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Experimental Techniques

Device fabrication and study of its characteristics require understanding of various tools and their working, so that one can monitor different parameters during fabrication of devices. Each component of a device can be optimized to obtain desired device performance, and it can only be achieved by having understanding of experimental tools and their control parameters. Few of the equipments that have been utilized to complete this working are discussed briefly in the below sections.

3.1 Spin Coating

This is a very basic and crucial machine for uniform and controlled deposition of solution processed materials on a substrate. Fig. 3.1.1 (a) shows the digital image of the machine. The basic working mechanism is illustrated in Fig. 3.1.1 (b-e). Initially, the substrate is properly cleaned and then placed on top of the vacuum chuck, that holds the substrate during rotation. The prepared liquid solution that is to be coated, is then carefully placed/poured on the substrate. The substrate is then rotated at certain speed for some specified time to obtain a uniform film. This obtained film then undergoes evaporation of solvent, and finally a thin film with desired thickness is obtained. It can be observed that thickness of the film depends on the properties of solution that is to be coated (viscosity and density), speed and time of rotation, and other environmental factors as well [Meyerhofer, 1978]. The earliest physics and equation given for thickness of the spin coated films took care of all these factors, except for the environmental factor (solvent evaporation) [Emslie *et al.*, 1958]. The equation is as follow

$$h = \frac{h_0}{(1 + 4K h_0^2 t)^{1/2}}; K = \rho \omega^2 / 3\eta \quad (3.1)$$

where, h is the thickness of the film, h_0 is a constant that corresponds to initial height, ρ is the density, ω is disk angular velocity, η is viscosity, and t is time of rotation. The equation was later improved by introducing various factors, but this remained the governing basic equation. Gelatin, egg-albumen, and pectin solutions were deposited by utilizing spin coating technique during my research work.

3.2 Thermal Deposition System

This system is primarily utilized to deposit thin layers of metal electrodes of the device. Fig. 3.2.1 (a) shows the digital image of Semicore thermal vacuum deposition system. This system is able

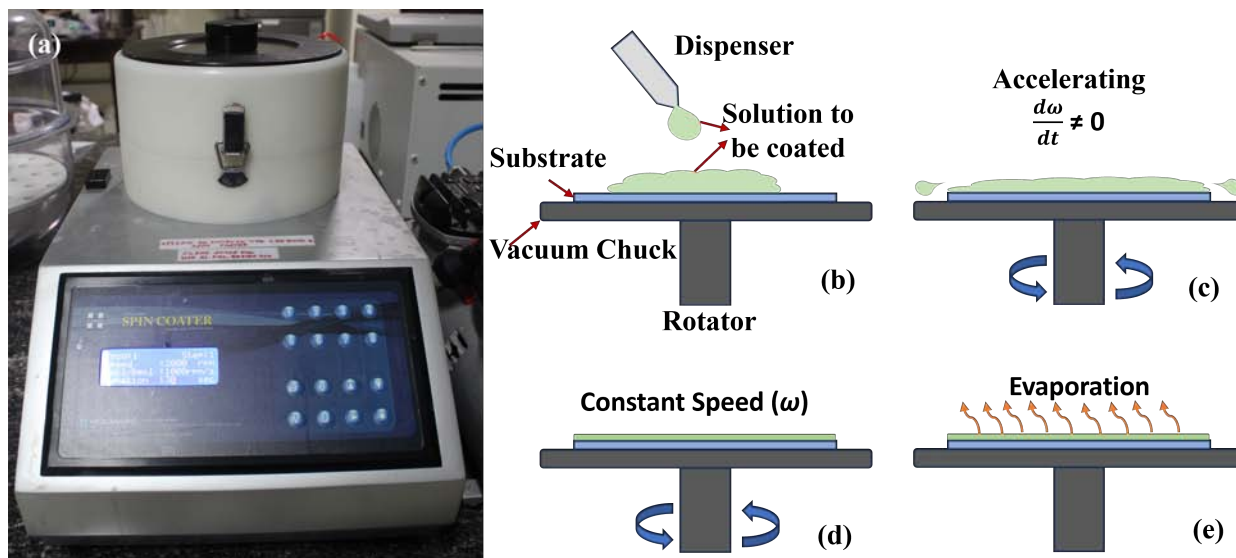


Figure 3.1.1: (a) Digital image of spin coater machine. (b-e) Steps and working schematics of spin coating process to obtain a uniform thin film.

to achieve a vacuum of 10^{-6} torr, resulting uniform deposition. Fig. 3.2.1 (b) shows schematic of the machine and its important components. The whole process occurs in a high vacuum chamber to reduce the number of particles in the path, and thus increasing the mean free path of the evaporated material and gives better quality film. The desired material is kept in a boat, and the boat is heated to the melting point of the material. The liquid material gives rise to a vapour cloud in the vacuum environment and the cloud starts moving upwards. The chamber is often equipped with calibrated Quartz Crystal Microbalance (QCM) to measure the thickness of deposited film by monitoring the rate and time of vapour travel. The shutters are employed to stop deposition process immediately at the desired film thickness, as it is not possible to stop the heating of the material instantaneously. As a boat is required to heat the material, this technique is limited to materials having melting point below that of boat only.

