up of

metallic materials, after the mixing process was completed, the resin was poured directly into the printer's vat and the printing process started.

To determine the homogeneity and particle distribution within the printed samples, different cross sections (perpendicular to the upper surfaces) were studied. The surfaces considered were analysed using the digital microscope Keyence VHX 700, from Keyence Deutschland GmbH (Neu-Isenburg, Germany).

2.2. Preliminary tests and comments

In the case of lithographic processes, photoinitiators act as absorption sites for the incident photons (from the UV light source) which supplies the energy needed to induce a chemical photopolymerization/ photocuring reaction (free radicals, cationic or hybrid). This will further form a highly cross-linked polymer, in the form of a solid set of elementary volumes called voxels (the 3D equivalent of the 2D pixel, presented in Figure 3). This implies that when solid particles are introduced, they will be embedded in the final product without any molecular connection between them and the resin. Most solid particles are opaque, which makes the penetration of light (inside the tank) dependent on the space between the particles, their reflectivity and in some situations, refractivity.

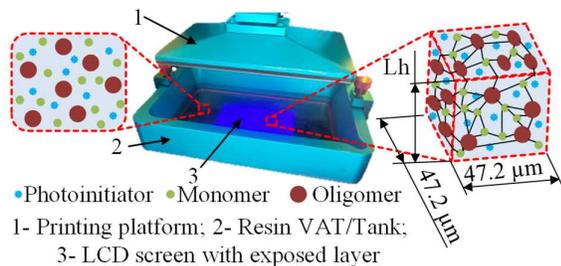


Fig.3. Transformation of the liquid resin (left side) into solid voxels (right side) as a result of exposure to UV light, on the Anycubic Photon S printer (middle)

The initial tests involved producing a mixture (80% photosensitive resin and 20% metallic particles, by weight, of Al with 45 μm APS and Cu with 45 μm APS). Due to the characteristics of the mixture (relatively large particle size, difference in density and viscosity of the resin compared to that of metallic materials) it was found that the particles did not remain in suspension and that after approximately one

minute the slow settling began. After five minutes a solid layer begins to form on the bottom of the container. The actual properties and behavior of the mixture make the use of LCD equipment difficult and therefore using a spreader during the printing process is recommended. Therefore, a significantly reduced amount (0.5 % respectively 1 % by volume) of metal particles was used during future experimental investigations.

As an alternative, such a solution (with a content equal or higher than 20% solid particles) can be used as a coating material for a thermoforming shell type mould made of plastics manufactured by FFF technology. This coating/shell provides an additional layer of protection against the heat resulting from the contact of the heated foil with the mould. Also, due to the high content of copper or aluminum (which have a high coefficient of thermal conductivity), a faster heat removal from the working zone can be obtained, especially if cooling channels are introduced inside the mould [12]. The particle size of approximately 45 μm makes the printing process with <0.05 mm layer heights (Lh) impossible and can also lead to complete or partial blocking/covering of the light source since the pixel size is 47.2 μm and to the alteration of the light beam path within the resin (Figure 4).

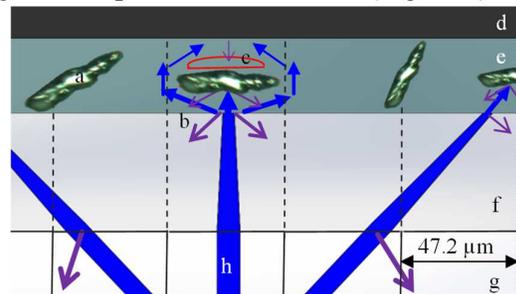


Fig.4. Problems occurring during the LCD printing process [13]

Where: a- solid particles in suspension, in different positions (grain sizes of approx. 45 μm), b- reflections of the light ray upon contact with the FEP foil (f- considered as not being 100% transparent and without damages), liquid resin (e- with the height equal to Lh), and solid particles, c- risk area, d- printing platform, g- LCD screen pixels with dimensional (47,2 μm) projection through FEP film and resin, h- decrease in intensity of the light beam due to absorption as it travels through the FEP film and resin.

