Micro 3D-fabrication of ceramic components using projection micro-stereolithography technique

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ABSTRACT

The additive manufacturing of complex three-dimensional micro-structures has confounded researchers for decades. Compared with all additive manufacturing techniques projection micro-stereo lithography proves that it is the quick and reliable technique to fabricate three-dimensional microstructures in a layer by layer fashion in a bottom-up approach. This paper is mainly about fabricating a very complex and a 3D micro component made of ceramic nano-powders using projection micro-stereolithography technique. In order to get the pure ceramic component, the fabricated components are then subjected to de-binding and sintering processes. The fabricated components are supposed to have high mechanical or thermal characteristics for which the nano-indentation test and the porosity test are carried out, followed by the stereo-microscopic analysis for its dimensional accuracy.

KEYWORDS

Projection
Micro-stereolithography,
MEMS,
Ceramic Microfabrication.

1. Introduction

Ongoing researches have portrayed that additive manufacturing innovation is a breakthrough in advanced manufacturing technology. This innovation empowers the creation of little amounts of complex parts effectively. [1] Additive Manufacturing comprises of a procedure by which advanced 3D structure data is used to develop a component in layers by depositing material. The progression of Additive Manufacturing (AM) methods has fundamentally improved the capacity to fabricate structures with precise geometries. These procedures incorporate fused deposition demonstrating, selective laser sintering, stereolithography, and so forth. Stereolithography is particularly flexible to fabricate submicron-sized structures with greater complexity in the design of the structures. Micro-stereolithography is identified with rapid prototyping advances, all the more precisely to Stereolithography (SLA), permitting the fabrication of 3D products in layer-by-layer curing of a photopolymer resin with a bright (UV) laser [2].

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1.1 Micro-stereolithography (Micro-SLA)

Micro-SLA is without a doubt the one among all microfabrication methods innovated till now, that can fabricate small 3D components with the most complex details and with high-resolution. Micro-SLA is a microfabrication technique that is not quite the same as the procedures ordinarily utilized for the assembling of the Micro Electro Mechanical System (MEMS) components [3]. Micro-stereolithography is otherwise called as micro-photo-forming, spatial forming, optical forming, micro-stereo photolithography, 3D optical demonstrating and so on relating to structure varieties in the devices [4].

1.2 Projection micro-SLA

The projection micro-SLA process is quite different from the conventional Additive Manufacturing which works on the vector by vector manner. The component like dynamic pattern generator/ digital micromirror device (DMD) is the key component in projection micro-stereolithography. 3D Design by CAD modelling software are oriented, scaled and sliced consistently along the picked plane. The slices are converted into bitmap picture documents and used to work DMD, starting to

project the images from the bottom of the object. The pattern generator/DMD shapes the beam originating from the source of light with respect to the image of the layer to be projected.

The image is focused on the photopolymer by specifically fixing the UV light so, a thin layer of cured resin of the required shape is fabricated. The first layer of the 3D component to be fabricated is cured at the surface of the stage [5]. When the solidification of a layer is finished, then the polymerized part is immersed in the resin vat sufficiently, so that the polymerized surface is totally covered with new resin. The polymerized layers are stacked to one another by the interpenetrating polymers.

Micro 3D fabrication also plays a vital role in the medical field by developing Biomedical implants and Tissue engineering and so on [6]. The use of miniaturized products is growing rapidly as there is an increasing need for high-resolution small-sized parts and also for Micro-Electro-Mechanical Systems (MEMS) [7]. In this Paper, Projection micro-stereolithography is tested with alumina nano-powders because of its good mechanical properties and the ability to withstand high temperatures. Also, this newly developed material can replace the existing materials used in different applications, such as the Development of Micro robotic sensors, Microfluidic sensors, for example, tactile sensors, flow sensors and so on [8].

2. Material Selection

The Materials were selected with respect to the configuration of the projection micro-SLA machine used in this experiment. Depending upon the applications, availability, and as per current research silica, alumina nano-powders are used widely for Micro-SLA, as it satisfies all the characteristics mentioned.