3.3 E-beam Deposition System

This system is also utilized to deposit metallic film on the substrate. It has most of its working very similar to that of previous thermal evaporation system. However, in this technique a directed beam of electron (e-beam) is used for vaporizing the source material. The e-beam is generated from a very hot filament and the generated beam is then focused on the source material by utilizing focusing magnets. This focused e-beam then hits the source material and vaporizes it to start the deposition process. One advantage of this technique is that it can be used for deposition of high melting point materials.

3.4 Atomic Layer Deposition System

Atomic layer deposition (ALD) is a very reliable and accurate thin film deposition technique. It deposits film layer-by-layer and thus provides greater control on the thickness of the obtained film. Earlier the term atomic layer epitaxy (ALE) was used, and the system was used to deposit

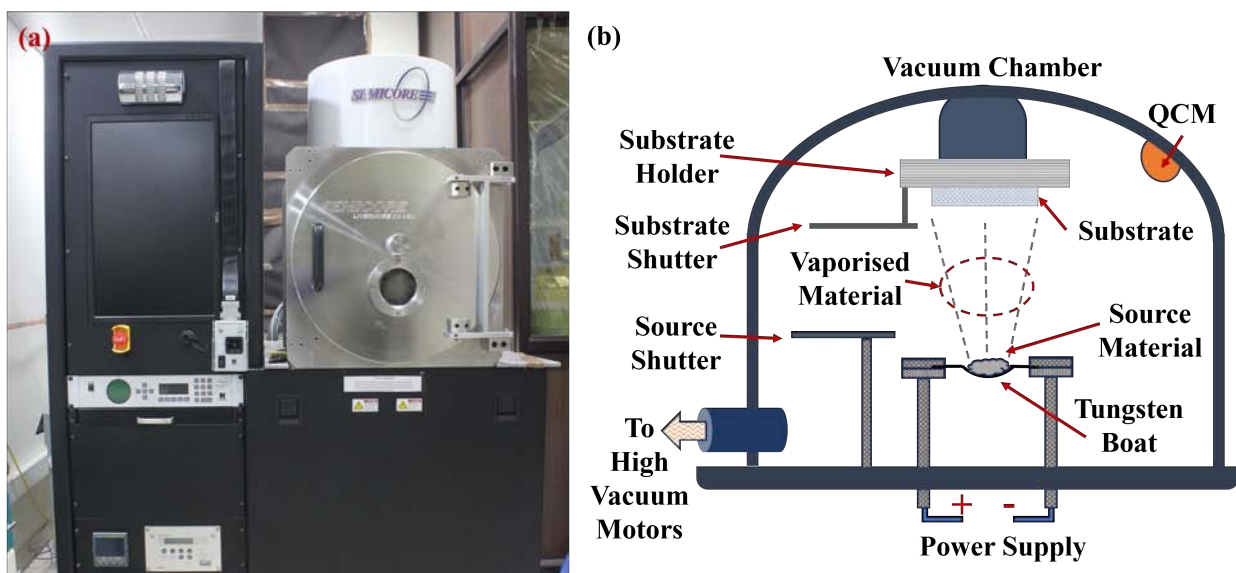


Figure 3.2.1: (a) Digital image of thermal deposition system. (b) Illustrative representation of the machine, its parts, and working.