In the case of DLP printers the effect of inhomogeneous brightness distribution occurs due to the given brightness distribution of the projector, reflection and absorption by the vat floor and natural vignetting at the vat floor [13]. Similar effects occur with LCD technology if each UV LED is considered as an individual projector. In addition to these phenomena, deterioration (e.g., matting/opacification, scratches, wrinkles, micro-cracks) of the FEP film (Fluorinated Ethylene Propylene film) and refractive index difference between the solid powder (in some cases) and the photosensitive resin must also be taken into account. These can lead to problems such as delamination of the layers, impossibility of sticking the first layer on the printing platform, incomplete curing of the resin or the appearance of air bubbles, deformation of the shape and reduced surface quality of the printed part.

2.3. Experimental study and results

In the experimental research a series of tests were performed to determine the printability of a resin with metallic fillers (see Table 1). The T1 virgin resin sample (with 0 % filler) is a reference sample and is used to determine the influence of the metallic powder on the printing parameters, the powder distribution inside the part and other defects/problems specific to the approach in question.

Table 1.

Test runs	Materials used during experimental tests		
	Infill percentage by vol. [%]	Powder material	Grains dimension [µm]
T1	0	-	-
T2	1	Al	5
T3	1	Al	5
T4	0.5	Al	5
T5	1	Al	45
T6	0.5	Al	45
T7	1	Cu	45
T8	0.5	Cu	45

The first T2 samples (Figure 5 b) showed major deformations in partial detachment of the piece from the printing platform (especially for the thin 15x15x2 mm section), delaminations of the layers, cracks and fissures. To prevent this (Figure 5 c), in contrast to the T1 and T2 sample (Figure 5 a), Et was increased by 212.5% (from

8 s for T1 to 25 s) for all the further tests and the bottom Et from 60 s to 75 s (for T3, T4, T5 and T6). The parts with copper filler materials (T7-Figure 5 d. and T8) were successfully printed without any problems. Because of the higher density of Cu, an increase of the buildplate movement speed from 3 mm/s to 4 mm/s (to induce a stirring effect of the resin inside the 3D printer tank) was also necessarily. The changes in the printing parameters lead to an increase in the production time by up to 120.27% for Al samples (1 h 32 min 30 s) and 95.25% for Cu samples (1 h 21 min 33 s), compared to the printing time of the T1 sample (41 min 46 s).

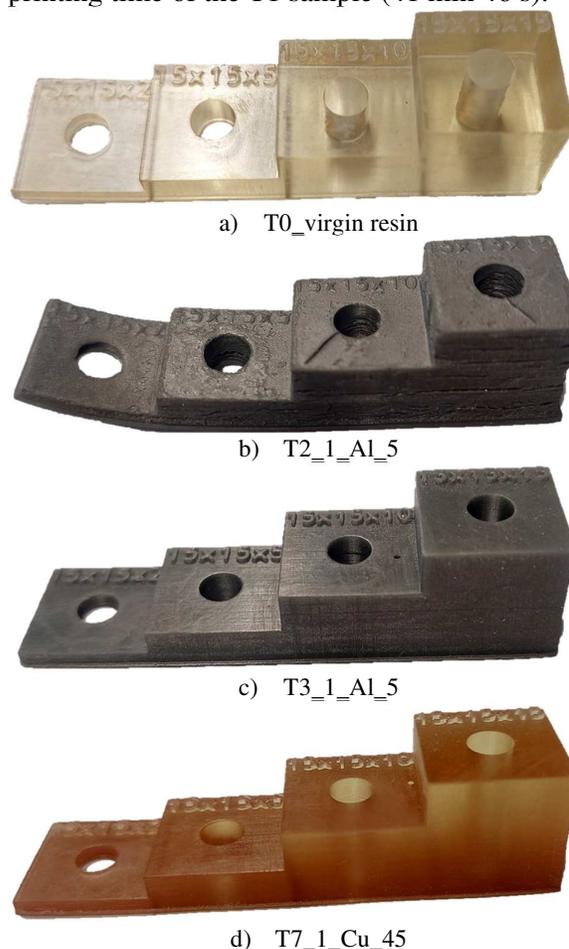


Fig.5. Studied 3D printed samples.*

*Sample code T2_1_Cu_45 meaning Sample/Run number_Infill percentage_ Powder material_Grains dimension

