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Organic ferroelectric evaporator with substrate cooling and *in situ* transport capabilities

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We report on the design, operation, and performance of a thermal evaporation chamber capable of evaporating organic thin films. Organic thin films are employed in a diverse range of devices and can provide insight into fundamental physical phenomena. However, growing organic thin films is often challenging and requires very specific deposition parameters. The chamber presented here is capable of cooling sample substrates to temperatures below 130 K and allows for the detachment of the sample from the cooling stage and *in situ* transport. This permits the use of multiple deposition techniques in separate, but connected, deposition chambers without breaking vacuum and therefore provides clean, well characterized interfaces between the organic thin film and any adjoining layers. We also demonstrate a successful thin film deposition of an organic material with a demanding set of deposition parameters, showcasing the success of this design. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4869874>]

INTRODUCTION

The ability to fabricate heterostructured thin films of organic/non-organic layers is essential for both the understanding of fundamental physical phenomena at the interface between the layers and the development of devices. An example of a fundamental interfacial phenomenon that occurs at organic/inorganic interfaces (and the driving force behind the evaporator described in this paper) is the magnetoelectric coupling between an organic ferroelectric thin film and a metallic ferromagnet which results in a pronounced effect on the magnetic anisotropy of the ferromagnetic film.¹ The low stiffness coefficient¹ of the polymer ensures that there is little to no strain coupling between these two layers, thus enabling the investigation of the effect of electric fields on magnetism in the absence of strain coupling. Magnetic tunnel junctions^{2,3} (MTJs) and ferroelectric tunnel junctions⁴ (FTJs) using organic tunnel barriers have displayed distinctive behavior that is tied to the properties of the organic barrier. Devices based on organic thin films include high efficiency solar cells,^{5–7} field-effect transistors (FETs),⁸ flexible organic light-emitting diodes (OLEDs),⁹ antireflection coatings,¹⁰ and even magnetic field sensors,¹¹ all of which require conducting electrodes in intimate contact with organic thin films. In all cases, a clean, well-characterized interface between the organic thin film and the adjoining layer(s) is desirable, necessitating *in situ* vacuum deposition processes for the entire heterostructure. Although other organic thin film deposition techniques, such as spin coating and Langmuir-Blodgett deposition, produce excellent thin films that are crystalline, defect free, and fully functional,^{4,12} these techniques are not compatible with metal deposition and are detrimental to the formation of a

clean interface between the organic thin film and adjacent metallic layers.

Developing a deposition system capable of making a heterostructured organic/inorganic device, such as an MTJ, can be quite difficult for several reasons. First, it is unlikely that a single deposition technique can be used for both the inorganic and the organic thin films, requiring the deposition system to be capable of effusion (for the organic) and another technique for metallic layers (e.g., sputtering). Additionally, many organic materials have more than one crystalline phase. The oligomer vinylidene difluoride (VDF), for example, has several crystalline phases, only one of which is ferroelectric. Successful thin film growth of this crystalline form necessitates low substrate temperatures during deposition;¹³ hence the second requirement for many organic deposition systems is the ability to control the substrate temperature during deposition. Finally, in order to prevent contamination, separate, connected chambers are necessary for the deposition of the organic layer and the inorganic layer(s). Therefore, a heterostructured sample deposition system must be capable of *in situ* sample transport. While sample transport from one chamber to another is easily accommodated by a transfer arm and a gate valve, a deposition system capable of sample transport and substrate cooling can be quite difficult to construct.

In this paper, we describe the design, construction, and performance of an organic thin film evaporator that can be easily integrated into an existing deposition system (in this case a dc/rf magnetron sputtering/e-beam evaporation system) and also meets all of the above requirements. Given that these design constraints must be accounted for concurrently, it is clear that other homemade organic evaporators^{14–16} or commercial systems, such as a self-contained Knudsen Effusion Cell,^{17,18} will not meet the needs of many researchers. Here, we present a system that is capable of evaporating up to

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FIG. 1. Photograph of the organic evaporator (left)/sputtering and e-beam chamber (middle)/load lock (right) system. Gate valves separate each of the three chambers. The organic evaporation chamber and Transfer Arm 2 were connected to an existing 8 in. port on the sputtering/e-beam deposition system.

four distinct materials onto a substrate and, more importantly, is capable of both substrate cooling to temperatures below 130 K and *in situ* sample transport.