First of all, the ceramic nano-powder is the primary element of the experiment and the ceramic nano-powder should be photopolymerizable. The average particle size of ceramic nano-powder should be less than 50nm because mixing the nano-powder with the resin shouldn't make the mixture too viscous which becomes difficult to fabricate. Alumina Nano-Powder is selected as Ceramic Nano-powder for this research. Comparatively, Alumina provides more strength and has an ability to withstand high temperature, and has a mean diameter of about 30nm with the density of 3.98g/ml and a Refractive index of 1.77. The resin and the photo-initiator should have the capability to cure between the 365nm to 410nm as the projection micro-SLA UV source used in this experiment is of the same range. The monomer binds the other similar monomer to form a polymer, large numbers of monomers combine to form polymers in a process called polymerization.

Monomer (resin) should be of low viscous to produce a smooth surface and the shrinkage should be less while doing polymerization and should possess high light absorption property with high curing speed so that it gets cured with low light penetration in a short span of time. Thus, 1,6-hexanediol diacrylate (HDDA) monomer is selected. HDDA has a density of 1.01g/ml, the viscosity of 6 to12Pas and the refractive index of 1.456.

Photo-initiator creates reactive species like free radicals, cations or anions when exposed to radiation, its selection is mainly depending on curing wave length of UV range. The Photo-initiator Diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide (TPO) which has high reactivity towards UV light which will generate a greater number of free radicals when exposed to UV light [9] and has a melting point at 88.92°C.

Surfactants are lowering the interfacial tension between two phases and avoids sedimentation of the Ceramic Nano-powder. The surfactant Oleic acid (Carboxylic acid) has a better affinity towards the C=C double bond, as a monomer and possess a density of 0.895g/ml and has a molar mass at 282.47g/mol with a Boiling point at 286°C is also consisting of it [10].

The ability of carboxylic acid to form stable metal carboxylate compound with ceramic particle surface depends on its miscibility with the solvent used for facilitating physisorption on the particle surface. Also, the solvent is used to ionize the surfactant/carboxylic acid, such that when it dispersed, it will separate the metal ions and hydrogen ions. Thus, Ethanol is selected as a solvent which has good solubility and has a boiling point at 78°C [9].

2.1 Preparation of ceramic nano-powder suspension

The resin HDDA and photo-initiator TPO are mixed together using a magnetic stirrer at 900rpm for 45 minutes. First oleic acid which is one of the carboxylic acids needs to be ionized hence for that

it is added in the ethanol i.e. solvent, because of that carboxylic acid (RCOOH) dissolves in ethanol, it ionizes into RCOO- and H-. RCOO- ions react with metal ions either through hydrogen bonding (Physical adsorption) or forming unidentate, bidentate linkage, or a bridge linkage (Chemical adsorption). RCOO- ions get physiosorbed on the particle surface at room temperature. The alumina nano-powder is added to it and kept in planetary ball milling at 90rpm for 1hr. After that, the mixture is heated in the oven at 90–130℃. Hydroxyl (OH) groups present on the alumina surface react with H- ions to form a water molecule, and RCOO ions of OA, get chemisorbed on the alumina surface to form aluminium oleate. This mixture heating at 130℃ results in highly hydrophobic powder. [11] The treated alumina powder is mixed with resin and photo-initiator mixture, using planetary ball milling at 150rpm for 1hr. Planetary ball milling makes the mixture to get a dense and more homogeneous mixture of alumina powder and monomer at slower rpm. Thus, the resin mixture is prepared that is to be used for fabrication.

3. Projection Micro-SLA Machine Characteristics

The setup PRO6500HR Ultrahigh-Resolution light engine is an in-house LED mask projection micro-stereolithography setup which was developed by CMTI. The user interface was setup by LabVIEW software. The stage from the company 'standa' is attached to the setup which gives XYZ direction motion and the accuracy of the stage is 0.5µm. The entire machine parameters are tabulated in Table No:1.

Table 1
Machine Characteristics.

