

Development of a liquid toner for electro-photographic solid freeform fabrication

Navin J. Manjooran^a, Ashok Kumar^b, Wolfgang M. Sigmund^{a,*}

^a Department of Materials Science and Engineering, 225A Rhines Hall, University of Florida, Gainesville, FL 32611, USA

^b Departments of Mechanical and Aerospace Engineering, 225A Rhines Hall, University of Florida, Gainesville, FL 32611, USA

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Abstract

Solid freeform fabrication is still a major challenge for ceramic materials. Here we discuss a novel solid freeform method, “Electro-photographic solid freeform fabrication” (ESFF) that needs specific qualities for solid (powder) and liquid (slurry) toner (electronic ink). The process is similar to photocopying using ceramic particles. Overall ESFF is based on slicing a 3D structure into 2D layers and depositing these layers one over the other by a photosensitive photoconductor drum. Here we present the development of a liquid toner based on submicron particles, a study of its flow behavior and electrophoretic tests confirming that the toner could be used for ESFF.

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1. Introduction

Since the early 1950's attempts have been made to generate 3D objects without tooling.¹ By 1980, the physical prerequisites were available with a specific consolidating feature via laser beam. In 1986, Hull and Charles^{2,3} patented the stereolithography apparatus—the first solid freeform fabrication process. By this process 3D plastic parts could be created directly from computer aided design (CAD) data. From then on many SFF techniques have been developed.

Solid freeform fabrication (SFF) is a method to enable fabrication of custom objects with certain desired properties from computer data.^{4,5} This is basically layer by layer manufacturing of three-dimensional objects. Due to this layer-by-layer building approach, quicker and cheaper production of prototypes can be made. The solid model of the part to be manufactured is created in CAD software and exported to the SFF process via a software interface.⁴ The SFF process deposits various materials layer-by-layer in the shape of the cross-section of the solid to create the part.

Solid freeform fabrication methods can be classified on the basis of the raw material used, lighting of photopolymers, and by the application range for which it is used.² SFF has been associated with manufacturing environments, where it is used for the rapid production of visual models, low run tooling and functional objects.⁶ The additive nature of SFF techniques offers great promise for producing objects with unique material combinations and geometries which could not be attained by traditional methods and are different from most machining processes (milling, drilling, and grinding, etc.), as these are subtractive processes that remove material from a solid block. So it can be used in diverse fields as aerospace, electronics, architecture, biomedical engineering and archaeology.⁶ SFF allows designers to quickly create tangible prototypes of their designs rather than two-dimensional pictures. These help in making less expensive and excellent visual aids for communicating ideas with co-workers or customers. It can also be used for design testing. For small production runs and complicated objects, SFF is the best manufacturing process available. The time required to build the prototype depends on the size and complexity of the object. The time saving allows manufacturers to bring products to the market faster and more economically.⁶

* Corresponding author. Tel.: +1 352 846 3343; fax: +1 352 846 3355.
E-mail address: wsigm@mse.ufl.edu (W.M. Sigmund).

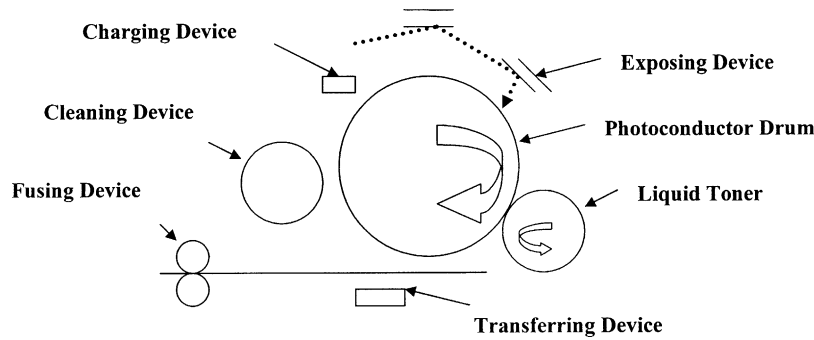


Fig. 1. Schematic of the electro-photographic solid freeform fabrication (ESFF) process.