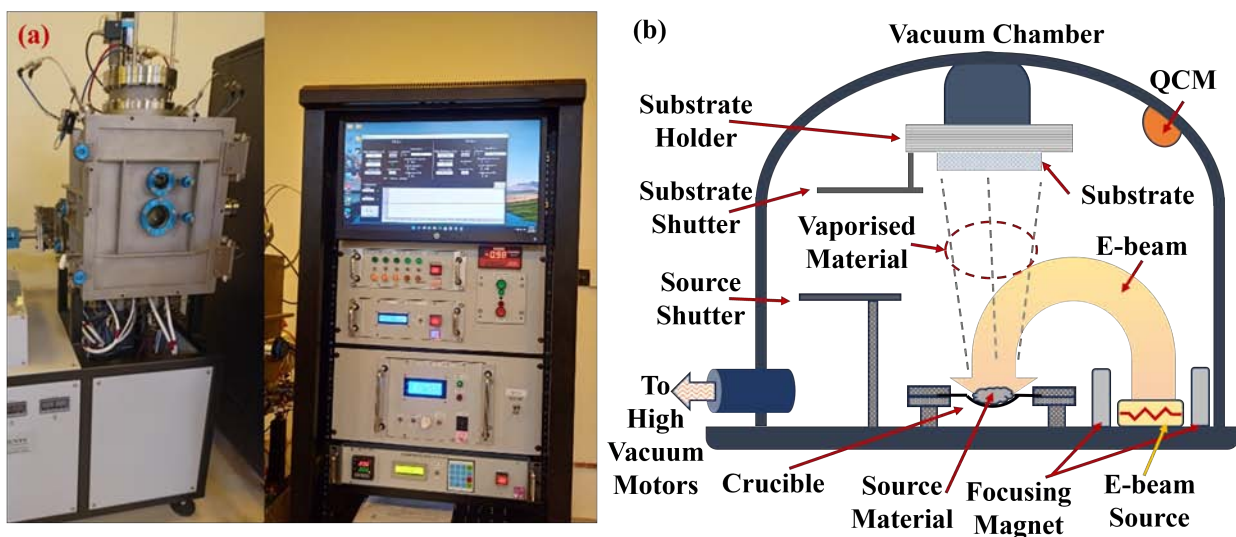


Figure 3.3.1: (a) Digital image of the e-beam deposition system. (b) Schematic of the e-beam deposition process.

ZnS by *Suntola and Antson* in 1977 for flat panel displays [Suntola and Antson, 1977]. Later the system was developed to deposit many materials non-epitaxially, and thus the term ALD was adopted. ALD belong to chemical vapour deposition technique family, however the basic difference lies in that fact that in ALD there is alternate exposure of precursors to form the desired material [Johnson *et al.*, 2014]. Fig. 3.4.1 (a) shows the Cambridge Nanotech's Savannah S200 ALD system. Fig. 3.4.1 (b) illustrates the working mechanism and process flow of the technique. A cleaned substrate is placed in the deposition chamber and heated to some specific temperature before starting the deposition process. Then, sequential alternating pulses of precursors (reagent 1 and 2) are supplied with precise control. Nitrogen gas as a carrier is utilized to supply gaseous

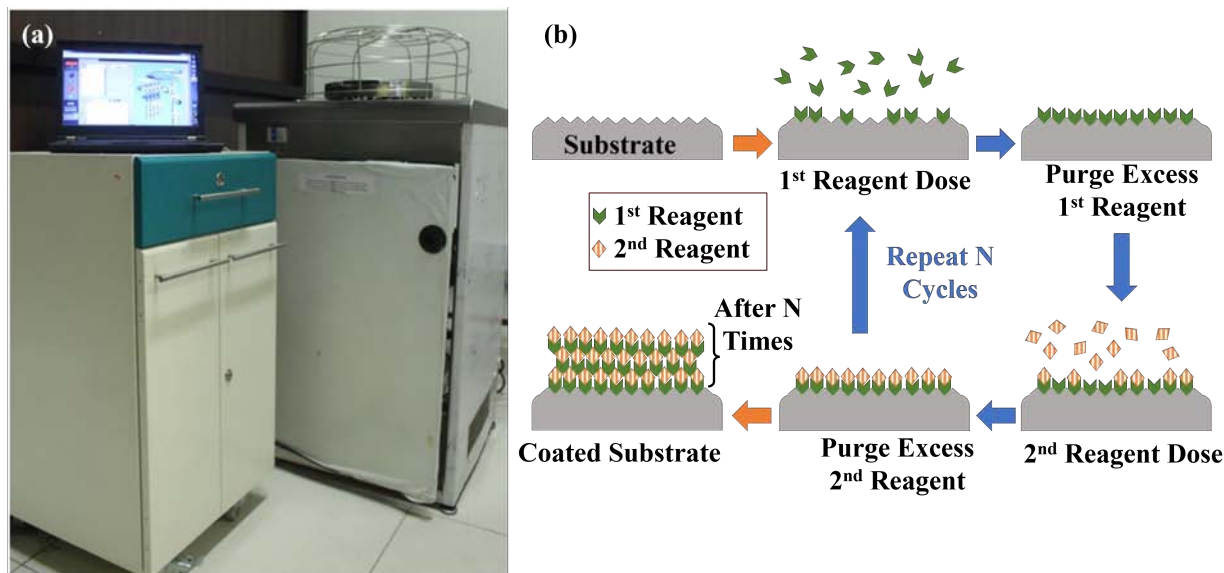


Figure 3.4.1: (a) Digital image of the Savannah S200 ALD system. (b) Schematic representation of the ALD process flow.

precursor to the deposition chamber. Excess reagent is removed after every half cycle before arrival of other reagent. The process is repeated multiple times till the desired thickness is obtained.

