

METHODS OF DEPOSITING ANTI-REFLECTIVE COATINGS FOR ADDITIVELY MANUFACTURED OPTICS

Z. J. Beller*, D. M. Keicher*, E. B. Secor*, J. Lavin*, M. Essien†, S. Whetten*, S. S. Mani*

*Sandia National Laboratories

†Integrated Deposition Solutions, Inc.

Abstract

Recent advancements in the field of additive manufacturing (AM) have enabled the production of high-fidelity optical components allowing for the design of novel fiber optic systems. In order to support this emerging technology, methods of depositing anti-reflective coatings (ARCs) onto these optical components must be developed. Work has begun to identify such coating materials; develop systems capable of accurately depositing controlled, uniform layers onto given substrates; establish deposition procedures for ensuring coating validity; and establish post-processing procedures to ensure the reliability of finished components. Areas of interest for finished components include their integration into high-bandwidth fiber optic systems, enabling further miniaturization of communication components. Methods of ARC deposition will be discussed along with final component performance and the identification of key process parameters affecting product performance.

Introduction

Through the use of fiber optic connections and components to transmit information signals via light, the given bandwidth and rate of communication of a system can be greatly increased compared to traditional wired connections. However, system complexity and material constraints can restrict the development of such optical systems. Novel additive manufacturing (AM) techniques offer a promising means to implement these fiber optic components in a versatile platform [1]. Through these techniques, optical connections can be deposited directly into complex systems, where traditional manufacturing methods would require changes to the existing system.

In order to facilitate the further design and development of novel fiber optic connections, methods of depositing anti-reflective coatings (ARCs) must be established. In traditional fiber optic systems, ARCs limit the loss of light at junctions due to either undesired feedback or emission into the environment [2,3]. These traditional systems typically utilize coatings of magnesium fluoride deposited through atomic layer deposition (ALD) [5,6]. In ALD, the coating precursors are deposited alternately from the gas phase with self-terminating reactions to form thin, uniform layers. However, ALD typically produces a conformal coating without spatial selectivity, and would expose the entire system assembly to the coating rather than only the connectors. To limit this system wide exposure to the coating material being utilized, a more targeted, conformal process of deposition must be established.

High performing ARCs, must meet a strict geometric profile. Primarily, the films must be of such a thickness that they satisfy the following condition in order to properly prevent the reflection of a given wavelength of light:

$$d_{ARC} = \frac{k \cdot \lambda}{4 \cdot n_{ARC}} \quad (1)$$

in which d_{ARC} is the thickness of the ARC film, λ is the wavelength of light of interest, n_{ARC} is the index of refraction of the film material, and $k=1, 3, 5$, etc [3]. Therefore, it is critical to the overall performance of the film that the thickness is as close to the calculated value as possible.

Experimental

A. Equipment

In order to establish a process for depositing controlled layers of aerosolized coating material onto a controlled substrate, it was necessary to either identify or establish a technology capable of depositing materials with high spatial resolution while mitigating flow out of the intended deposition area and overspray onto the substrate. Additionally, due to the critical nature of final film geometry, such a process would also need to deposit materials into layers of extremely precise thicknesses. It was due to these demands that an IDS NanoJet (NJ) system was utilized for this investigation, as it had the ability to produce films from low viscosity inks with a thickness resolution as fine as 50 nm [7].