Features	**Description**
Make and customized	CMTI
Light source	UV LED (385nm)
Interface	Lab VIEW software for effective control
Technical Specifications:	
Exposure resolution	5µ minimum
DMD mirror array	1920x 1080 i.e. (1073,600)
Max_ part dimensions	9mm x 5mm x 25mm
Layer Thickness	5 µm-50 µm (Machine accuracy: 0.5 µm)
Final pixel size (Light engine)	0.5 µm
Spot size	Minimum 10µm^2, Maximum 14.515x8.1.65mm
Build volume	11.52mm x 6.48mm x 25mm
Light engine	TI DLP6500 D M D.
Stage movement	Standa stage with maximum XYZ (25mm)
Working Distance	15mm
Power at 100%	15mW
Temperature	25°c +/-2

3.1 Working of projection micro-SLA

Basically, Projection micro-stereolithography builds a solid micro-component by stacking the fabricated layers, wherein each layer is entirely cured by one-shot irradiation of light source using DMD (Digital Micro-Mirror Device). Thus, layer by layer bottom-up approach curing of the prepolymer is done by translation of the stage in the negative Z-direction [12]. The LED light is projected on the aligned array of micro-mirrors on the DMD device according to the input mask pattern, and then, the modulated light is reflected through a reduction objective lens. Hence, a precisely reduced projection pattern is formed on a curable resin surface with a reduced shape and feature size. In each layer, the projected pattern is solidified simultaneously under one exposure due to the process of photopolymerization, while the dark regions remain liquid. After the selective curing of one layer, the substrate is immersed into the UV curable resin as per the slicing thickness parameter of the CAD model and the new layer is fabricated on top of the existing structure.

4. Working Principle

The chemistry or curing behaviour of photopolymerizable materials which are undergoing a polymerization reaction. Most Stereolithography pre-polymers contain the vinyl monomers and acrylate monomers. These vinyl monomers are significantly known as monomers containing carbon-carbon double bonds. Acrylate monomers are a subset of the vinyl family with the carboxylic acid group (-COOH) attached to the carbon-carbon double bond. For an acrylate

Resin System, the usual catalyst is free radical. In Stereolithography Radical is generated photochemically. The photochemically generated radical was because of the photo-initiator, which is an actinic photon. This produces radical (indicated by a larger dot) that catalyzes the polymerization process [13]. The whole polymer network is developed due to the cross-link between the monomer. With this property, when the ceramic nano-powders added with the resin and photo-initiator followed by exposure to UV radiation. Exposing the ceramic suspensions to UV light, the nano-particles get capsulated by the crosslinked polymer network giving the green ceramic part. These parts are then cleaned and the uncured resin is cleaned followed by the de binding process and the sintering process to get the pure ceramic parts [9].

5. Fabrication of Ceramic Component in Micro-SLA

30ml of HDDA resin is well mixed using a magnetic stirrer, with Photo-initiator TPO which is weighed 1wt% with respect to HDDA resin. The ceramic nano-powder is weighed for 40vol% with respect to HDDA and mixed using a planetary ball mill. The solvent ethanol which is of the amount 1ml/gm with respect to ceramic nano-powder and 0.5ml of Oleic acid. The product is to be fabricated with a layer thickness of 25µm having the power intensity to 75%. The images areprojected with a projection time of 1500ms.

5.1 3D model

The 3-Dimensional model of the product to be fabricated is shown in Fig No:1.

5.2 Before de-binding

The Product then fabricated with the earlier discussed parameters and Fig No:2 shows the image of the fabricated product which is then de-binded.

5.3 Post-processing

In micro-stereolithography of ceramic parts, de-binding and sintering are required to remove the binder or resin from the fabricated object so that the object becomes completely of ceramic. And it also helps in minimizing the pores and their sizes from the final fabricated part. de-binding is performed at 600℃ at a raft of 5℃/min for 3hrs. and thereafter sintering is performed to a temperature of 1550℃ at a raft of 10℃/min for 5 hrs. It is suggested that both processes must be carried out under the inert gas environment for diffusion control and defect-free structure [14].

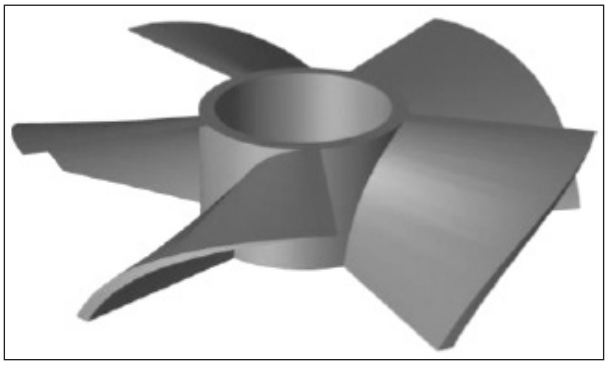

Fig. 1. Image of actual design of the turbine blade.