DESIGN AND CONSTRUCTION

The design of the evaporation chamber was dictated by an existing commercial AJA International, Inc. ATC-2000¹⁹ four gun sputtering/e-beam evaporation system used for metal deposition, which we briefly describe below. The cylindrical sputtering/e-beam chamber (shown in Fig. 1) is connected to a load lock chamber and a sample transfer arm (Transfer Arm 1). The load lock chamber contains a substrate cassette elevator with a six cassette capacity. Substrates and masks are attached to a ring/disc system of cassettes (3.75 in. diameter discs, 0.0625 in. thick, set within a stainless steel ring) which can be loaded into the substrate cassette elevator in the load lock. A mask changer in the load lock enables the deposition of multiple layers with different mask patterns without breaking vacuum. A cassette carrier on the end of Transfer Arm 1 is able to transfer the substrate cassettes into and out of the sputtering/e-beam chamber's cassette holder. The cassette holder and transfer arm of the organic deposition chamber (Transfer Arm 2) are designed to be compatible with this system.

The main body of the organic deposition chamber consists of a six-way cross with standard 8 in. ConFlat[®] (CF) flange ports, as shown in Figure 1 and illustrated in Figure 2. Defining the optical window as the front, the sputtering/e-beam chamber is connected to the right port via an 8 in. gate valve,²⁰ Transfer Arm 2 is connected to the left, a quartz crystal deposition monitor and the vacuum pumps are mounted on the back, the effusion cell is connected to the bottom, and finally the sample mount and cooling system are connected to the top port.

The manually controlled gate valve separating the organic thin film deposition chamber from the sputtering/e-beam chamber prevents cross-contamination during the deposition process. The 36 in. long Transfer Arm 2 is connected to the organic deposition chamber via an eight to six inch reducer flange and moves cassettes between the sputtering/e-

beam chamber and the organic deposition chamber. The 4 in. bore of this reducer flange allows for the end of the transfer arm to be fully retracted out of the six-way cross. The 4.5 in. T flange mounted on the back of the cross serves as the connection for both the vacuum pumps and the quartz crystal

FIG. 2. External side view (a) and top view (b) of the organic thin film deposition chamber. The customized flanges attached to the top and bottom of the chamber are described in the text.

FIG. 3. Side view (a) and bottom view (b) of the custom 8 in. flange mounted on the bottom of the six-way cross.

deposition monitor. The deposition monitor is mounted on a horizontal bellows with 100 mm of travel, allowing the deposition monitor to be fully retracted during sample deposition.

The customized flange mounted on the bottom of the cross, shown in Fig. 3, contains the effusion cells (Fig. 4), which are based on a commonly used Knudsen Cell design.^{21,22} Five 1.33 in. half-nipple flanges were welded to holes bored out of a standard 8 in. blank CF flange. Two of these half-nipple flanges serve as ports for power feedthroughs to provide current to filaments in the effusion cells and two serve as ports for thermocouple feedthroughs to monitor effusion cell temperature. The fifth 1.33 in. half-nipple in the center of the 8 in. custom flange serves as a port for a rotary motion actuator²³ that controls a three-quarter circle shaped shutter (visible in the inset of Fig. 6(b)) above the effusion cells that selects a single cell for deposition. Stainless steel tubing, 0.25 in. in diameter, was welded into two additional smaller holes to serve as both structural support and water coolant lines for the effusion cells. Because the effusion cells are so close together, it is necessary to cool the platform on which the cells are mounted (labeled in Fig. 4) in order to prevent heat transfer between cells and the consequent evaporation of material from other cells. A four-way

FIG. 4. Perspective view of the effusion cells with the water cooled mount.

sheet metal cross (not shown) between the effusion cells further limits cross-contamination.

