Layered Micro-Wall Structures from the Gas Phase

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Abstract: *The use of 3-D LCVD with volumetric rate feedback was investigated in the fabrication of micromechanical wall structures. These were constructed by recursive laser scanning and resulted in layered wall composed of recursive line deposition.*

Experiments were designed to uncover the relationship between scan rate, volumetric deposition rate, pressure and laser power for pyrolytic graphite from an ethylene precursor. Results point to a conduction dominated heat transfer which greatly limits the volumetric deposition rate at the wall. This also results in a highly unstable deposition process, since volumetric deposition increases by orders of magnitude as soon as rod growth is initiated.

An unexpected results of this work is the ability to grow rods at an angle to the laser axis, with good control of the linear growth rate. This is achieved by adaptive laser scanning during rod growth.

Keywords: *3-D LCVD, SALD, Process Control.*

1 INTRODUCTION

Our interest is to develop 3D-Laser Chemical Vapor Deposition (3-D LCVD) into a free-form fabrication tool from the micro-scale (0.1μm) to the macro-scale (cm). As such, 3-D LCVD offers a micromechanical complement to SALD and SALDVI [1], potentially allowing single stage mesoscale fabrication.

To test the adequacy of 3-D LCVD for the deposition of layered structures, we have performed a series of experiments to investigate the practicality of making upright panels by direct deposition. Both open and close loop deposition of layered walls will be described in this paper. To some degree, this work is reopening the research pioneered by Zong [2], in which a small layered cube structure was deposited using SALD. The main contribution over Zong's work is the evaluations of various strategies for closed-loop feedback control of the deposition process.

For close loop control, a new method that utilizes a previously developed feedback mechanism [3] has been enhanced. Various control methods were attempted and some exhibit promise in the field of manufacturing at the scale of micromachines and beyond. An unexpected result of this investigation is that the method allows one to achieve control over the type of structure deposited so that the user has the option of building either a wall or an angular rod.

2 EXPERIMENTAL

2.1 Equipment

A schematic diagram of the Rensselaer 3D-LCVD system is shown in Figure 1 The 3D-LCVD reactor consists of a custom quartz tube with ports for viewing and laser input. The chamber is connected to a pumping station via a gate valve. The vacuum chamber and gas-delivery systems are enclosed within a ventilated hood for safety purposes. For the growth of pyrolytic graphite, 133 - 665 mbar (100-500 Torr) ranges of ethylene pressures were employed.

The beam source was a Coherent. model CR-18 argon ion laser with a maximum output of 12Watts (multi-mode) at the 488/514 nm primary lines. To vary the laser beam power, a liquid-crystal retarder and polarizing beam splitter were placed in series, allowing peak-to-peak power swings in under 200 ms. Incident powers reported herein represent total beam power at the deposit. Observation of the sample during growth and laser alignment is made with a custom-built short-focus telescope and CCD camera.

A photodetector, covered with narrow band filters, was mounted to one of the chamber windows, at a distance of roughly 200 mm from the sample. Using a two-decade pre-amplifier, the sensor could measure emissions as small as 0.25 mW at the substrate (at 656nm). The amplified signal was recorded by an Omega Nubus data acquisition system, with a typical sample period of 0.05-0.10 seconds, and was later time averaged as needed for real-time control.

At the core of the system is a precision five-degrees-of-freedom manipulator. This micromanipulator was designed to allow beam scanning not only in the plane of the substrate, but at any