Electro-photographic solid freeform fabrication (ESFF) is a novel solid freeform fabrication technique. It is a new rapid prototyping method patented by Kumar.^{1,7,8} It uses the electro-photography technique to deposit the particles layer-by-layer on a specially designed platform. During the electro-photography process, the particles are picked up by a charged surface and deposited on oppositely charged surface. Therefore, it is important to know the characteristics, especially the charging characteristics of the particles in this process. Kumar¹ has designed a test-bed called ESFF machine, which deposits the particles in the requisite cross section layer-by-layer on a numerically controlled two-axis platform. A heating and compacting system is used to fuse each layer of particles deposited. Fig. 1, shows a schematic presentation of how the ESFF process works. The photoconductor drum is charged with the help of a charging roller using a corona discharge. The laser image projector makes the image on the photoconductor drum by removing the charge from the drum at the required regions. From the image developer the particles are attracted to the magnetic development drum, by applying a high voltage to it. The particles then get transferred to the photoconductor drum at the regions required because of the opposite polarity. The developed image is then transferred onto the build platform with the help of an electric field and then permanently fixed by fusing. The photoconductor surface needs to be cleaned using physical or electrical methods, before the process is repeated to get the three dimensional object.

Of late there has been a great demand world wide for electro-photography based full color printing devices for both Small Office and Home Office (SOHO) and for the heavy volume commercial application.^{9–12} The printing “ink” or “toner” (electronic ink) can be either in the powder form or the toner particles can be suspended in a dielectric liquid (liquid toner). There has been an increasing demand for a high quality short run printing and the liquid development process using a liquid toner can meet this demand well because with this we can achieve high resolution, good image quality and a better packing uniformity.¹³

Rheology plays an important role in the development of a liquid toner. The choice of the dispersant, its critical amount,

solids loading, relative viscosity, shear rate and shear stress response are important parameters that need to be taken into account. In liquid toner systems, non-aqueous dispersions consisting of micron sized particles stabilized in an adsorbed layer of the polymer and charge control agents in aliphatic hydrocarbons are used as toners.¹⁴ This paper aims to develop a liquid toner that can be used for the electro-photographic solid freeform fabrication. The experiments allowed us to define the simplest system producing acceptable deposits that can be used for ESFF.

2. Materials selection

Decahydronaphthalene ($C_{10}H_{18}$) was used as the solvent. As, ISOPAR (ISO-PARaffinic hydrocarbon-highly branched alkanes with 7–15 carbon atoms), the solvent used in most industrial applications for the development for the liquid toner’s structure not well understood, a solvent with better properties than ISOPAR and a clearly defined structure was selected.^{15–17} The properties considered were a high flash point ($57.22^\circ C$ for decahydronaphthalene), non-polar nature, non-conductivity, chemical inertness, relatively non-viscous nature and volatility. Decahydronaphthalene meets the majority of these requirements.

Silicon carbide (SiC) sub-micron sized particles were used, as they are an important ceramic for structural and electrical applications, because of their excellent mechanical and electrical properties at high temperatures.^{17,18} Sub-micron sized particles were made use of as these particles are significantly smaller than dry toner particles and hence are capable of producing very high resolution images.¹³

To find suitable dispersing agents for SiC and $C_{10}H_{18}$, the literature values for the refractive indices at $25^\circ C$ were looked up 2.649¹⁹ and 1.475,²⁰ as well as the materials dielectric constants 9.71²¹ and 2.196,²² respectively. Using these values in the Tabor Winterton Approximation (TBA), the Hamaker constant was calculated to be 2.233×10^{-19} J for the SiC- $C_{10}H_{18}$ -SiC system.²³ The Derjaguin-Landau-Verwey-Overbeek [DLVO] theory for sphere-sphere interactions at $25^\circ C$ was used to calculate the dependence of