The top flange and the accompanying low temperature cassette holder are the focus of this paper. The top flange consists of a standard, blank 8 in. CF flange with seven holes bored into it (as shown in Figure 5). 1.33 in. half-nipple

FIG. 5. Side view (a) and top view (b) of the custom 8 in. flange mounted on the top of the six-way cross.

FIG. 6. (a) Photograph of the substrate cooling system. OFHC copper sheets allow for vertical motion of the cassette holder while providing a thermal link between the liquid nitrogen reservoir and the cassette holder. (b) Photograph of the assembled organic deposition system as viewed through the front window, showing the need for vertical motion. The transfer arm (just visible on the left) brings a cassette into the chamber while the linear motion actuator allows the sample to be lifted off the transfer arm and put into position above the effusion cells. Also visible in this picture is the deposition monitor in its fully retracted position (center). The inset of (b) provides a view of the shutter above the effusion cell housing.

flanges were welded into five of these holes. Two of these half-nipples will be used as ports during future experiments and were closed off with blank 1.33 in. flanges, another leads to an ion gauge,²⁴ and a fourth to a nitrogen gas line used to vent the chamber. A linear motion actuator²⁵ is connected to the central half-nipple flange and is attached to the cassette holder (shown in Figure 6). Two open-ended 0.75 in. diameter stainless steel tubes are welded into the last two holes. The ends of the tubes that extend into the volume of the deposition chamber are welded to a 100 cm³ rectangular stainless steel liquid nitrogen reservoir with an oxygen-free, high thermal conductivity (OFHC) copper bottom that is silver brazed to the stainless steel reservoir, forming a leak-free seal. The reservoir and tubes are open to the atmosphere and sealed off from the chamber vacuum. The reservoir itself is within the volume of the six-way cross to minimize the distance between it and the substrate, since it is the liquid nitrogen within the reservoir that cools the substrate.

The organic deposition system's cassette holding stage and transfer arm have been made to match the specifications of the cassette transfer system of the sputtering/e-beam deposition system. The cassette holder, made of OFHC copper to maximize thermal conduction, is attached to the end of the linear motion actuator as shown in Figure 6. The linear motion actuator allows for the height of the cassette holder to be adjusted as needed, allowing for the cassette to be placed onto and removed from the transfer arm, and also allowing for the distance between the sample and effusion cells to be changed *in situ*, controlling the deposition rate and the uniformity of the deposited film. As shown in Fig. 6, the copper bottom of the liquid nitrogen reservoir is connected to the cassette holder via eight sheets of OFHC copper, each 0.004 in.

thick, 3 in. wide, and 9 in. long. The sheets are attached to the OFHC copper bottom of the liquid nitrogen reservoir and the top of the cassette sample holder along their width. The thermal conductivity of these copper sheets at liquid nitrogen temperatures is approximately 580 W/m K.²⁶ Many deposition systems that require substrate cooling rely on a rigid cold finger^{27,28} which does not allow for *in situ* motion, unlike the copper sheets, which are flexible enough to allow for vertical motion of the sample holder.

As discussed above, samples are loaded onto cassettes and transferred from the sputtering/e-beam system to the organic deposition system via Transfer Arm 2 (see Figure 1). These cassettes (described above) are OFHC copper discs mounted on stainless steel rings. This disc/ring configuration is essential to the *in situ* mask exchange process performed in the load lock, and therefore the design of these cassettes cannot be changed. Because the only thermal contact with the copper disc is via the lip of the stainless steel ring, which in turn sits on the lip of the copper cassette holder, additional thermal contact is provided by the arrangement shown in Fig. 7(a), which consists of four sets of hangers constructed from 0.007 in. thick beryllium copper (BeCu) foil cut to resemble a comb. These hangers, fastened onto the cassette holder, are stiff enough to maintain contact to the cassette disc, but flexible enough to be pushed upwards by the motion of the transfer arm when a cassette is being removed or inserted into the cassette holder. Four sheets of OFHC copper foil are attached under the BeCu hangers. These copper foils wrap around the BeCu hangers and extend over the width of the cassette holder (3 in.) to maximize the thermal conduction area. The combination of the stiff BeCu comb-like hangers and the more flexible OFHC copper allows for both good