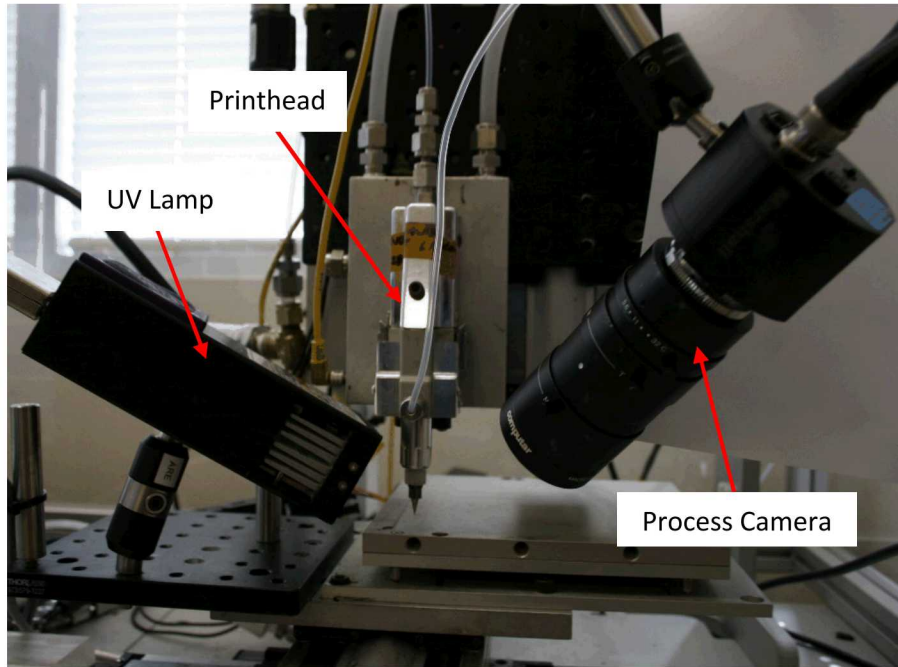


Figure 1. Photograph of the NanoJet system used in this study. The system features a Marshall compact 2MP HD-SDI camera for process monitoring, a 1.5-Watt FireFly FF200 UV lamp for soft curing of the material, and the printhead to house, aerosolize, and deposit the materials of interest.

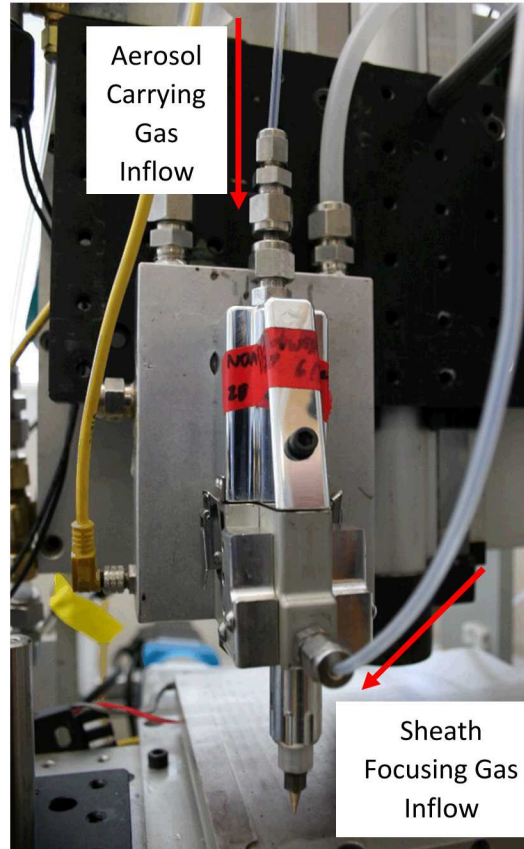


Figure 2. Close-up photograph of printhead. Inflows for both the aerosol carrying gas and sheath focusing gas are designated on the image.

In the NanoJet process, a liquid ink is aerosolized with an ultrasonic atomizer within the printhead. The aerosolized material is then transported via a carrier gas to the main flow channel of the printhead. In this main flow channel, an annular focusing gas is introduced to accelerate and collimate the aerosol stream. Due to the importance of the two gas flows and their control over the rate at which the aerosolized material is deposited, either flow rate can be altered to adjust the rate at which material is deposited.

B. Materials

The primary ink used to generate these coatings was a solution of isobutyl acetate and a commercially available UV curable optical adhesive (Norland Optical Adhesive NOA 89), and the substrate was a silicon wafer. Two different inks were tested with varying concentrations, the first containing 14:1 by volume isobutyl acetate/NOA 89 and the second with a 20:1 ratio by volume. These specific mixing ratios were selected for two primary reasons, to reduce the viscosity of the base optical adhesive and therefore promote atomization, and to reduce the concentration of the optical adhesive to more accurately control the thickness of each deposited layer, as less of the film material would be deposited per pass of the tool head. The resultant improvement in thickness resolution would enable broader tunability of films for different wavelengths. In both inks, the isobutyl acetate was believed to be of sufficiently high volatility so as to completely evaporate from the solutions following atomization or very rapidly upon impacting the substrate, leaving only the optical adhesive.

C. Process Parameters

During the deposition process, a cross-hatched toolpath was used that alternated depositing the material along the x and y-axes in subsequent layers. Such cross-hatching was used to prevent the development of unintended flow and grain development in the films across multiple layers. Additionally, the target thickness of each film was dictated by the wavelength of light for final applications, and this was adjusted by varying the total number of layers printed.