In Figure 5 d, an uneven particle distribution is visible. Images from Figure 6 a (obtained by using a digital microscope) were taken from inside the sample at certain heights for a more detailed analysis. In the case of several samples

(T5, T6, T7 and T8) a gradual decrease in the concentration of metal particles is observed up to heights of about 7 mm followed by a major reduction in the concentration of filler material for heights greater than 7 mm. This indicates an almost complete sedimentation of the particles up to a certain distance and a decrease in the influence of the mixing effect due to the movement of the printing platform.

This phenomenon of resin stirring during the printing process is also supported by the distribution of particles on the bottom of the 3D printer's vat (Figure 6 b and c) and mainly affected by the type of filler materials (density and APS). The particles will move and remain in suspension for a longer period of time as a result of turbulence within the tank and subsequently settle in a more uniform manner (section 15x15x2 and 15x15x5 in Figure 6 b, compared to similar regions in Figure 6 c).

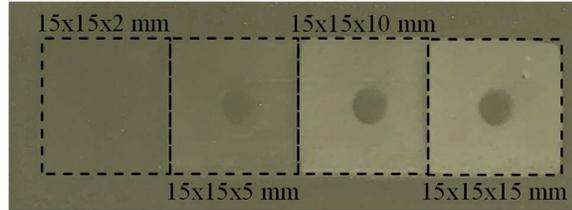
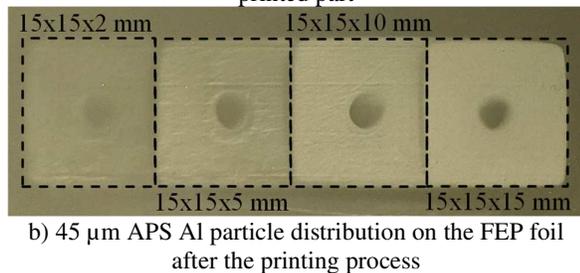
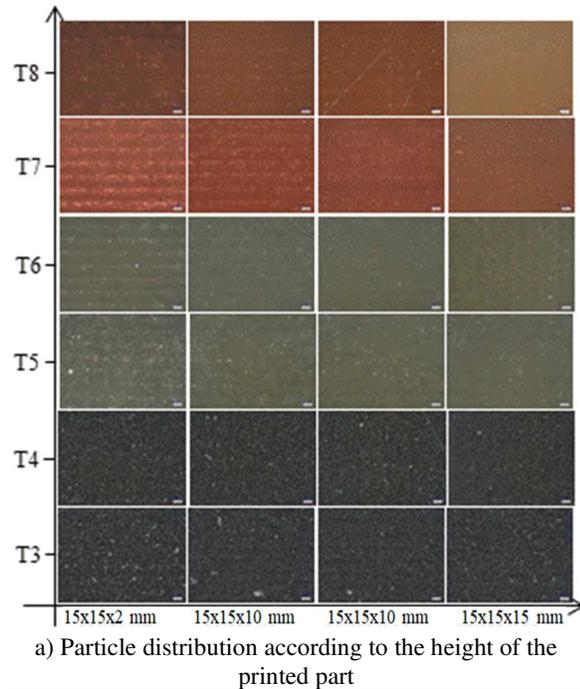
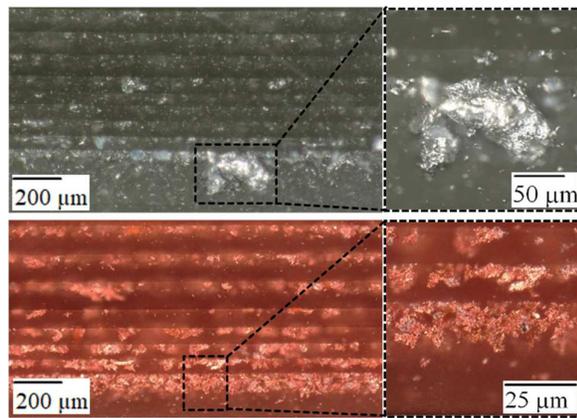