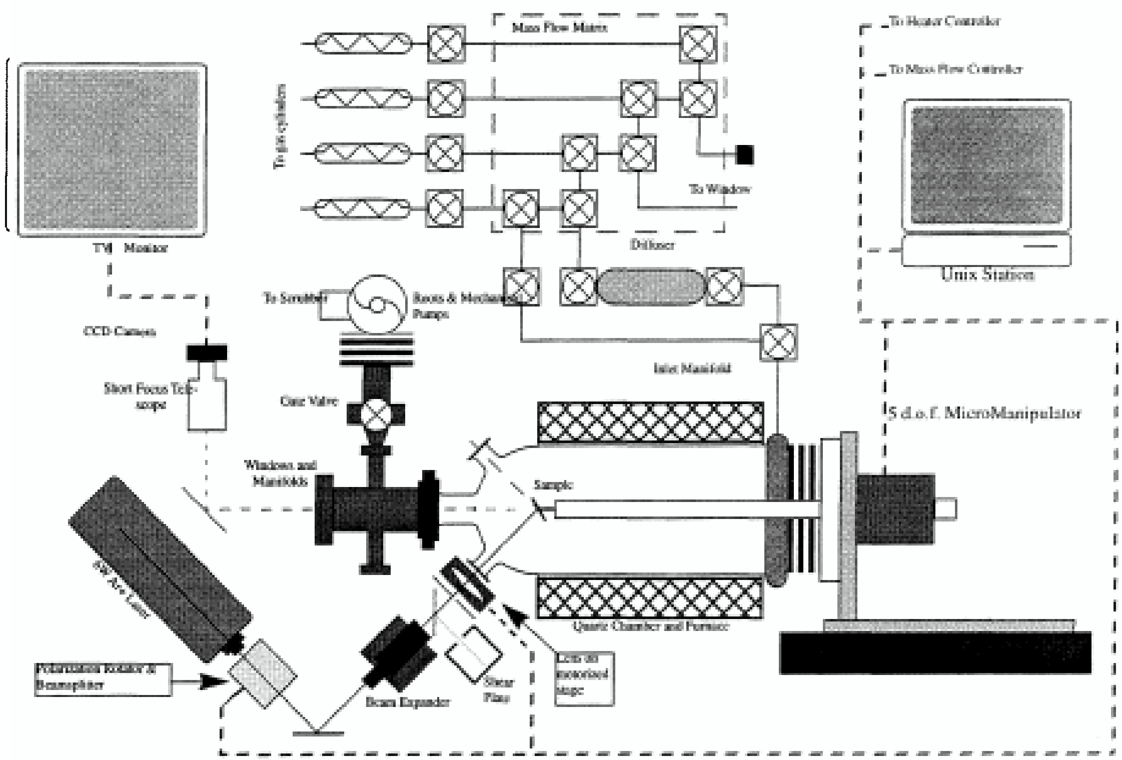


FIGURE 1. Schematic diagram of the 3-D LCVD reactor.

angle and orientation to the sample. This is effected through a computer-driven 5-axis micromanipulator. This tool allows sub-micron positioning with stepping motor control from outside the vacuum chamber. The translational and angular resolutions of this micro-manipulator are 1 μm and 0.005° at the full step control mode.

The manipulator arm holds the sample within the chamber. The manipulator attaches to the vacuum chamber via a flexible bellows to limit vibrations transmitted to the sample. Both the manipulator and the laser lie on a vibration-isolated table, their relative position is fixed.

2.2 Feedback Techniques

Various feedback techniques were attempted in order to determine the best approach:

1. The sum algorithm method: The volumetric reaction rate —as measured by the technique of Maxwell et al. [3]— is discretely integrated until it reaches a predetermined value. The sum is then reset and the appropriate stepper motor advances a given distance.
2. The threshold method: The reaction rate is monitored until it reaches a preset value. The appropriate stepper motor advances a given distance.
3. The volumetric deposition method: The feedback control unit attempts to maintain a reference emission value for constant scanning speed by adjusting the laser power with the LCD polarizer.