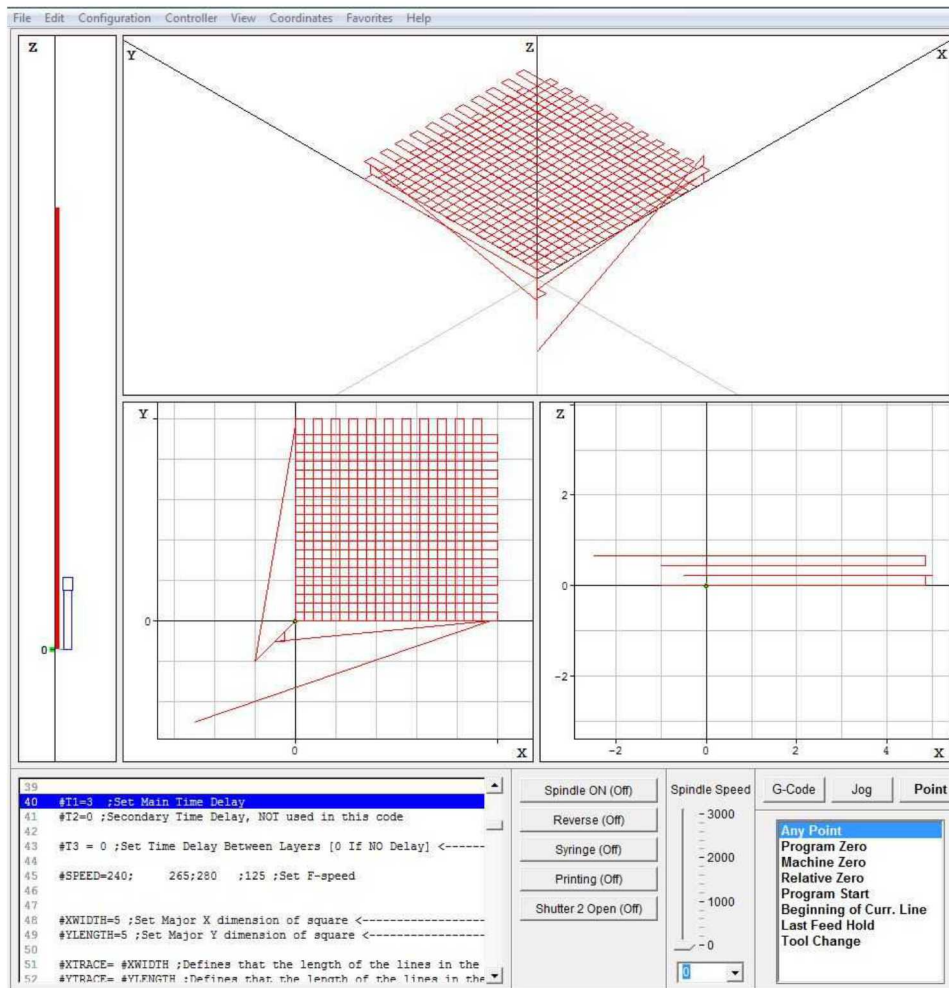


Figure 3. Screenshot of the toolpath generated to deposit the film material in a uniform geometry of controlled thickness.

During deposition, compressed air was used for the aerosol carrier gas and the sheath focusing gas, with flow rates of 5.5 sccm and 10.0 sccm, respectively. The atomizer voltage was set to 37 V, and the substrate was heated to 60 °C. Finally, throughout the deposition of the film, a 1.5-Watt FireFly FF200 UV lamp was run at 40% power to soft cure the material as it was deposited. After each layer, the lamp was also allowed to cure the completed layer for 180 seconds before the next layer was deposited.

Depending on the concentration of the material being deposited, two additional variables were altered. For material mixed at a 14:1 concentration, the toolpath was run at a feed rate, or

print speed, of 265 mm/min, with an infill space between adjacent lines of 0.210 mm. For material mixed at a 20:1 concentration, a feedrate of 240 mm/min was used, with an infill space between adjacent lines of 0.220 mm.