Fig.6. Particle distribution inside the printed parts and on the bottom of the 3D printer vat

Also, a higher concentration of particles was noticed (Figure 7) in the contact area of the part with the printing plate (corresponding to the “First Bottom layers” printing parameter). This indicates a start of the sedimentation process of the particles in the whole tank volume, after a time less than the sum of the bottom Et and the off time of the first layer. In this case, the sum value is 62 s (corresponding to the preliminary test results) for the Cu printing test and 79 s for the aluminum. The effect is more pronounced in the case of particles with APS 45 μm. Also within the first layers, the formation of agglomerations of filler material is visible (Figure 7 right side).



3. CONCLUSION

The paper demonstrated the capability of 3D LCD printing on desktop-type equipment with photosensitive resins (commercially available) with metallic fillers. Different metal powders (Al and Cu) with different grain sizes (5 μm and 45 μm) were added in concentrations of 1 % and 0.5 % respectively (by volume).

This approach implies printing with minimum layer heights equal to or greater than the APS of the metal powder and increasing the exposure time of each layer (implicitly the fabrication time of the part). In all samples, we obtained parts with a higher concentration of particles in the lower part (especially inside the first layers) and decreased density in the upper part. This particle sedimentation on the bottom of the primer tank is influenced by part height, particle size, density of filler and resin viscosity.

LCD printing allows the control of the printing platform movement (height and speed travel) which induces a stirring effect during the manufacture of the part. Following the tests, it is recommended to use a recoater, regularly, during the printing process, especially when high density and grain size fillers are used, but also in case of a low viscosity base resin, that does not facilitate the suspension of particles.

Several tests are required to accurately determine the concentration of metal particles inside the part. Also resins with higher viscosity and different powders can be considered (preferably with APS < 5 µm), such as polymeric ones which have a lower density than metallic powders, thus minimizing the effect of agglomeration on the first layers, maintaining the suspension for a longer period and thus decreasing printing times.

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Caracterizarea condițiilor de printare a rășinii cu pulbere de ranforsare pentru procesul de fabricare aditivă LCD

Tehnologia LCD, ca parte a grupului de fotopolimerizare în cuvă, este cunoscută de zeci de ani și utilizată ca atare în fabricarea componentelor cu rășină fotosenzitivă. Datorită evoluției de-a lungul timpului a materialelor și echipamentelor, astăzi este posibilă printarea 3D de piese macroscopice cu rășini 6K sau 8K, rășini elastice pe bază de poliuretan, rășini eco- și bio-compatibile. Caracteristicile funcționale și estetice ale componentelor imprimate prin tehnologia LCD cu rășină clasică pot fi îmbunătățite prin adăugarea de materiale de adaos/întărire sub formă de pulberi, pigmenți sau alți aditivi. Utilizând o abordare sistematică, lucrarea prezintă efectele asupra procesului de imprimare cu o astfel de rășină modificată. Modificările au constat în adăugarea de pulberi metalice (aluminium și cupru) în rășină. Modificările au constat în adăugarea a două pulberi metalice cu granulație diferită (aluminu și cupru) în rășina de bază. Rezultatele au arătat nu numai posibilitatea de a printa cu succes, ci și problemele ce pot apărea în timpul procesului. Mai multe teste au fost efectuate pe o imprimantă 3D LCD de birou, ceea ce demonstrează aplicabilitatea abordării propuse pe orice tip de echipament dedicate tehnologiei considerate.

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