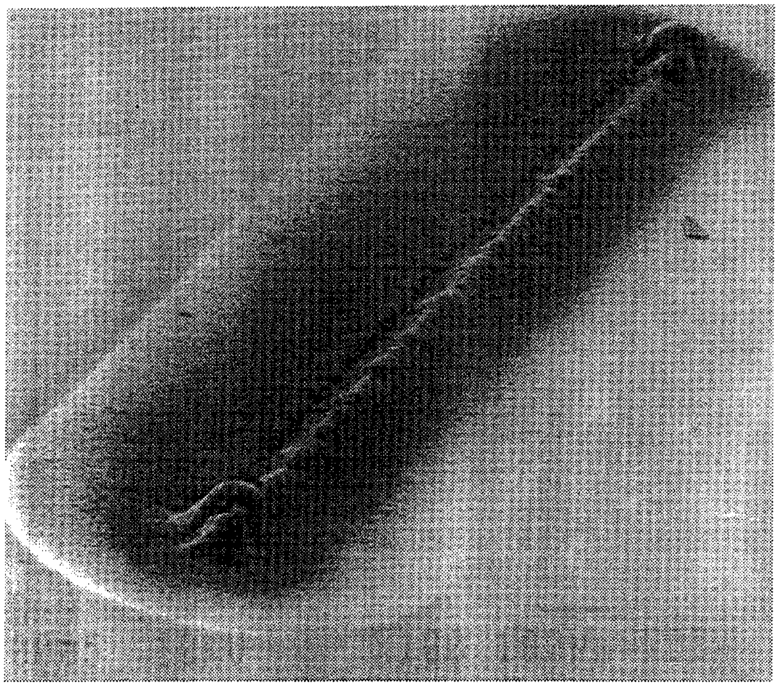


FIGURE 2. Sample linear scan results. Precursor: Ethylene at 200 Torr. Laser power: 5.6 Watts. Scanning Speed: 200μm/s.

FIGURE 3. Sample scan results along a close contour. Precursor: Ethylene at 150 Torr. Laser power: 5.6 Watts. Scanning Speed: 200μm/s.

3 RESULTS AND DISCUSSION

3.1 Open-Loop Scanning

Figure 2 shows an attempt at building a 2mm long wall from ethylene without volumetric deposition control. The Ar+ CW laser was scanned at a constant speed of 200μm/s with a constant power of 5.6 Watts. The total number of passes (i.e. layers) was 100, with a 15 second delay (0 Watts) at the end of each pass. The promontories at both ends of the ridge are a result of the increased loitering time due to the directional change of the laser. The pressure was 200 Torr. The substrate was Ti-coated quartz.

In an attempt to reduce the promontory effect at wall's end, attempts were made at building walls along a closed loop cross-section. Figure 3 shows a 2x1.5 mm rectangular wall structure built by repeated scanning of the laser around the closed contour, with no volumetric deposition control. Mark the beading throughout the upper layers especially on the shorter sides of the rectangle. These beading are characteristic or the Arrhenius relation controlling the deposition rate and are the cause of instabilities in the growth. As we shall see, volumetric control does provide a slightly better result. The total number of passes was 125. The laser power was 5.6 watts. The chamber pressure was 150 Torr. The scan rate was 200 mm /s.

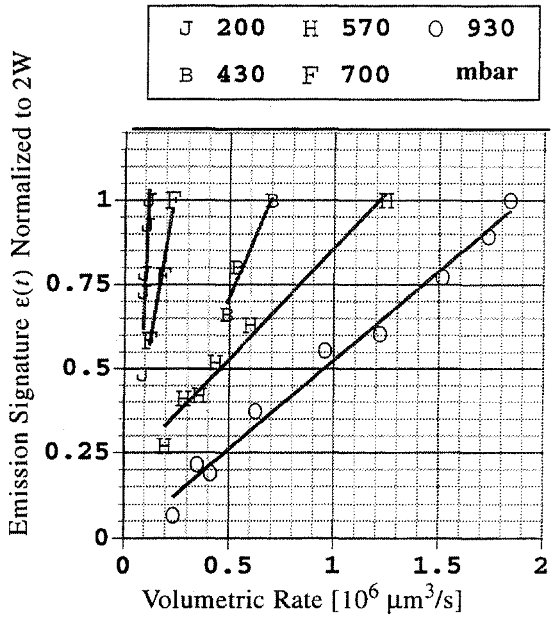


FIGURE 4. Correlation of steady state 656 nm emission intensity to volumetric deposition rate (from [3]).

