A Low Pressure Organic Vapor Phase Deposition Chamber

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Abstract— In this paper we present a low Cost, low pressure Organic Vapor Phase Deposition chamber. The chamber allows multilayer deposition of organic materials in a same batch. The chamber was made on a very low budget, considering similar systems used in micro component fabrication. Anthracene, an organic material, was deposited onto Indium tin oxide (ITO) coated glass to test and characterize the chamber. Depositing was proved and analyzed regarding uniformity, adhesion and thickness.

I. INTRODUCTION

Silicon based circuits has revolutionized the electronic industry and enabled mass production of electronic devices. New vast markets have been created based on the electronic devices and gadgets. Today almost every human being around the world possesses one or more gadget containing silicon based electronic such as computers, cell phones, mp3 players, toys and etc. This requires huge amount of processed and purified silicon. Silicon itself is a material that can be regarded as environmental friendly, but purifying it requires large amount of energy and complex chemical processes with toxic waste. In addition, silicon based electronics contain metallic and non-metallic substances which can be harmful to the environment. As the world becomes more aware of the vulnerable environment, the effort for minimizing the environmental challenges related to silicon based electronics has increased. Efforts such as making the processing of silicon more environmental friendly or looking for other materials for replacing silicon in circuit production. One approach is use of organic materials as substitute for silicon in production of active circuits [1]. Researchers around the world are developing new organic materials and more efficient fabrication technologies for production of organic based electronic devices. Organic Vapor Phase Deposition (OVPD) [2] has been demonstrated to be an effective deposition technology for high-performance thin-film active devices, such as Organic Field Effect Transistors (OFET) [3], Organic Light Emitting Diodes (OLED) [4, 5] and Organic Photo Voltaic (OPV) [6]. What makes this technique suitable for industrial applications is that it combines advantages of a similar technique, namely Vacuum Thermal Evaporation (VTE) [7] together with low temperature which gives good process control, along with a large choice of material which can give potentially very high through puts. Today, a significant part of all surface mounted electronic devices, especially optoelectronic devices, are made by using various types of Vapor Phase Deposition (VPD) techniques. In VPD the material which is later to be deposited, is first introduced to the system in a solid phase. Once placed inside a low pressurized sealed chamber, the material is heated to the point of evaporation. In a VPD system, it is desirable that this transition goes straight from solid to vapor, without going through the liquid state. Such a phase transition is called sublimation which is achieved by involving low pressure and elevated temperature. Once the phase transition is completed, the gas molecules are lead to the substrate by a hot inert gas. This approach can also be used for deposition of small molecule organic materials and called Organic Vapor Phase deposition (OVPD).

II. THE OVPD FURNACE

The schematic of the OVPD system presented in this paper is shown in figure 1. Each of the parts shown in the schematics are seen as vital components in order to obtain a deposition of high quality.



Temperature sensor

Fig. 1. Schematic of gas delivery system flow: 1) carrier gas source. 2) adjustable manifold. 3) MFC. 4) furnace. 5) gas heaters. 6) evaporation chambers. 7) manifold. 8) deposition chamber.

A. Gas flow

Figure 1 can be explained by following the gas flow in the gas delivery system [8]. It starts at the source of the carrier gas (no.1), which is connected to a manifold (no.2). This manifold have adjustable outputs, which together with the Mass flow Controllers MFCs (no.3) controls the flow of gas going into each of the evaporation chambers (no.6). Having two levels for adjusting the gas flow makes it easier for fine-tuning, as the velocity of the flow for this machine were set to be considerably low. Before the carrier gas reaches the evaporation chamber, it needs to be heated to the same temperature as the evaporated material. The carrier gas continues its path through the evaporation chamber where the sublimed material is located. From here, the sublimed material is transported further into a 5-to-1 manifold (no.7). The five inputs of this manifold are connected to each of the four (6.1-6.4) evaporation chambers plus one dilution line. From the output of the manifold, the gas flow containing the evaporated material is led out of the furnace and into the deposition chamber (no.8) where it passes a shower head before it is deposited on to a substrate. The dilution line is constructed the same way as the other flow paths, only there is no evaporation chamber connected. In figure 1 it is seen as the pipeline 5.3 in the middle of the five lines going through the evaporation chambers. It is used for better control of the growth rate by diluting the gas flow from the other evaporation chambers during deposition. Most of the system is placed inside a furnace (4) as a controlled temperature is a critical parameter for this type of systems. Only the control units such as Mass Flow Controllers (MFCs) and temperature regulators are placed outside the furnace, together with the deposition chamber.