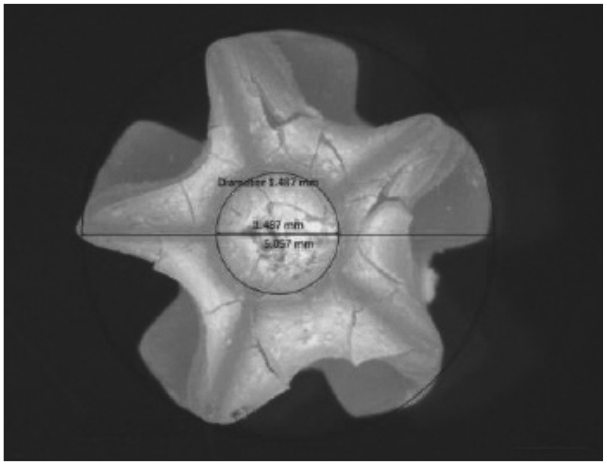

Fig. 2. Turbine blade before de-binding.

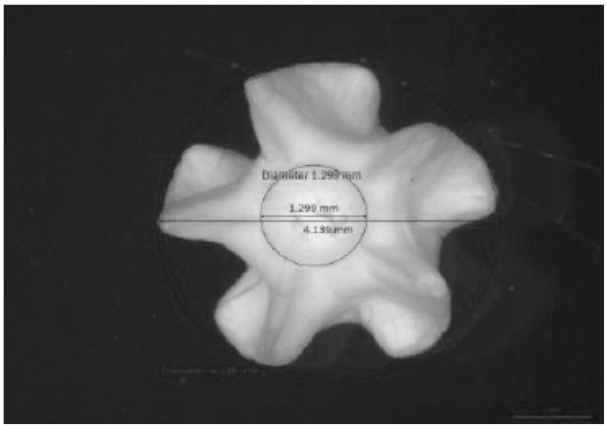

Fig. 3. Turbine blade after de-binding.

5.4 After de-binding

After fabrication of these green parts, they are subjected to de-binding and sintering at 600℃ at a raft of 5℃/min. Fig No:3 shows the image of the fabricated product after the De-binding process.

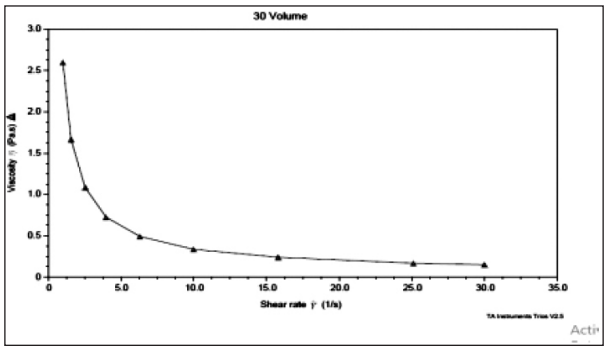

Fig. 4. Graph of viscosity vs shear rate for 30 vol% solution.

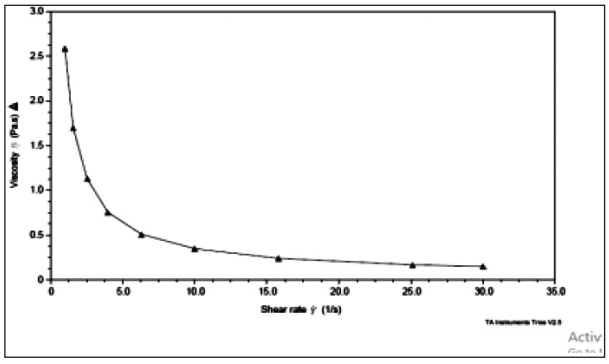

Fig. 5. Graph of viscosity vs shear rate for 40 vol% solution.

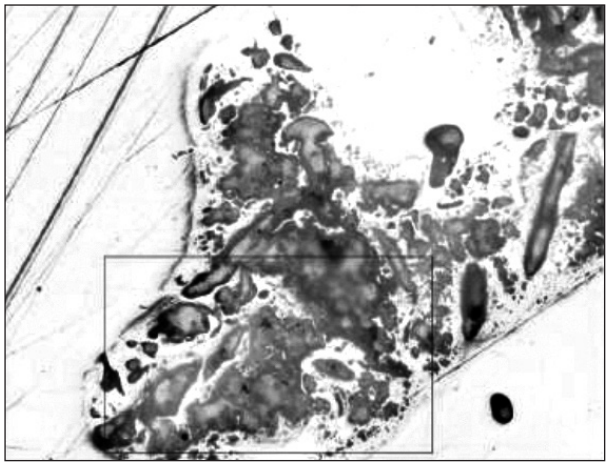

Fig. 6. Porosity Image of 30 vol% part.