The most noticeable result about open loop deposition is the poor quality of the deposit shape and the slow deposition rate associated with scanning speed 200μm/s. Open loop attempts performed at lower scanning speeds result in spurious local rod growth and an even more irregular deposit. In the next section, we shall monitor the volumetric deposition rate for various constant low scanning speeds.

3.2 Emission Calibration

One of the fundamental results obtained by Maxwell et al. [3] was the correlation of hydrogen by-product emission (at 656 nm) to the volumetric deposition rate. This linear relationship is captured in Figure 4, which provides us with emission (Volts) to volumetric rate ($\mu m^3/s$) conversion coefficients.

3.3 Volumetric deposition rates at low scanning speeds

In an effort to increase the volumetric deposition rate, the scanning speed must be significantly lower than the 200μm/s used in Section 3.1. The main problem with lower speeds, however is that the deposit growth tends to be extremely unstable. Experiments were conducted to monitor volumetric deposition rates at various scanning speeds ranging from 2 to 8 μm/s and pressures ranging from 100 to 440 Torrs.

Low pressure experiments, illustrated by Figure 5, were conducted with a constant laser power of 4.8 Watts for carbon deposit from ethylene onto a quartz substrate. We know from Maxwell et al. [3] that at the pressures of 100 and 150 Torrs, the axial growth rate of the deposit is below 2μm/s and reaches that bound for pressures of the order of 200 Torrs. It is interesting to note that at both 100 and 150 Torrs, the volumetric deposition rate remains relatively constant at scanning speeds over 2μm/s. In all instances, the deposit appears to be somewhat similar to a rod laid flat on the substrate, as denoted by the noticeable speed transitions on the deposit. At the upper pressure of 150 Torr, as the scanning speed becomes of the same order as axial growth, note that explosive volumetric growth leads to rod growth. The combined increased axial growth and scanning speed in this instance lead to a rod growing at an incline.

A series of experiments were conducted to determine the transition between line deposition and rod growth. A series of scanning deposition at constant laser power was conducted, while the scanning rate was decreased from 8μm/s to 1μm/s every 2μm. These experiments were conducted for different pressures. The transition scanning rates for different pressures are summarized in Table 1.

Pressure (Torr)	Transition Scan Rate (μm/s)
500	10
400	8
350	8
300	6
250	6
200	4
150	2

TABLE 1. Line to rod growth transition scan rate for pressures ranging from 500 to 150 Torr.