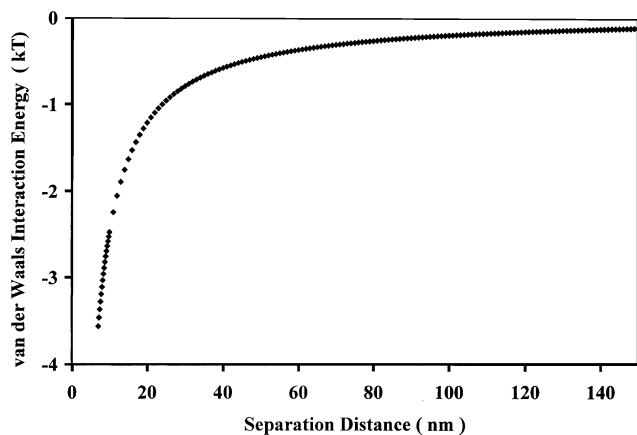


Fig. 2. Theoretical calculation of van der Waals interaction energy (kT) for the sphere-sphere interaction of SiC particles. ($T=25\text{ }^{\circ}\text{C}$, particle radius: $R=0.55\text{ }\mu\text{m}$, Hamaker constant: $A_{121}=2.23\times 10^{-19}\text{ J}$ (calculated using the Tabor Winterton approximation)).

separation distance of the SiC particles with respect to the van der Waals interaction energy. The results are plotted in Fig. 2. There is an increase in the magnitude of the van der Waals interaction energy as the particle separation distance decreases. As soon as particles approach closer than 40 nm the van der Waals interaction energy becomes larger than the thermal energy. If a surface barrier at 20 nm is introduced on the particles by using a polymer, coagulation of the particles due to van der Waals interaction should be avoided. The first selection criterion for suitable polymers is the solubility. The inner pressure, which indicates the solubility of polymers, was used for choosing the polymers. The solubility parameter handbook lists polybutadiene at $18.0\text{ (MPa)}^{0.5}$ polystyrene at $18.6\text{ (MPa)}^{0.5}$ similarly to that of $\text{C}_{10}\text{H}_{18}$ with $18.0\text{ (MPa)}^{0.5}$.²⁴ In a next step the molecular weight for these polymers was chosen based on estimates for the radius of gyration (r).²⁵

$$r^2 = cnl^2$$

Where, ' l ' is the segment length, ' c ' is a constant and ' n ' is the number of segments. The average molecular weights required for polystyrene and polybutadiene were found to be in the order of 10^5 and 10^3 , respectively. Actual solubility tests showed that polybutadiene and polystyrene were well soluble in the non-polar solvent. Thus, polybutadiene, polystyrene and for comparison, Hypermer LP1 (High performance polymer-an industrially used polymeric dispersant) were chosen.

In addition to the colloidal properties of the slurry, it is important to control the charging of the toner for enhanced deposition. Typically, particle charge controlling agents are added in industrially used systems to control the magnitude and sign of the surface charge of the particles.^{17,26–28} Therefore, CCA7, an industrially used negative charging agent, was introduced into the slurries to study the effect of these compounds.

3. Raw materials and specimen preparation

The starting materials were 6H-alpha-silicon carbide (Grade UF-15, H.C. Starck, Canada), *cis-trans* decahydronaphthalene (Aldrich), polystyrene (Aldrich), polybutadiene (Aldrich), LP1 (Uniqema, Belgium) and Charge Controlling Agent 7 (Avecia-Inc.). The silicon carbide (SiC) and Charge Controlling Agent 7 (CCA7), particle sizes, were measured with the Brookhaven instruments—Zeta plus particle sizing and were found to yield a d_{50} of 0.52 ± 0.02 and $0.42\pm 0.02\text{ }\mu\text{m}$, respectively. CCA7 is primarily a chromium complex designed for use in negative toner systems. The average molecular weights for polystyrene, polybutadiene and LP1 are $\sim 230,000$, ~ 5000 and ~ 6000 , respectively. The surface area of SiC, measured by BET (AREAMETER II) N_2 adsorption is $15\text{ m}^2/\text{g}$ (H.C. Starck). Polystyrene, polybutadiene and LP1 are used as dispersants for the different experiments and their amounts are based on the wt% of the dry SiC powder.

