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#### Abstract

Gas phase solid freeform fabrication research at The University of Connecticut focuses on two main procedures, Selective Area Laser Deposition (SALD) and Selective Area Laser Deposition Vapor Infiltration (SALDVI). A SFF research laboratory is under construction at UCONN, with two new operation systems. These systems possess temperature control, data acquisition capabilities, *in-situ* video monitoring, and the ability to fabricate SALDVI parts up to four inches wide by four inches long. The procurement of a harmonic generating Nd:YAG six watt laser, capable of producing output at 532, 355, and 266 nanometer wavelengths, as well as a coupled effort with the Photonics Center at the University providing laser diodes at a variety of wavelengths, presents the opportunity to explore interactions involved in gas reactions driven by lasers. Investigations of material systems will include ceramic carbides, nitrides, and their composites, as well as metals.

# Selective Area Laser Deposition (SALD)



Figure 1--Schematic of SALD

## Introduction

SALD<sup>1-4</sup> and SALDVI<sup>5</sup> are two gas phase SFF techniques that utilize local chemical vapor deposition (CVD) to create shapes and structures in an additive manner. Both form solid deposition products from the decomposition of gas precursors by a laser beam in an environmentally controlled vacuum chamber. Because the decomposition is localized around the beam spot area, the size and shape of the solid reaction product can be manipulated by computer-operated scanning of the laser beam. The differences between the two procedures involve the interplay between the solid product and the substrate upon which deposition takes place. SALD merely uses the substrate as a base for beginning deposition on, and shapes are created from consecutively scanned layers. In SALDVI, the solid product infiltrates a thin layer of powder at the substrate, filling the porous spaces between powder particles. Once a powder layer has been scanned and infiltrated, a fresh powder layer is spread out over the previous one and the infiltration process begins again. Figures 1 and 2 represent the SALD and SALDVI processes, respectively. An example where SALD and SALDVI has been performed is with tetramethylsilane as the gas precursor for silicon carbide deposition, according to the following equation:

*thermal energy* Si(CH3)4 (g) -----> SiC (s) + 3 CH4

(g)





# Figure 2--Schematic of SALD

where carbon is also unintentionally deposited by decomposition of the methane by-product.

## SFF Laboratory

At present, the SFF laboratory entails two systems, a larger 15 inch tall by 28 inch diameter water-jacketed, stainless steel vacuum chamber and a smaller 8 inch tall by 8 inch diameter stainless steel vacuum chamber. The larger 15 inch tall system encloses a powder delivery system (PDS) for performing SALDVI experiments. The smaller chamber is used for SALD experiments. The main components of the PDS are a supply piston, a target piston, a roller, and three motors with encoders. The roller spreads a controlled amount of powder from the supply piston to the target piston when cued by the computer. Shapes up to 4 inches by 4 inches by 4 inches in size can be fabricated in this system. Each system has a custom built laser stand capable of supporting any one of the lab's three a 50W CO<sub>2</sub> (10.64 µm wavelength), a lasers: 150W Nd:YAG (1.06 µm wavelength), and a harmonic generating Nd:YAG with a  $\overline{6W}$  output at the primary 1.06 µm wavelength, as well as various diode lasers. Two computer controlled -xy stages provide the motion to generate the 2-D scan in each layer. In the larger system, the chamber and the laser are stationary, and the stage moves the laser beam by translating the optics. The smaller chamber rests on the -xy stage and moves under a stationary laser beam. An optical pyrometer based on a two head design with a range from 200 to 1500°C monitors the deposition temperature and also provides feedback for closed loop temperature control by adjusting the laser power. A video camera provides *in-situ* visual monitoring and recording of the deposition process. A gas delivery manifold delivers multiple gas precursors to the reaction chambers, operating presently in a static gas mode. A photograph of the large system and hood is found in Figure 3.