3.4 Closed-Loop Scanning

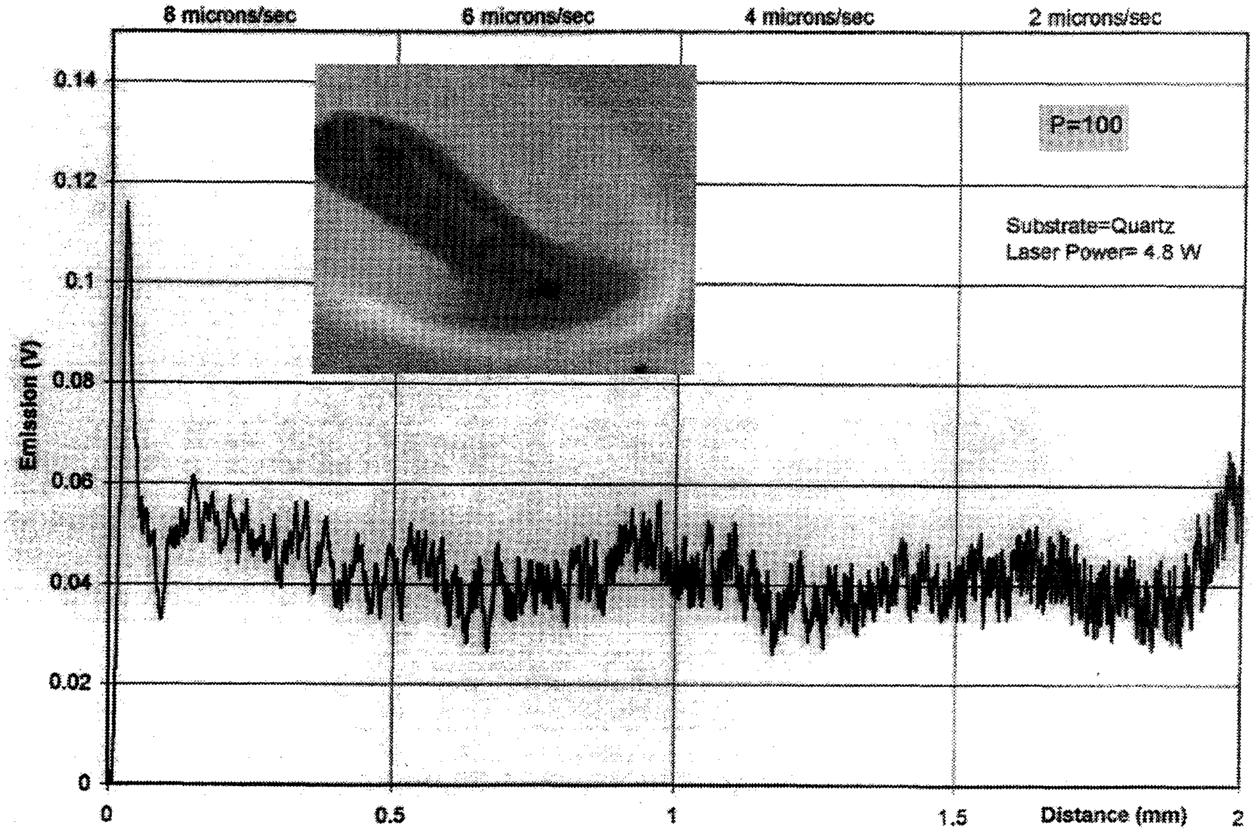
Among the various feedback control methods experimented, both the threshold method and the volumetric feedback proved more useful at building rod structures.

For walls, it was found that the sum algorithm gave best results for building a wall structure. Figure 6 shows three views of a wall constructed using the sum algorithm. The reaction rate was integrated until it reached a set value (.05), which triggered a 10 μm step. Notice that using the feedback control, only 30 passes were needed to build a nearly 1mm high wall. This compares to 125 passes in Figure 2 to build a structure 20μm high, and already exhibiting instabilities. The structure exhibits a Pan flute formation but all of the members are fused together. This picture shows the potential of the sum algorithm method in obtaining an extended, relatively flat, and vertically developed structure. The pressure was 220 Torr. The laser power was 5.8 Watts. The length of the sample is 1 mm. This method appears to be the most promising for forming elevated panel structures. However, the structure still exhibits large irregularities in height and cross-section.

Emission measurements were recorded during wall growth and averaged for different height ranges. These measurement corroborate an average volumetric growth rate of the order 0.5μm^3/s. The results are shown in Figure 7. Note that most irregularities in deposition rate occur at the substrate. Once wall growth has commenced, volumetric deposition rates seem to reach a steady state.

4 CONCLUSION

In this work we investigated the layered fabrication of micro-wall structures by direct deposition using 3-D LCVD. The feasibility of this construction was demonstrated by fabrication of sample vertical panel structures 1mm x 1mm. These panels were obtained using closed-loop feedback control of the reaction. By product emissions were used as direct measurement of the volumetric deposition rate and integrated until a set volume was reached, upon which the laser focus was stepped by 10μm.