Measured amounts of decahydronaphthalene and the polymer (polystyrene or polybutadiene or LP1) are taken in a beaker and placed on a hot stirrer till the polymer dissolves in the carrier. This is followed by the addition of measured amounts of CCA7 and the SiC powder then placing the mixture in an ultrasonicator (misonix sonicator 3000) for 4 h. The generator provides high voltage energy pulses at 20 kHz and takes care of the changes in the load conditions such as viscosity and temperature. A titanium disruptor horn transmits and focuses oscillations of the piezoelectric crystals and causes radiation of energy that under a phenomenon called "cavitation" (formation and destruction of the microscopic vapor bubbles that the sound waves generate) produces the shearing and tearing action necessary for the slurry formation. This procedure is used for making all slurries.

Viscosity measurements were performed using a modular compact rheometer (MCR 300, Paar Physica) with a concentric cylinder system using the US200 universal software. The inner cylinder diameter is 27 mm. The shear flow measurements are operated at $25\text{ }^{\circ}\text{C}$. The shear rate changes from 10^{-3} to 10^3 s^{-1} . The temperature control unit is TEZ150P, which features Peltier heating. For all experiments the slurry is pre-sheared at 800 s^{-1} for one minute, then kept stationary for 10 s to allow for a steady state and then the measurement is carried out. The relative viscosity is calculated as the ratio of the viscosity of the suspension to the viscosity of decahydronaphthalene at the same temperature.²⁹

The voltage measurements during the electrophoretic deposition are done using a DC voltage source (1–5 kV, Matsusada). Stainless steel electrodes act as cathode and anode in a glass container containing the slurry. The gap between the electrodes is fixed at 6 cm. One of the electrodes is grounded and on the other a developing bias voltage is applied. The application of several kV leads to the deposition of SiC particles on the cathode. The optical density, which is the mass of dried slurry per unit area deposited, is measured with varying voltages and times as well as chemical composition of slurry.

The optical density gives information on the darkness of the print.^{13,30–32}

4. Results and discussion

4.1. Determination of optimum amount of polymer

The amount of the polymer added to the suspension must be just enough so that it covers the SiC surface completely. The addition of excess may cause an increase in the viscosity and if less than the required amount is added, bridging flocculation due to incomplete surface coverage may result. With the optimum amount of polymer dispersant, the repulsive barrier potential will be large enough to overcome the attractive potential. At the same time the smallest amount of free polymer is in solution and no surface sites for adsorption are left so bridging flocculation is hindered.^{25,29} The optimum amount was studied with rheology, since all the above parameters are expressed in rheological responses. It can be defined more simplified as the amount of dispersant per solids loading with the lowest viscosity. In Fig. 3, the optimum amount for polystyrene, polybutadiene and LP1 are found to be 0.4, 0.3 and 0.4 wt% of SiC, respectively, for a 5 vol% SiC slurry.

4.2. Determination of optimum amount of charge controlling agent

In industrial systems, particle charging agents are added to control the magnitude and the sign of the charge, for better dispersability and to adjust the triboelectrically generated charge of the toner.¹⁷ Since the charge-controlling agent contains inorganic components that will not burn out, it would be best to avoid them for ESFF. However, as the later data of this investigation shows, the charge-controlling agent has quite dramatic effects on the quality of the ceramic toner. Therefore, we define the optimum amount of the charge-controlling agent in the suspension as the minimum amount that must be added to get the highest amount of uniform deposition.

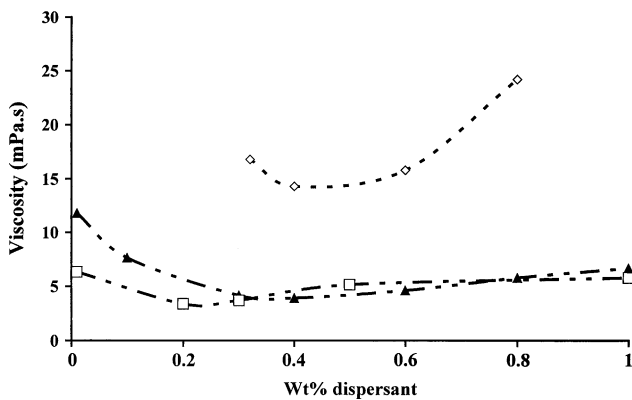


Fig. 3. Viscosity plots for 5 vol% SiC slurries in decahydronaphthalene with varying amounts of dispersants: polystyrene (◇), polybutadiene (□) and LP1 (▲).