FIG. 7. (a) Photograph of the partially disassembled cassette holder showing the BeCu/Cu hangers. (b) A cassette in position within the cassette holder (as viewed from below). (c) Simple diagram showing how the hangers perform during cassette loading. As the transfer arm moves the cassette into position, the hangers bend out of the way (top). When the cassette is in position relative to the cassette holder, the cassette holder is moved upwards via the linear motion actuator and lifts the cassette off of the transfer arm. As the cassette is being lifted, the hangers relax back to their natural position so that they are always in contact with the cassette itself (bottom). This configuration allows for samples to be moved *in situ*, and remain in good thermal contact with the cassette holder during deposition.

thermal contact as well as the ability to move substrates in and out of the chamber. Figure 7(b) shows how this hanger system works during cassette loading, and Figure 7(c) shows a cassette in position for a deposition in the cassette holder (during an actual deposition, a substrate would be affixed to this cassette). To remove the cassette from the cassette holder after

the deposition, the linear motion actuator lowers the cassette onto Transfer Arm 2. The cassette is then removed from the cassette holder by moving the transfer arm to the left (refer to Fig. 7(b)), ensuring that the hangers bend in the same direction for both cassette loading and unloading. This customized top flange, along with the sample cooling system discussed above, allows for both sufficient substrate cooling and *in situ* detachment from the cooling stage.

OPERATION AND PERFORMANCE

The temperature performance of this system was repeatedly measured using a Si thermocouple wafer loaded on a cassette in the substrate position. Liquid nitrogen is transferred to the reservoir from a standard 230 L liquid nitrogen dewar under a pressure of 22 psi via insulated copper tubing. The flow is adjusted so that the reservoir stays full with little overflow. Reproducible cooling curves are shown in Figure 8. A substrate temperature of 132 K is achieved after 100 min of cooling, with an ultimate temperature of 128 K after another 55 min (black line). The temperature stability is approximately one degree over a period of 35 min, more than enough time for a typical thin film deposition. Clearly, these data show that the flexible BeCu/Cu hanger system provides sufficient thermal conductivity while still allowing for sample transport.

Several other hanger designs were tested before this BeCu/Cu hanger system. Previous iterations were either incapable of reaching the desired temperature of 130 K or incapable of reaching it in a timely fashion. The results of these previous configurations are also shown in Fig. 8 and described briefly below. The blue line corresponds to hangers made of uncut BeCu sheets. These uncut sheets of BeCu were too stiff to bend out of the way of the cassette during loading

FIG. 8. Substrate temperature versus time for both cooling and warming cycles. The black line corresponds to hanger design described in detail above (exponential fit in red). After about 160 min, the liquid nitrogen reservoir was allowed to empty and the sample warmed back up to room temperature in a linear fashion. Other hanger configurations were also tested, but were found to be inadequate. The blue, green, and pink lines correspond to failed hanger designs also described above.

(Fig. 7(b)) and therefore not a viable option. The green line corresponds to hangers made of uncut sheets of Cu foil, which were not stiff enough to maintain sufficient contact with the cassette once in position. The pink line corresponds to a hanger system of cut (comb-like) BeCu wrapped in full sheets of Cu identical to the hanger system described in the section titled Design and Construction. However, in an attempt to minimize the strain on the linear motion actuator, there were only four sheets of Cu connecting the liquid nitrogen reservoir and the cassette holder during this test. This last iteration of the cooling system could reach an ultimate temperature close to that of the final design, but took much longer to do so.