B. Evaporation Chamber

The evaporation chamber is where the desired material for deposition will be introduced to the system. This chamber is crucial to the whole process of deposition considering this is where the sublimation part takes place, meaning that the material which is introduced as solid form goes a through one single-phase change from solid to vapor. The developed system holds four of these chambers that can be controlled individually regarding the flow rate and temperature. After the material is evaporated inside the chamber, it is ready to be transported by the carrier gas to the deposition chamber, which holds the substrate. Therefore the gas inlet and outlet of the chamber had to be placed in a way so the gas flow through the chamber as smooth as possible. Simulations of the gas flow were used to find the best solution. Figure 2 shows the simulation of the final design, with a velocity of 4 l/min. The gas input was placed on the top of the chamber close to the chamber-door. The inlet pipe going through the wall of the chamber from top was cut with a special profile in order to lead the gas in the right direction towards the outlet.

Figure 3 shows the final design of 4 chambers next to each other with sealed doors each mounted by wing 2 nuts. The chambers and doors were made using stainless steel.



Fig. 2. Simulation of gas flow through evaporation chamber with a resistive heater placed under it.



Fig. 3. Final design of 4 evaporation chambers placed next to each other

III. DEPOSITION CHAMBER

The deposition chamber is an important part of the system since it is where the deposition occurs. It was designed based on the schematic shown in figure 4 and placed outside the furnace. Important parts of the chamber are the Shower head and the water cooled substrate holder with adjustable distance to the shower head.



Fig. 4. Deposition Chamber

Many parameters effect the deposition properties such as thickness, uniformity and adhesion to the substrate. These parameters are gas speed and temperature, substrate temperature, distance to the shower head and the form of the shower head

[1, 11, 12].

A. shower head

If the shower head is designed and placed properly, it should ensure an uniform deposition. Without this component, the evaporated material will deposit unevenly, making the layer unsuitable for creating electronic circuit. The shower head is one of the most complicated to design, as it requires lot of calculation and simulation, not only because the ability of spreading the material as uniformly as possible, but also to obtain a high temperature as a part of the gas flow system. Figure 5 shows the designed shower head with internal heating to avoid deposition in the shower head.



Fig. 5. Shower head 3d drawings: 1) holes for attachment to top of deposition chamber. 2) sunken holes for standard screws to attach perforated plate and pipe. 3) grow for resistance wire. 4) grow and hole for thermocouple. 5) hole for pipe, carrying the evaporated material. 6) perforations for spreading the gas. 7) bottom side of top heat plate.

Figure 6 is the flow simulation of shower head. Based on these simulations we found the optimal form and distance between shower head and the substrate to achieve uniform deposition.



Fig. 6. Shower head simulation: (a) Streamline velocity field (b) Velocity magnitude measured in m/s

B. Substrate Cooling

Continuously cooling of the substrate and Ability of adjusting the distance from substrate to shower head was important to achieve an optimal deposition. Previous experiments states[9, 10] that the temperature of the substrate when depositing should be between 20-80 C. For better uniformity of the deposition and also to easier place the substrate on top of the cooling block, a stand that could be adjusted in the vertical direction was constructed. Lauda CP C6 is a compact thermostat that cools and circulates the water in a closed system.

The cooling system for the substrate is highly necessary for obtaining a good deposition. The surroundings of the evaporated material must have a temperature higher than the point of deposition. Thus the piping, shower head and also the surrounding air have to be between 250-350 C, depending on the material to be deposited. If the temperature drops to a point where the phase transition starts, the gas molecules may crystallize in the air or deposit on undesirable places inside the system.



Fig. 7. Full functioning OVPD Chamber

IV. DEPOSITION AND MEASUREMENT



Fig. 8. The structure of Anthracene, with 3 benzene rings

After the manufacturing and initial test of the OVPD furnace was finished, as shown in figure 7, we performed deposition tests to evaluate the systems performance. In our tests we used Anthracene as material to be deposit on different substrates. Anthracene is part of the grouping of chemicals called Polycyclic Aromatic Hydrocarbons (PAH). PAH is found in nature, and is created when products like coal, gas and other carbon based materials are burned, but the combustion process is not complete. Anthracene is commonly used for research purposes, because its characteristics are very similar to other materials in the PAH group. Anthracene can also be used by itself as an organic semiconductor. Chemically Anthracene is a PAH consisting of three fused benzene rings. In a solid state it is colorless but exhibits a blue fluorescence (400 nm to 440 nm wavelength) under ultraviolet light.