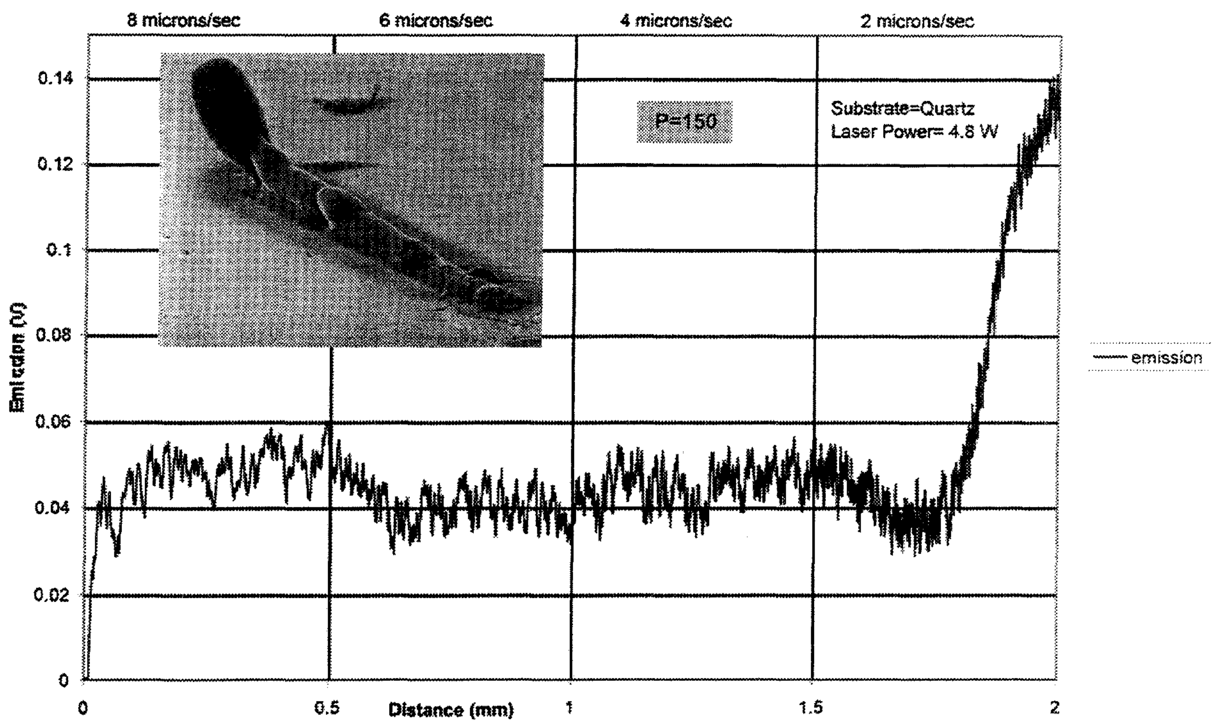


FIGURE 5. Low pressure emission / Volumetric deposition rate for scanning speeds ranging from 8 to 2 µm/s for carbon deposit from ethylene at 100 Torrs (top) and 150 Torrs (Bottom).

While closed loop control has proved vastly superior to open loop in these experiments, it remains that the structures fabricated exhibit large shape irregularities; mostly due to the instability of the deposition process. In addition deposition rates are hindered by the layered nature of the fabrication as opposed to rod growth, which exhibit volumetric rates an order of magnitude larger [6].

In the process of investigating the parameters and the conditions for wall growth it turned out that some rods were formed instead. As it turns out, scanning the laser focus during rod growth allows for a new method of forming angled rods similar to that demonstrated by Bauerle [4], and Lehmann and Stuke [5]. The results of this work are reported in a companion paper [6]. This process has the particular advantage that the laser remains perpendicular to the substrate at all times, simplifying the geometry of construction. It also features near optimal volumetric deposition rates as compared to layered growth.

It is therefore the conclusion of this research that 3-D LCVD is ill-suited for layered fabrication of volumes greater than $10^6 \mu m^3$. Alternative uses of the process however appear very promising in pursuit of tessellated (rod-based structures). This remark opens a new approach to freeform fabrication, away from layered manufacturing.