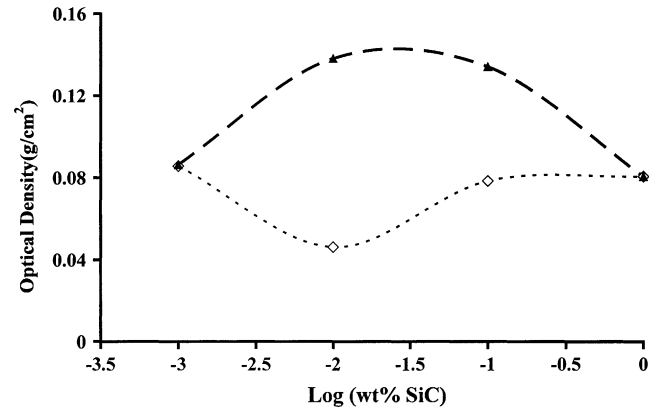


Fig. 4. Optical density (deposited mass per area) in dependence of charge-controlling agent (CCA7) for dispersants LP1 (▲) and polystyrene (◇). Fixed conditions: 5 vol% SiC in decahydronaphthalene, amount of LP1 or polystyrene (PS) 0.4 wt% of SiC, $T=25^{\circ}\text{C}$.

Fig. 4 shows that there is an increase in the optical density with addition of CCA7 to the LP1 dispersed slurry and after further addition of CCA7 the value decreases. A maximum is found when the amount of CCA7 is 0.1 times the amount of polymer. On the other hand, polystyrene slurries appear to respond more complex. Since a clear maximum is missing in this data, the optimum amount of CCA7 is chosen as 0.1 times the amount of the added polymer based on additional observations such as the visual quality of the deposited layer.

4.3. Volume fraction dependence of the relative viscosity-modified dougherty krieger fit

The viscosity of the slurry depends on the maximum solids loading and this value reaches infinity at the maximum volume fraction (Φ_m). The maximum volume fraction depends on the particle size and the particle shape. At the

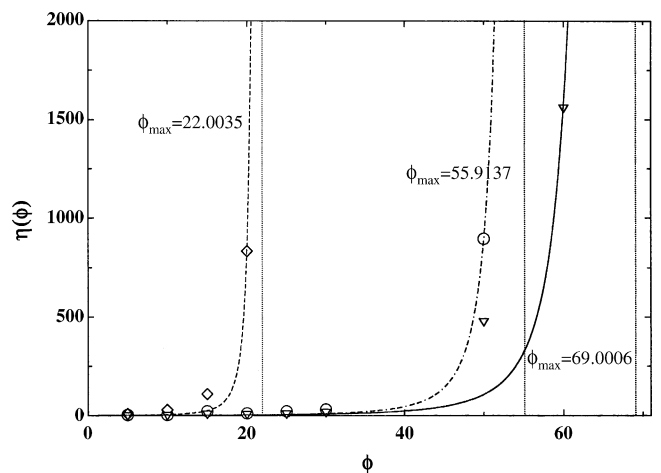


Fig. 5. Dependence of relative viscosity $[\eta(\Phi)]$ on solids loading (Φ) of SiC in decahydronaphthalene for dispersants LP1 (○) and polystyrene (◇) and polybutadiene (Δ) at 0.4, 0.3 and 0.3 wt% SiC, respectively. The amount of charge controlling agent [CCA7] added was 0.04 wt% of SiC for LP1 and polystyrene and 0.03 wt% of SiC for polybutadiene.

maximum volume fraction, the particles in the slurry are so close together that they start touching which hinders flow and causes transition into gelled body.^{23,25} The rheological data are presented in Fig. 5. The experimental points have been fitted to the modified Krieger-Dougherty²³ equation (shown below), where Φ_m and n are fitting parameters:

$$\eta_r = \left(1 - \frac{\phi}{\phi_m}\right)^{-[\eta]\phi_m}$$

The best fit of the experimental data shows that Φ_m is drastically lower for the suspension with polystyrene ($\Phi_m = 0.23$) compared to the LP1 suspension ($\Phi_m = 0.52$) or the polybutadiene suspension ($\Phi_m = 0.62$). The lower Φ_m value illustrates that the packing behavior is poor in these cases.