6. Results

The Solution was homogeneous and in a proper viscous form so that the self-levelling of the mixture on the silicon wafer was possible. There was a shrinkage of about 20 to 30% in their actual dimension. The cracks on the surface of part are minimized as compared to the previous experiment part. The cracks are minimizing with an increased amount of alumina powder, but also the viscosity of the solution is increasing, so, it is decided to carry out the experiment to determine the optimum amount of alumina powder, which can be added into the solution, so as the solution can be self-levelling.

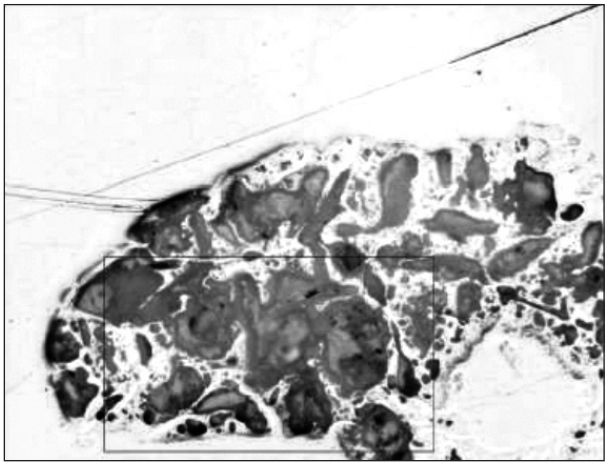

Fig. 7. Image of 40 vol% part.

7. Tests and Observations

The microstructures of the cured part were analyzed on the layer distribution cure depth. Geometrical and dimensional accuracy of the fabricated component by stereomicroscope at CMTI. The micro-components were viewed at magnifications of 50x and 150x. The Dimensional characterization has been done by using Carl Zeiss's Discovery V20. Viscosity test is done for the solution which is consisting of 30% and 40% of alumina powder, to define the viscosity limits. The test is done using Discovery hybrid Rheometer from TA Instruments, USA. Both tests are conducted at 25ºC, and at a shear rate of the $30s^{-1}$ at CMTI. And as per literature the critical value of self-levelling is 3pa.s.

7.1 30 volume % solution viscosity

The Fig No:4 shows the graph of Viscosity for the 30 volume % solution.

7.2 40 volume % solution viscosity

The Fig No:5 shows the graph of Viscosity for the 40 volume % solution.

The porosity test is conducted by using a nosepiece type microscope Eclipse LV150N, from Nikon, Japan, at CMTI. Porosity test reports for 30 volume% solution partis giving 1.5% porosity, as shown in the Fig No:6, the voids can be clearly visible.

Porosity test reports for 40 volume % solution part is giving 3.2% porosity, as shown in Fig No: 7, only the portion inside the rectangle having void is giving porosity.

8. Conclusion

The micro 3D component was fabricated successfully using 40 vol% of ceramic nano-powders with respect to resin binder. The primary aim of avoiding sedimentation of alumina powder, which requires modification from hydrophilic to hydrophobic is accomplished. The compatibility of carboxylic acid adsorbed on ceramic particles with a UV curable monomer plays a major role in forming a stable colloidal suspension. The dispersant Oleic acid disperses alumina in the HDDA monomer by improving stability and decreasing viscosity of the suspension. The ceramic nano-powder solution sedimentation cannot be permanently avoided, sedimentation will be there with time but in very little amount. This paper has proved that it is possible to obtain rather dense ceramic parts with a homogeneous microstructure by Micro-Stereolithography using suspensions of powder in a UV curable monomeric system. Sintering must be carried out on fabricated parts in the future, so that hardness, strength and high-temperature resistivity for the developed material part can be found. Dense parts can be fabricated, if continuous heating is provided to the vat during fabrication, which in-turn will reduce the viscosity of the solution.

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