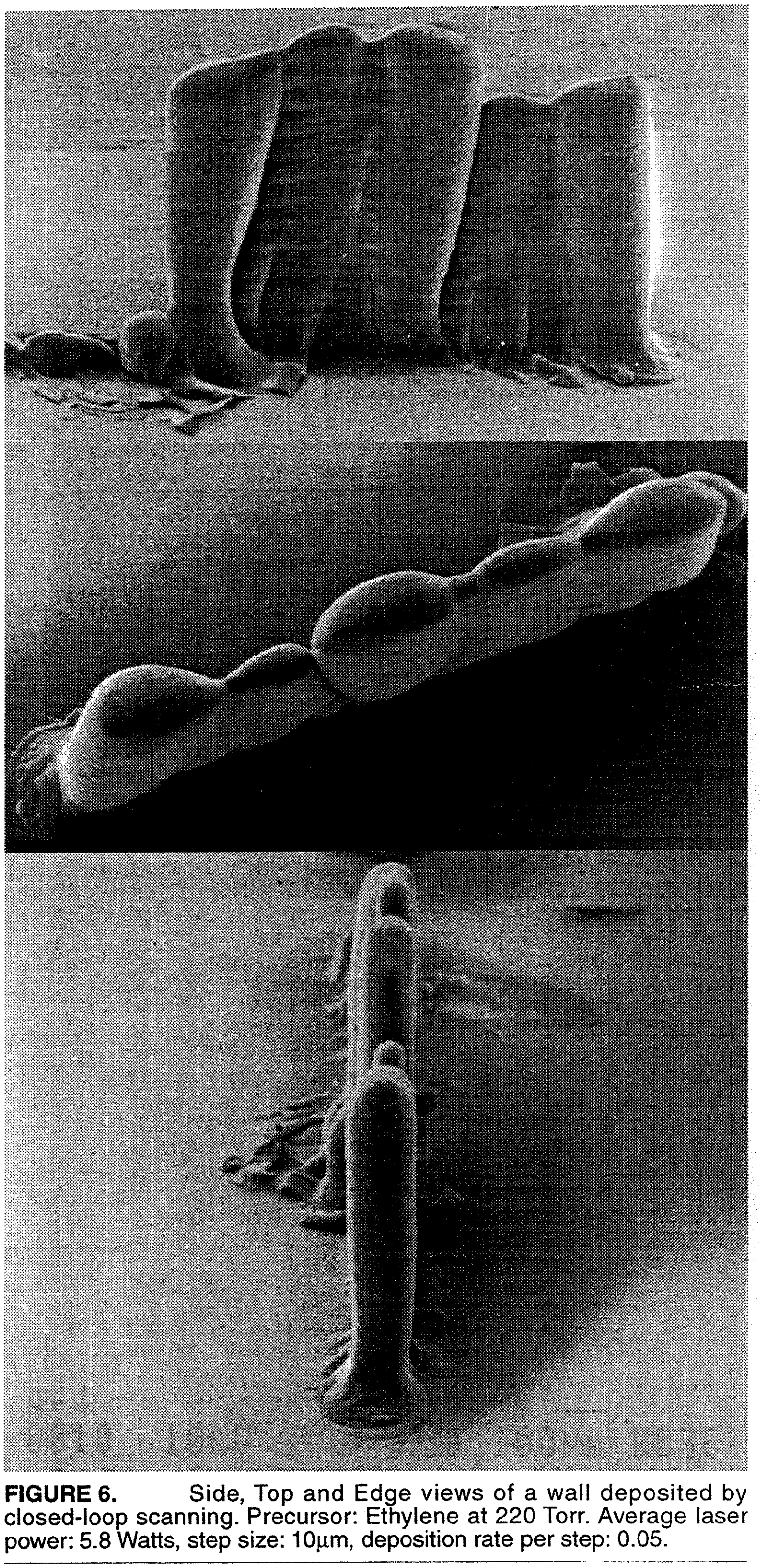


FIGURE 6. Side, Top and Edge views of a wall deposited by closed-loop scanning. Precursor: Ethylene at 220 Torr. Average laser power: 5.8 Watts, step size: 10μm, deposition rate per step: 0.05.