ESFF requires the deposition of the toner on a charged surface and the subsequent transfer of this deposited layer onto the 3D object that is being developed. The non-charged surface of the electrode should not have any particles sticking

to it. Only charged surfaces of the electrode should be covered with particles. Therefore, the adsorption and adhesion of particles on the electrode surface is of importance. This was studied with non-adhesion tests with variation in dispersant chemistry and solids loading.

For the non-adhesion tests, the steel electrode was dipped into slurries with LP1, polybutadiene and polystyrene as dispersants and at different solids loading of 5, 10, 15, 20, 25, 30, 50 and 60 vol% SiC. The objective was to find the slurry composition that had a minimum or no SiC particles deposited on the electrode even though it was dipped and kept in the slurry for one minute. In this test slurries with lower volume fractions performed better. Furthermore, slurries containing polystyrene and LP1 at 5 vol% SiC showed best results. So, further experiments were carried out with 5 vol% SiC using these two polymer dispersants. In contrast, polybutadiene slurries always tend to adhere to the electrode surface and therefore were not further considered in this study.

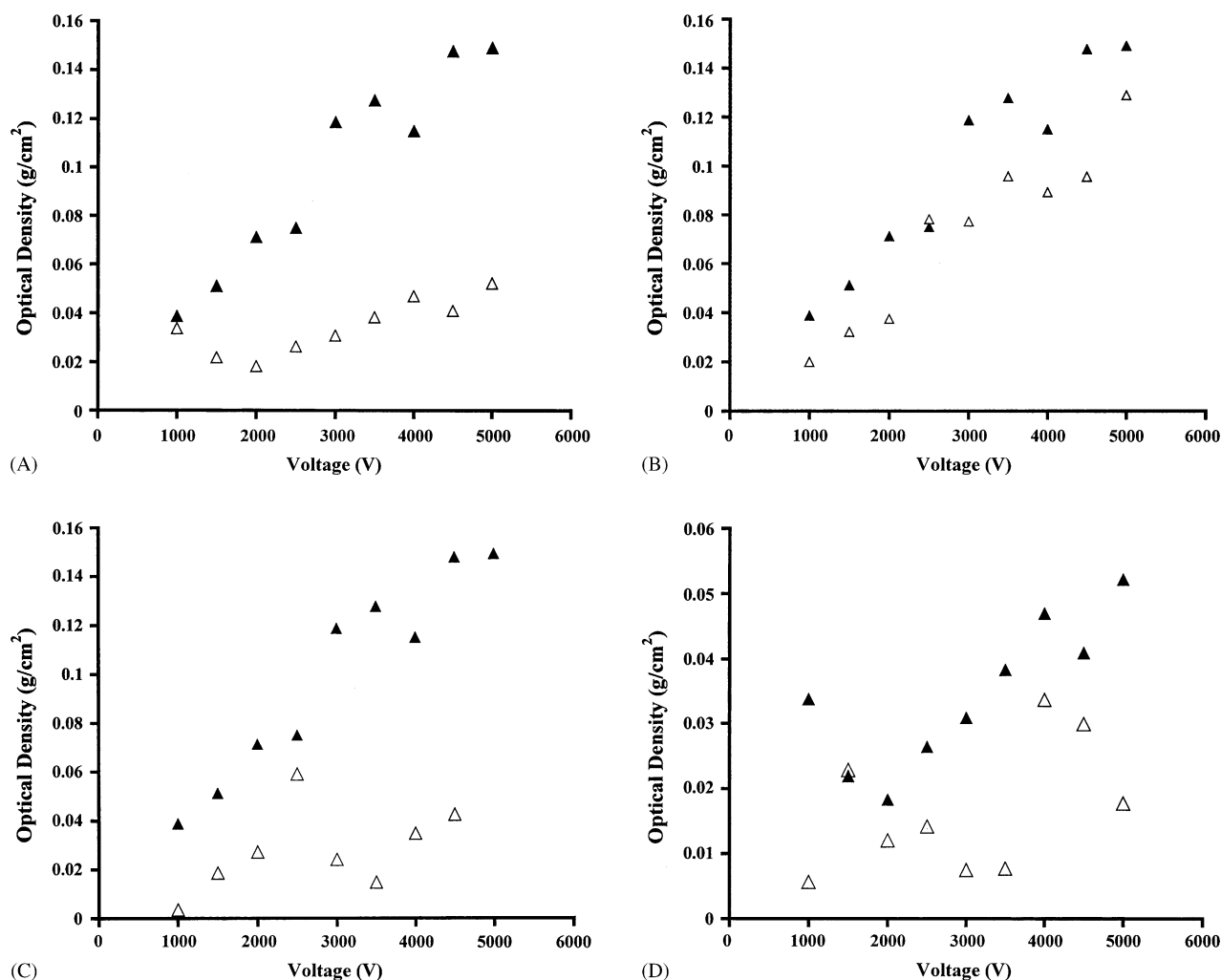


Fig. 6. Optical density in dependence of applied deposition voltage. Fixed conditions: Plots for 5 vol% SiC in decahydronaphthalene with 0.4 wt% LP1 and polystyrene (PS) of SiC. CCA describes slurries where 0.04 wt% CCA7 of SiC was added. W-CCA stands for slurries without CCA7. The presented time (min) is the actual deposition period. (A) LP1, 2 min, CCA (\blacktriangle) and PS, 2 min, CCA (\triangle). (B) LP1, 1 min, CCA (\blacktriangle) and LP1, 2 min, CCA (\triangle). (C) LP1, 2 min, CCA (\blacktriangle) and LP1, 2 min, W-CCA (\triangle). (D) PS, 2 min, CCA (\blacktriangle) and PS, 2 min, W-CCA (\triangle).

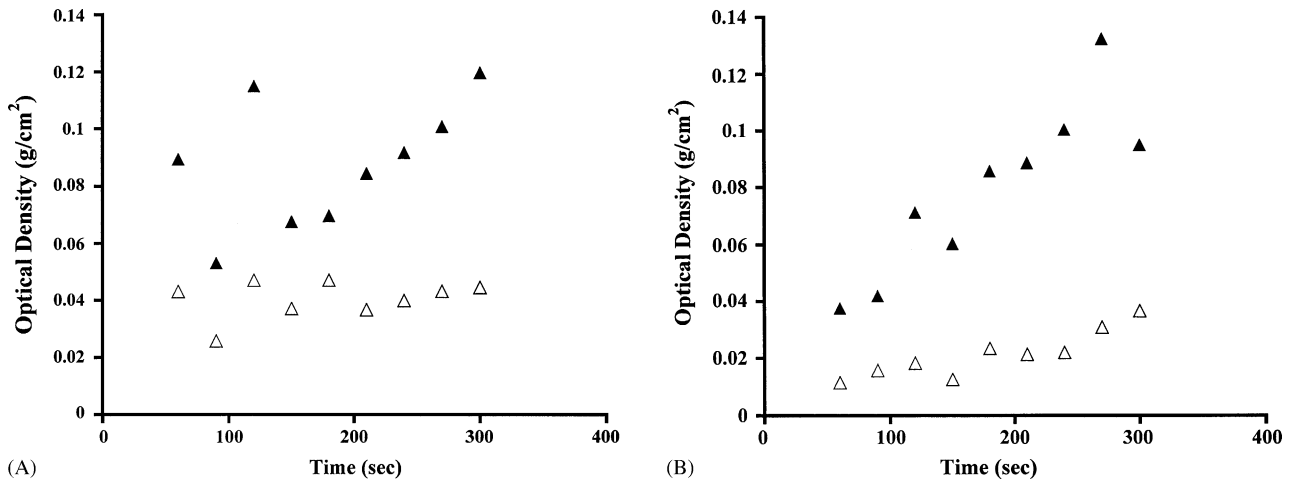


Fig. 7. Optical density in dependence of deposition time for 5 vol% SiC in decahydronaphthalene with 0.4 wt% LP1 or polystyrene (PS). CCA indicates slurries where 0.04 wt% CCA7 of SiC was added. Voltages are DC deposition voltages. (A) LP1, 4 kV, CCA (▲) and PS, 4 kV, CCA (△). (B) LP1, 2 kV, CCA (▲) and PS, 2 kV, CCA (△).