#### **Experiments**

To demonstrate the laser scanning capability of the larger system, two shapes were deposited using the SALD process. Two different scanning programs produced silicon carbide deposits in the shapes of a disc and a rectangle. The operating conditions for deposition included 12.5 watts of power from a CO<sub>2</sub> laser, a 2 millimeter diameter beam spot, 75 torr of tetramethylsilane gas precursor, and an approximate scan speed of 1 millimeter per second. Each scanning program was repeated ten times to yield multilayer deposits. Figure 4 presents a 500 X magnification SEM micrograph of the 10 layer rectangular scan sample. Based on an estimated laser spot dwell time of 6 seconds at points in the layer, a growth rate of 50 microns per minute was calculated. This growth rate calculation assumes an adequate deposition temperature (*hot spot*) across the entire 2 millimeter diameter laser beam, with a roughly circular beam shape.

A third SALD deposit was produced using the novel approach of a diode laser for the energy source. This diode laser is a McDonnell Douglas #ISO-550, operating at 808 nanometers and producing 50 watts continuous wave(cw) and 600 watts pulsed maximum output. The diode laser experiment, run under a static motion condition, produced a circular deposit with some infiltration into the powder substrate. The growth parameters included 15 watts cw laser power for 5 minutes, 75 torr of tetramethylsilane gas precursor, and an approximate 5 millimeter beam spot diameter with a gaussian distribution.



Figure 3--Workstation in SFF Lab at UCONN



Figure 4--SEM micrograph of 10 layer SiC SALD deposit

#### Future Work

A variety of experimental areas in the SALD and SALDVI arena will be addressed. The control of the composition of material deposition will be a major factor in the silicon carbide foundry concept. The goal of the foundry is to deposit silicon carbide with *in-situ* electromechanical devices. Other investigations will focus on the role of laser wavelength in the gas precursor decomposition. Attempts will be made to couple certain laser wavelengths to specific material system excitations in order to produce partial photolytic, partial thermal decomposition mechanisms. Finally, efforts to work with different material systems will involve boron nitride, using boron tribromide and boron trichloride precursors, and metals such as titanium and cobalt, along with various ceramic/ceramic and ceramic/metal composites.

#### Conclusions

The Solid Freeform Fabrication laboratory at The University of Connecticut has been under assembly and construction for much of the past year. Experimental work in gas-phase SFF recently began, and emphasis will be placed on the SALD and SALDVI processes. Process parameters, process interactions, and a variety of material systems will be under investigation under a closed loop, feedback controlled system. The SFF program at UCONN maintains a World Wide Web site at: http://www.ims.uconn.edu/~hmarcus.

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#### References

1. G. -S. Zong, R. Carnes, H. G. Wheat, and H. L. Marcus, "Solid Freeform Fabrication by Selective Area Laser Deposition", <u>Proceedings of the Solid Freeform Fabrication Symposium</u>, edited by Joseph J. Beaman, Harris L. Marcus, David L. Bourell, and Joel W. Barlow, The University of Texas at Austin, Austin, Texas, August 6-8, 1990, pp. 83-90.

2. J. L. Maxwell, J. Pegna, and A. G. Ostrogorsky, "Thermal Analysis and Modeling of Steady-State Rod Growth During Gas-Phase Solid Freeform Fabrication", <u>Proceedings of the Solid</u> <u>Freeform Fabrication Symposium</u>, edited by Harris L. Marcus, Joseph J. Beaman, Joel W. Barlow, David L. Bourell, and Richard H. Crawford, The University of Texas at Austin, Austin, Texas, August 9-11, 1993, pp. 253-270.

3. F. T. Wallenberger and P. C. Nordine, "Strong, Pure, and Uniform Carbon Fibers Obtained Directly from the Vapor Phase", *Science*, vol. 260, pp. 66-68, 2 Apr. 1993.

4. O. Lehmann and M. Stuke, "Laser-driven Measurement of Three-dimensional Microstructures Generated by Laser Rapid Prototyping", *Science*, vol. 270, pp. 1644-1646, 8 Dec. 1995.

5. B. R Birmingham and H. L. Marcus, "Silicon Carbide Shapes by Selective Area Laser Deposition Vapor Infiltration", <u>Proceedings of the Solid Freeform Fabrication Symposium</u>, edited by Harris L. Marcus, Joseph J. Beaman, Joel W. Barlow, David L. Bourell, and Richard H. Crawford, The University of Texas at Austin, Austin, Texas, August 8-10, 1994, pp. 348-355.