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First observation of the effect of the layer printing sequence on the molecular structure of three-dimensionally printed polymer, as shown by in-plane capacitance measurement

Naga B. Gundrati[a], Patatri Chakraborty[a], Chi Zhou[b], D.D.L. Chung[a,*]

[a] Composite Materials Research Laboratory, Department of Mechanical and Aerospace Engineering, University at Buffalo, The State University of New York, Buffalo, NY 14260-4400, USA

[b] Department of Industrial and Systems Engineering, University at Buffalo, The State University of New York, Buffalo, NY 14260, USA

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A B S T R A C T

The layer printing sequence in three-dimensional (3D) printing affects the molecular structure of the printed layers, as shown for bottom-up stereolithography printing using an ultraviolet-curable acrylate ester resin. The first printed layer has less molecular alignment than the last printed layer, due to the longer ultraviolet exposure and consequent more curing in the former. Increasing the layer thickness from 12.4 to 26.3 μm decreases this effect, due to the greater molecular alignment for a smaller layer thickness. These results are obtained by measuring the in-plane capacitance for the two opposite surfaces of the printed material. The technique is unprecedented in 3D printing.

1. Introduction

Three-dimensional (3D) printing involves layer-by-layer deposition of a material and is powerful for the fabrication of objects with complex shapes [1–7]. It is most commonly performed using polymers, which are easier to process than metals or ceramics, due to the typically lower required temperatures. A polymer consists of long molecules that tend to have a kinked conformation. The length of a molecule is typically large compared to the thickness of a printed layer. As a consequence, a degree of alignment of the molecules in the plane of a layer is expected. In fact, photopolymerization-induced in-plane molecular alignment has been reported for an acrylate polymer film, based on optical observation [8]. In-plane molecular alignment has also been reported for other polymer films [9–22] and molecular films [23,24]. All of these films in the prior work are not 3D-printed multilayers. It is well-known that the molecular alignment affects the mechanical properties of polymers [25–30]. Thus, the texture associated with the molecular alignment is expected to cause the mechanical properties (strength and elastic modulus) of the printed layer to be superior in the in-plane direction than the out-of-plane direction [31]. Molecular alignment in a polymer is to be distinguished from the alignment of fibers [32], nanofibers [33] or nanotubes [34,35] in a polymer matrix.

During curing, a resin undergoes cross-linking, which covalently connects the different molecules at points, thereby causing the resin to become a solid, in addition to causing a degree of three-dimensional networking of the molecules. Due to the networking, the cross-linking increases the strength and modulus, while tending to decrease the degree of texture.

In case of 3D printing involving a photocurable resin and light for the curing shone from below, such that the printed object is built up layer-by-layer from the bottom upward, the printed layer sequence (e.g., the first layer printed vs. the last layer printed) is expected to affect the molecular structure of the layers. In particular, with this method of printing, the first layer printed is exposed to the light during the period in which it and all subsequent layers are being printed. As a consequence, the first layer printed is expected to be more completely cured than the last layer printed. In spite of such expectation, there is no prior work on the effect of the layer printing sequence on the structure or properties of the various layers in the sequence.

This paper is primarily aimed at investigating the effect of the layer printing sequence on the molecular structure of a 3D printed polymer printed by using ultraviolet radiation [24,25] for curing the resin. For this purpose, the first layer printed and the last layer printed, which are located on the opposite sides of a printed multilayer, are studied electrically in this work.

The electrical method used in this work involves the measurement of the in-plane capacitance of the surface region of either side of the multilayer. The in-plane capacitance is to be distinguished from the

* Corresponding author.
E-mail addresses: nagabhar@buffalo.edu (N.B. Gundrati), patatric@buffalo.edu (P. Chakraborty), chizhou@buffalo.edu (C. Zhou), ddlchung@buffalo.edu (D.D.L. Chung).
URL: http://alum.mit.edu/www/ddlchung (D.D.L. Chung).