5. Results from deposition

5.1. Dependence of optical density with voltage

Fig. 6 shows the optical density dependence of the LP1 and polystyrene slurries with and without CCA7 at different voltages. It can be inferred from the data that LP1 slurries are better than polystyrene slurries as they have a much higher optical density at the same voltage (See Fig. 6A). Therefore, better liquid toners can be made using the LP1 slurries. It can also be inferred from the data that a higher optical density is obtained with the presence of the charge-controlling agent (See Fig. 6C and D). Furthermore, the longer the deposition time the larger was the optical density (See Fig. 6B). Also, a high positive voltage caused deposition on the anode with the LP1 slurries and no deposition on the cathode. Reversing the voltage even allows completely removing the adhering layer. This property of the LP1 slurries is most crucial for electrophotographic solid freeform fabrication, since the layer needs to be transferred to the 3D part in development. This behavior was not observed for polystyrene slurries. Particles seemed not to have uniform charge distribution since both negative as well as positive voltages yielded the deposition of particles on the steel electrodes. Reversing the electric field did not clear the electrode surfaces either. Therefore, these polystyrene slurries cannot be used as a liquid toner as they do not completely follow the adhesion-non-adhesion behavior with the application of alternating positive and negative voltages. Overall a DC voltage of +4 kV was found to be best in this current investigation since uniform deposition was seen to take place at this voltage.

5.2. Dependence of optical density on time

Fig. 7 shows the optical density dependence of LP1 and polystyrene slurries with the addition of CCA7 at different

time intervals. LP1 slurries are found to yield a much higher optical density as compared to polystyrene slurries for the same deposition time (See Fig. 7A and B). Therefore, it can be concluded that better liquid toners can be made using LP1 based slurries. As expected an increase in voltage gave a higher optical density (comparing the data of 7A and 7B) but the impact is less strong than the variation in the dispersant chemistry. Due to the transport phenomenon an expected increase in optical density with deposition time is also confirmed. For liquid toner applications shorter deposition times are preferred. The achievable optical density for LP1 slurries with shortest times indicates their usefulness as a liquid toner. Furthermore, it was found via visible inspection that the uniformity of the deposited layer improved with voltages around +4 kV.

6. Conclusions

The lowest viscosity for polystyrene, polybutadiene and LP1 polymer stabilized SiC slurries were found to be 0.4, 0.3 and 0.4 wt% of SiC, respectively. It is hypothesized that these amounts cover the submicron SiC particle surface completely in decahydronaphthalene while having a minimum amount of free polymer in solution. A maximum deposited layer of SiC particles, which is equivalent to optical density is found when the amount of CCA7 is 0.1 times the amount of LP1 in the slurry. At lower volume fractions the electrophoretic adhesive-non-adhesive behavior was seen better for slurries with polystyrene and LP1, with a perfect examples seen at 5 vol% SiC. Polybutadiene slurries did not make a favorable response in the non-adhesion tests and so were considered “not good” for making a liquid toner. Overall, better liquid toners can be designed using LP1 stabilized slurries as they have a much higher optical density as compared to polystyrene slurries under similar conditions. Polystyrene

stabilized SiC slurries cannot be used as a liquid toner as they do not completely follow the adhesive-non-adhesive behavior with the application of alternating positive and negative voltages. Good quality of deposited layers was found for +4 kV DC. The introduction of a charge-controlling agent (CCA7) to the slurry strongly enhances the optical density. Furthermore, better optical densities are observed with longer deposition times. A stop in increase in the deposited amount is reached when the electrode is completely covered by SiC particles.

Hence from the different results obtained the ideal liquid toner or slurry to be used for electro-photographic solid freeform fabrication is a decahydronaphthalene slurry that contains 5 vol% SiC, has 0.4 wt% SiC of the polymer LP1 and has 0.04 wt% CCA7 of SiC as charge-controlling agent. An ideal deposition voltage was found to be + 4 kV.

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