Isobutyl Acetate:NOA 89 Concentration	14:1	20:1
Print Speed (mm/min)	265	240
Infill Spacing (mm)	0.210	0.220
Aerosol Flow Rate (sccm)	5.5	5.5
Sheath Flow Rate (sccm)	10.0	10.0
Atomizer Voltage (V)	37	37
Substrate Temperature (C)	60	60
UV Lamp Output (% output)	40	40
Time Delay Between Layers (sec)	180	180

Table 1. Process parameters used to deposit each material mixture in the investigation. Only the print speed and the infill spacing were varied depending on the material.

Once fully deposited, each film was cured under an ABM, Inc Exposure System at 500 W for 20 minutes to achieve a final, full cure. Beyond the heated platen during deposition, no temperature based curing or treatment was applied to the material. The primary purpose of the heated platen was to ensure the complete and rapid evaporation of the ink solvent.

Results and Discussion

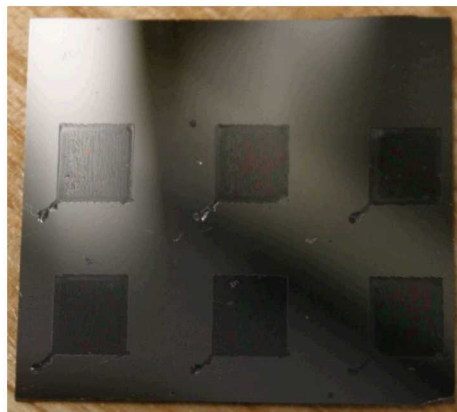


Figure 4. An image six test films (5mm X 5mm) of the 14:1 concentration ink deposited on a silicon wafer substrate.

A. Film Thickness and Layer Resolution

This section will contain profilometry data collected from films produced with each mixture of materials. This data will not only be used to discuss the overall thickness, uniformity of thickness, and surface roughness of films produced, but will be used to calculate the average thickness per layer in order to quantify the resolution of the process.

B. Film Performance and Absorption of Near-Infrared Light

This section will contain data collected via spectroscopic ellipsometry, for both mixtures of materials. The data should pertain to the resultant index of refraction of the films and their absorption of near-infrared light (850 nm). The results of this data will then be used to discuss the overall performance of the films and their suitability for implementation in fiber optic systems.

Conclusions

To develop additive manufacturing methods suitable for optical systems, the presented work has aimed to identify materials and processes to fabricate anti-reflective films. By varying the ink composition and printing parameters, control over film thickness with high resolution was investigated. With precise control of film thickness, the overall performance of AR films and spectral characteristics can be tailored, with possibilities for more complex designs providing broadband AR functionality. The ability to pattern such films with versatile, additive printing methods and high spatial resolution offers numerous advantages for advanced manufacturing of optical systems. Material and methods from this work are readily adaptable and provide insight for implementation in additively manufactured fiber-optics.

Acknowledgements

Thank you to the following people for assistance and input in this work: Brain Kaehr, Matthew Roach, Seth Johannes, Chase Kayser, and Rick Adcock.

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.

References

1. Wu, L.; Dong, Z.; Li, F.; Zhou, H.; Song, Y., Emerging Progress of Inkjet Technology in Printing Optical Materials. *Advanced Optical Materials* **2016**, *4*, 1915-1932.
2. Moayedfar, M.; Assadi, M. K., Various Types of Anti-Reflective Coatings (ARCs) Based on the Layer Composition and Surface Topography: A Review. *Reviews on Advanced Materials Science* **2018**, *53*, 187-205.
3. Hedayati, M. K.; Elbahri, M., Antireflective Coatings: Conventional Stacking Layers and Ultrathin Plasmonic Metasurfaces, A Mini-Review. *Materials (Basel)* **2016**, *9*.
4. Li, X.; Yu, X.; Han, Y., Polymer thin films for antireflection coatings. *Journal of Materials Chemistry C* **2013**, *1*.
5. Johnson, R. W.; Hultqvist, A.; Bent, S. F. A brief review of atomic layer deposition: from fundamentals to applications. *Materials Today* **2014**, Volume 17, Issue 5, 236-246.

6. Pilvi, T.; Puukilainen, E.; Kreissig, U.; Leskelä, M.; Ritala, M. Atomic Layer Deposition of MgF_2 Thin Films Using TaF_5 as a Novel Fluorine Source. *Chemistry of Materials* **2008**, 20 (15), 5023-5028
7. Essien, M. Apparatuses and Methods for Stable Aerosol Deposition Using an Aerodynamic Lens System. US Appl. 14/927380, 2016.