We conducted several tests on the machine to ensure stability regarding the temperature and pressure before deposition. Figure 9 shows two depositions of Anthracene on ITO coated glass with parameters as shown in table 1.

Deposition	cooling temp	sample weight	duration	pressure
1	40'C	102 mg	5 min	166 mbar
2	20'C	200 mg	10 min	166 mbar

 TABLE I

 Deposition parameters for each individual test.



Fig. 9. Pictures of the surface from deposition 1 and 2 with 5X magnification. Right area is Anthracene and lest side is substrate on each picture



(a) Tape test photo.

(b) 3d topology after tape test.

Fig. 10. Tape test photo from Test 1: 1) One tape removal. 2) Two tape removals. 3) Three tape removals. 4) Four tape removals.

A tape test was conducted to obtain a reference point of what is adhesion and what is not. The substrate used in test was divided into different sections, where a bit of tape was used to check the adhesion. Depending on which section, the tape test was repeated from one to four times. The result of this tape test is showed figure 10, where point 1 to 4 is after 1, 2, 3 and 4 tape removals. After 4 tape removals, the deposited layer of Anthracene had vanished.

V. CONCLUSION

After having completed the fabrication of the OVPDmachine, we conducted several depositions. The testing results showed that the evaporated material did flow all the way through the system, and deposited onto the substrate. This



Fig. 11. Two dimensional pictures of deposited sample using interferometer. Deposited Anthracene on the right.

concludes that the pressure and temperature obtained in the system were enough to demonstrate vapor phase deposition. The machine can mix and deposit the mix of material or deposit each material separately to create multilayer deposition on different types of substrates. There are many parameters, such as flow speed, temperature which effect the deposition quality. This requires several controlled depositions with different parameters to identify the effects of these on depositions in this system.

REFERENCES

- [1] Hagen Klauk. "Organic Electronics: Materials, Manufacturing, and Applications", ISBN: 978-3-527-31264-1 June 2006
- [2] Richard R. Lunt, Brian E. Lassiter, Jay B. Benziger, and Stephen R. Forrest. "Organic vapor phase deposition for the growth of large organic electronic devices", Applied Physics Letters, 95, 2009.
- [3] Kunic, S.; Sego, Z., "OLED technology and displays", in ELMAR, 2012 Proceedings, vol., no., pp.31-35, 12-14 Sept. 2012
- [4] "OLED Fabrication", Available from:www.umich.edu/ocm/tools/linear.html
- [5] S. Hartmann, A Janssen, M Gersdorff, N Meyer, M Reinhold, M Schwambera, G Strauch, B Marheineke, M Heuken, HH Johannes, "First Hybrid OLED by Organic Vapor Phase Deposition and Its Advantages in Deposition Rate Control for OLED Manufacturing",... SID Symposium Digest of Technical Papers 05/2004; 35(1):1388-1391.
- [6] Fan Yang. "Thin film solar cells grown by organic vapor phase deposition", Ph.d Thesis, Princeton University, 2008. ISBN: 9780549747420.
- [7] Donald M. Mattox, "Vacuum Deposition, Reactive Evaporation, and Gas Evaporation", ASM Handbook Volume 5, Surface Engineering, ASM International, 1994, Pages: 556 - 572 (17)
- [8] Yung ho Chou, Tienpao Su, M.S.K. Chen, S.M. Fine, M.A. George, J.T. McGuire, S. Dheandhanoo, J.H. Yang, R.J. Ciotti, and D.F Yesenofski. "Design and operation of reactive gas delivery systems for IC fabrication", in Semiconductor Manufacturing Technology Workshop, 2000, pp.243-250,
- [9] Cedric R., Soeren S., Kris M. and D. Cheyns, Stijn Verlaak, Jan Genoe, and Paul Heremans. "Pentacene devices and logic gates fabricated by organic vapor phase deposition", Applied Physics Letters, 89(20), 2006.
- [10] Cedric Rolin, Karolien Vasseur, Jan Genoe, and Paul Heremans. "Growth of Pentacene thin films by in-line organic vapor phase deposition", Organic Electronics, 11(1):100108, 2010.
- [11] M. Shtein, J. Mapel, J.B. Benziger, and S.R. Forrest. "Effects of film morphology and gate dielectric surface preparation on the electrical characteristics of organic-vapor-phase-deposited pentacene thinfilm transistors", Applied physics letters, 81(2):268-270,2002. 4, 54
- [12] Pyung-yong Um. "Shower head of chemical vapor deposition apparatus", 2009. http://www.freepatentsonline.com/20090159001.pdf