To perform a test deposition of the ferroelectric oligomer vinylidene difluoride ($\text{CF}_3\text{-(CH}_2\text{CF}_2)_n\text{-I}$, $n \approx 20$), the 100 μL volume crucibles in the effusion cells are loaded. Earlier work has shown that evaporation of this oligomer results in a ferroelectric phase with polarization perpendicular to the plane of the substrate *only* under stringent growth conditions of low substrate temperatures and low deposition rates.¹³ A glass substrate is mounted on a cassette and inserted into the load lock, which is then pumped down to a pressure of 9×10^{-8} Torr. The cassette is then moved to the sputtering/e-beam chamber (with a base pressure of 1×10^{-8} Torr), where a bottom metallic layer (in this case Pt/Co) is deposited using the desired mask. The cassette, with the substrate and mask, can then be moved back to the load lock, where the mask used for the bottom electrodes may be changed or removed. Using a two stage process and both transfer arms, the cassette is moved into the organic deposition chamber, with a base pressure of 9×10^{-8} Torr, via the sputtering chamber. Transfer Arm 2 moves the cassettes from the sputtering/e-beam chamber's cassette holder to the position in the organic deposition chamber shown in the upper portion of Figure 7(b). The linear motion actuator is then used to raise the cassette holder, lifting the cassette off the transfer arm. The transfer arm is then fully retracted in the evaporation chamber, and the reservoir is filled with liquid nitrogen. The effusion cell is heated using an applied current of about 0.45 A. Deposition rates were measured using the quartz crystal monitor. The deposition versus time curve shown in Fig. 9 indicates that the deposition rate

FIG. 9. Deposition rate (blue), total thickness (black), and crucible temperature (green) as functions of time. Opening the shutter on the effusion cell at the appropriate time exposes the sample to only the linear portion of the thickness versus time curve (linear fit in red).

FIG. 10. Pyroelectric hysteresis loop taken on a VDF oligomer thin film grown in our deposition system. This loop shows switching between the two ferroelectric polarization states and demonstrates that the chamber is capable of both stable deposition and substrate cooling, both of which are simultaneously necessary to obtain the ferroelectric crystalline phase with polarization pointing out-of-plane.

is almost constant once the temperature of the crucible stabilizes at 57–58 °C, which occurs ~ 40 min after the application of current. For the deposition rate test shown in Fig. 9, the shutter on top of the effusion cell housing was left open the entire time.

The test sample, created using the combined techniques of sputtering and organic thin film deposition, consisted of glass/Pt (50 nm)/Co (1.5 nm)/VDF (200 nm)/Al (20 nm). The shutter over the effusion cell was opened 40 min into the application of a 0.45 A current to the effusion cell (at a point corresponding to the “start” label in Fig. 9) to ensure a uniform deposition rate of 0.25 Å/s. Using the Chynoweth Method,²⁹ a pyroelectric hysteresis loop of the VDF was measured (Figure 10). The pyroelectric current is directly proportional to the out-of-plane ferroelectric polarization of the oligomer. This loop clearly demonstrates polarization switching behavior, a hallmark of ferroelectricity. Hence we have successfully fabricated a thin film of the ferroelectric crystalline phase of VDF with out-of-plane polarization.

CONCLUSION

We have described the design, construction, operation, and performance of an evaporative chamber capable of substrate cooling and *in situ* sample transfer between deposition chambers. This design includes a quartz crystal deposition monitor for thickness calibration. Our comb-like hanger system is capable of cooling the substrates to a temperature of 128 K, while still allowing samples to be moved without breaking vacuum. Although the design of this system was largely governed by an existing chamber, the design we present here could be easily adapted for a stand-alone system, since the ability to adjust the vertical position of the sample is useful for regulating the uniformity of the deposited film.

Depositing the oligomer VDF in its ferroelectric crystalline phase in direct contact with a metallic layer demands simultaneous control of a very specific set of deposition parameters. We have successfully achieved this, as evidenced by the characteristic ferroelectric hysteresis loop clearly indicating that the VDF thin film grows in its ferroelectric crystalline phase with polarization pointing out-of-plane, with a clean interface at the underlying metallic layer. This deposition system opens a new channel for the exploration of physical phenomena associated with organic thin film heterostructures.

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