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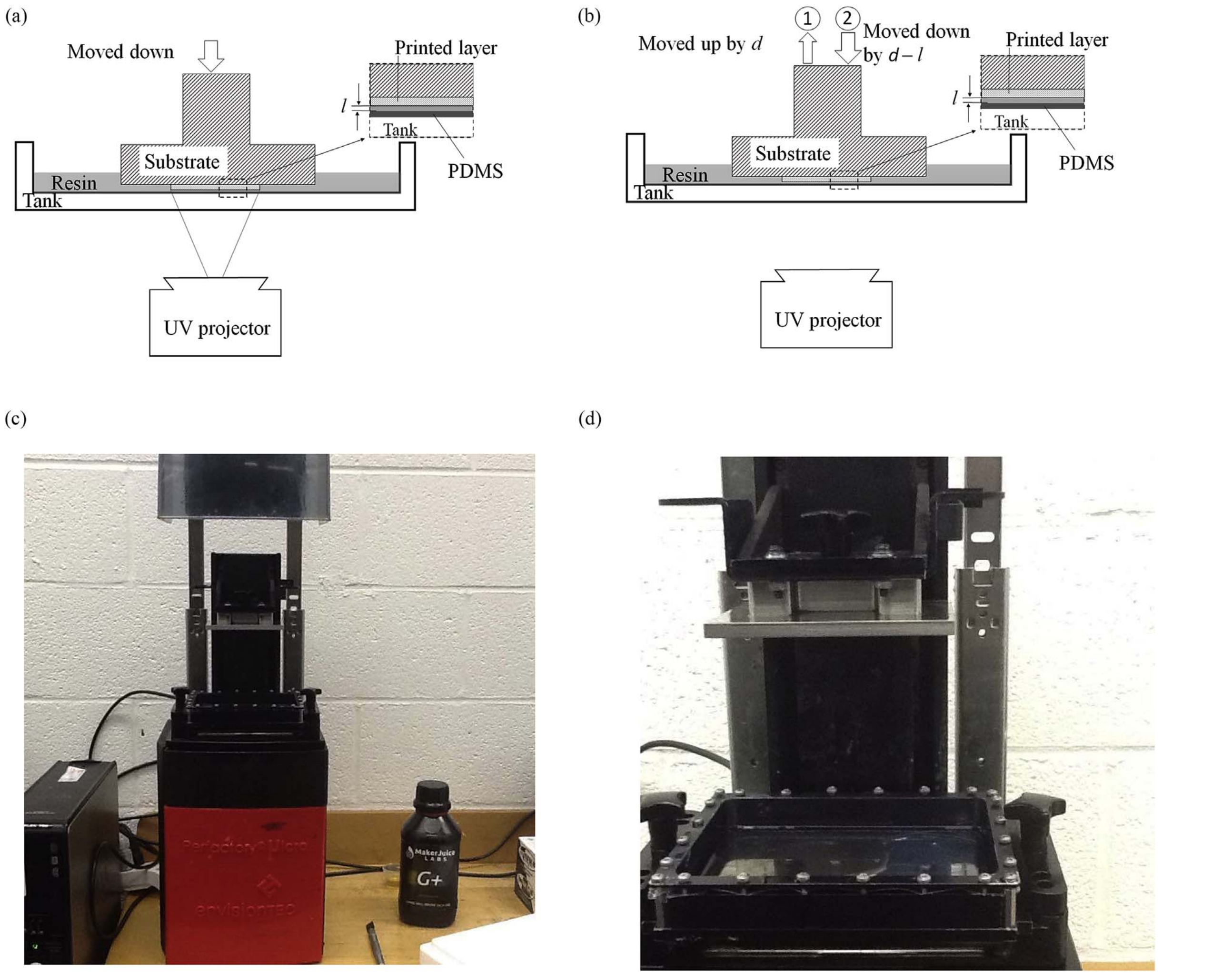

Fig. 1. The 3D printing method. (a) The motor powered substrate is moved down until the gap between the substrate and the tank bottom is equal to the intended layer thickness *l*. Then a converted image of a layer is projected on the tank bottom (which is transparent to UV light) using UV light for a selected time period. (b) The UV light is turned off and the substrate is moved up for distance *d* ($d << l$) to let the resin flow back to the projected area. Then the tank is moved down by $d – l$. (c) Photograph of the full printer. (d) Close-up photograph of the platform and tank part of the printer.

through-thickness capacitance of our prior work [36,37]. It is measured by using electrical contacts that are on the same surface, so that the current path between the two electrodes is mainly in the in-plane direction. In contrast, the through-thickness capacitance of our prior work is measured by using electrical contacts that are on the two opposite surfaces of the specimen, so that the current path is in the through-thickness direction [36,37]. Based on the through-thickness capacitance and the thorough-thickness relative permittivity that is obtained from this capacitance, the prior work [36,37] has shown that a higher degree of preferred orientation of the molecules results in greater polarizability in the direction of the alignment, and hence a higher value of the through-thickness relative permittivity.

In spite of the explosive growth of 3D printing, the control of the process is inadequate, as shown by the fact that each printer has its own "personality" and that essentially no information on the quality of the printing can be obtained during the printing. No information is available to guide the adjustment of the printing conditions during the printing. There is a critical need for the monitoring of the printing process. No effective way of monitoring has been previously reported.

After printing, stress relaxation tends to occur, as shown by the tendency for the printed product to curl up. The stress is built up during the printing due to the difference in the degree of cure or the temperature among the layers that are being printed layer-by-layer. In case of printing involving a resin, additional curing tends to occur in the printed product after printing. Furthermore, during the use of the printed product, the quality of the product may degrade due to the applied stress or other environmental conditions. Thus, there is also need to monitor the quality of the printed product after printing and during use.

Prior work on the monitoring of the 3D printing process has involved measurement of the temperature distribution using an infrared camera [38]. Although the temperature distribution gives information on the degree of spatial uniformity of the printing process, it does not give quantitative information on the quality of the material being printed and the spatial resolution of the infrared camera is low.

A secondary objective of this work is to provide the science base for a new method of monitoring 3D printing by in-plane capacitance measurement. The in-plane capacitance measurement is more suitable for practical implementation during 3D printing than the through-thickness capacitance measurement, but it does not provide a determination of the permittivity. In contrast, the through-thickness measurement allows a determination of the permittivity. The capacitance method does not involve the embedment of sensors in the printed layers. The embedment would cause degradation of the mechanical properties of the printed product.

This paper pertains to the printing of a polymer rather than a polymer-matrix composite. The ability to monitor without the need for the incorporation of a filler in the polymer or resin feedstock material is

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