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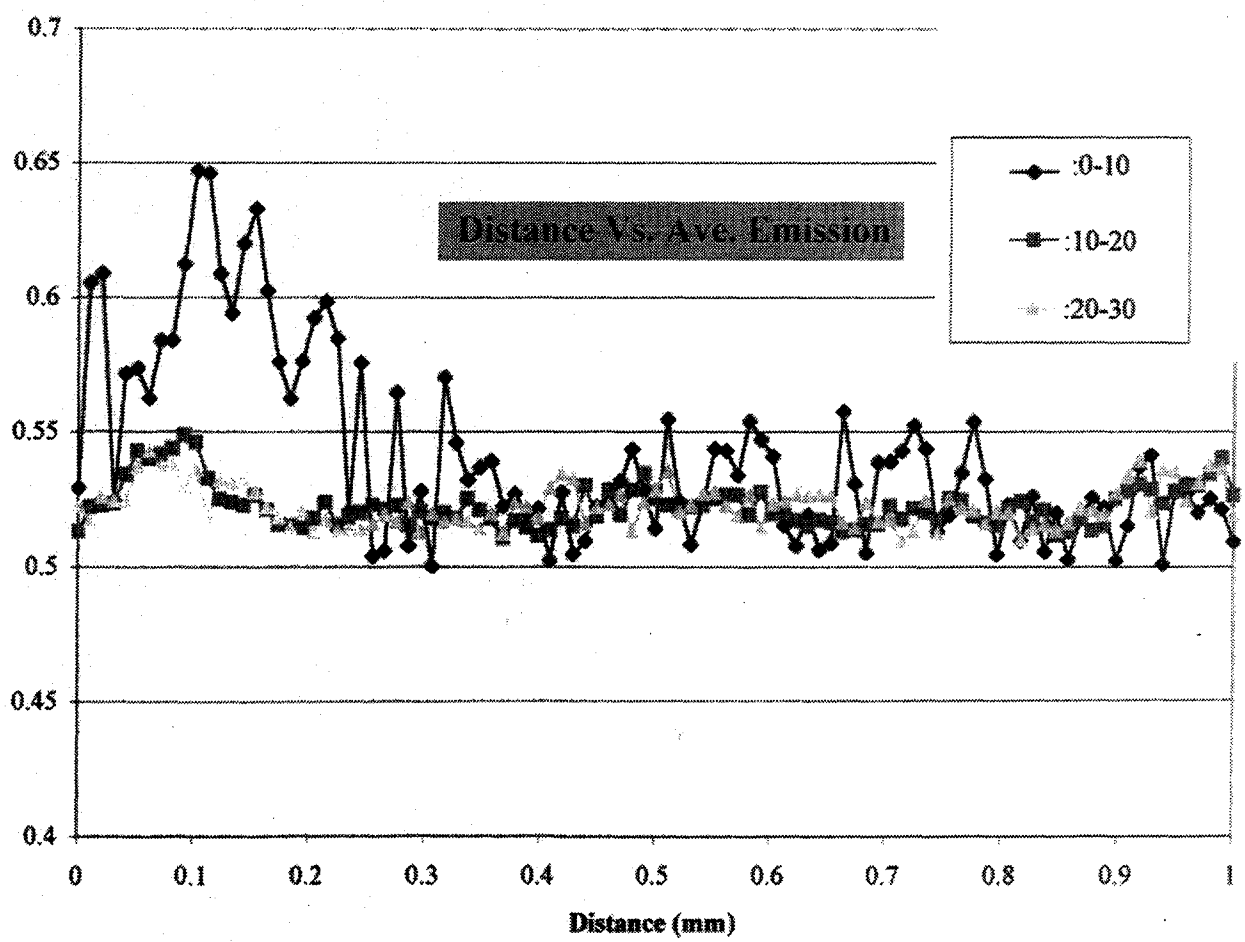

FIGURE 7. Averaged emission measurements for 3 height ranges during wall growth.

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