3.5 Atomic Force Microscopy

Atomic Force Microscopy (AFM) is a very powerful surface imaging technique that helps in study of any surface (conducting or non conducting) at atomic and molecular level. The technique was invented by Binnig, Quate, and Gerber in 1985, where they had utilized microscopic diamond as tip and silver foil as cantilever [Binnig *et al.*, 1986]. Fig. 3.5.1 (a) shows a modern Park systems AFM machine. The digital image of output software during an ongoing measurement can be seen in Fig. 3.5.1 (b). The working principle of AFM can be roughly represented as shown in Fig. 3.5.1 (c). The AFM tip interacts with the surface of the sample by Van der Waals force, and the deflection arising from this interaction is reflected by the laser beam on to a position sensitive photo detector. The generated signal is sent to detector circuit and software that maps the deflection into surface roughness. The relative position of AFM probe is controlled by the piezo-electric scanner. There are three modes of operation of an AFM, first is contact mode in which tip maintains contact with the surface continuously, second is tapping mode in which the cantilever oscillates at a given frequency and the tip moves on sample by tapping on the sample, and the third is non-contact mode where the oscillating tip is kept few nanometers away from the surface of the sample.

3.6 2-D Surface Profilometer

The technique of stylus surface profiling is among the oldest techniques for measurement of surface topography. The stylus scans the surface in a predefined direction at specified speed and stylus force in contact mode to generate surface topography. The surface variation of the film is reflected in stylus movement, which is further captured by the linear variable differential

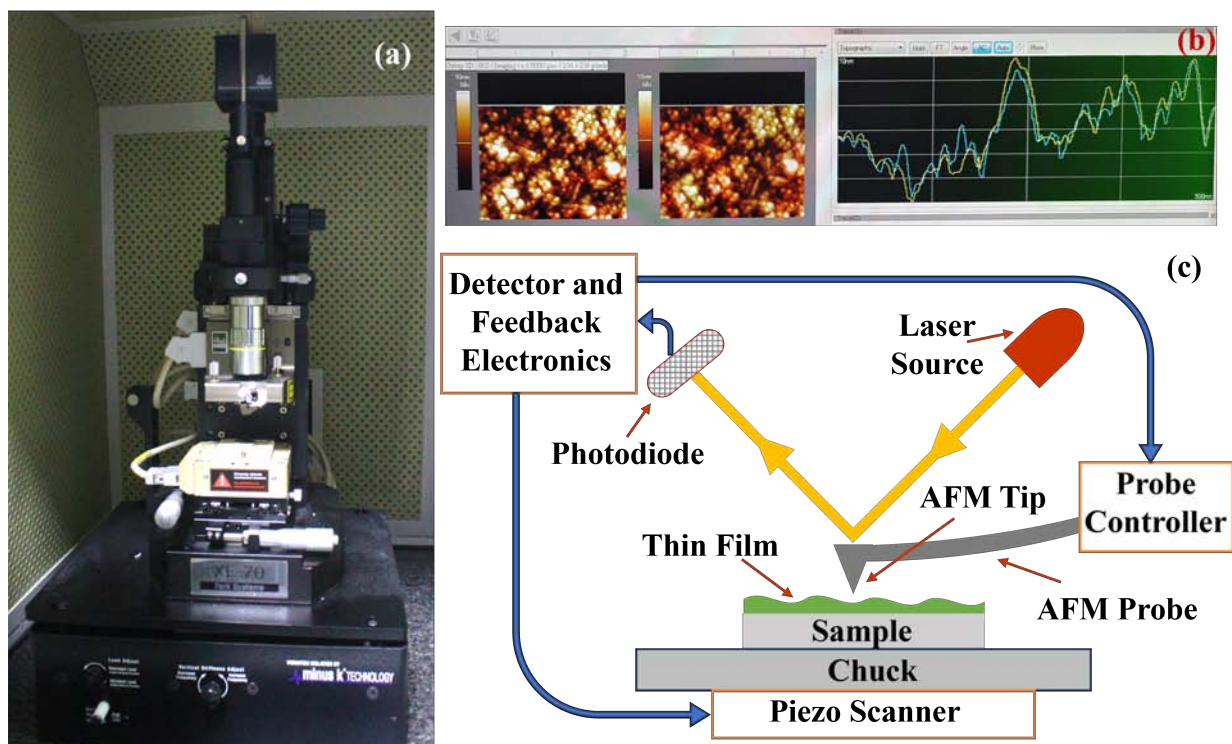


Figure 3.5.1: (a) Digital image of Park Systems XE-70 AFM machine. (b) Basic schematic of working principle of an AFM machine.

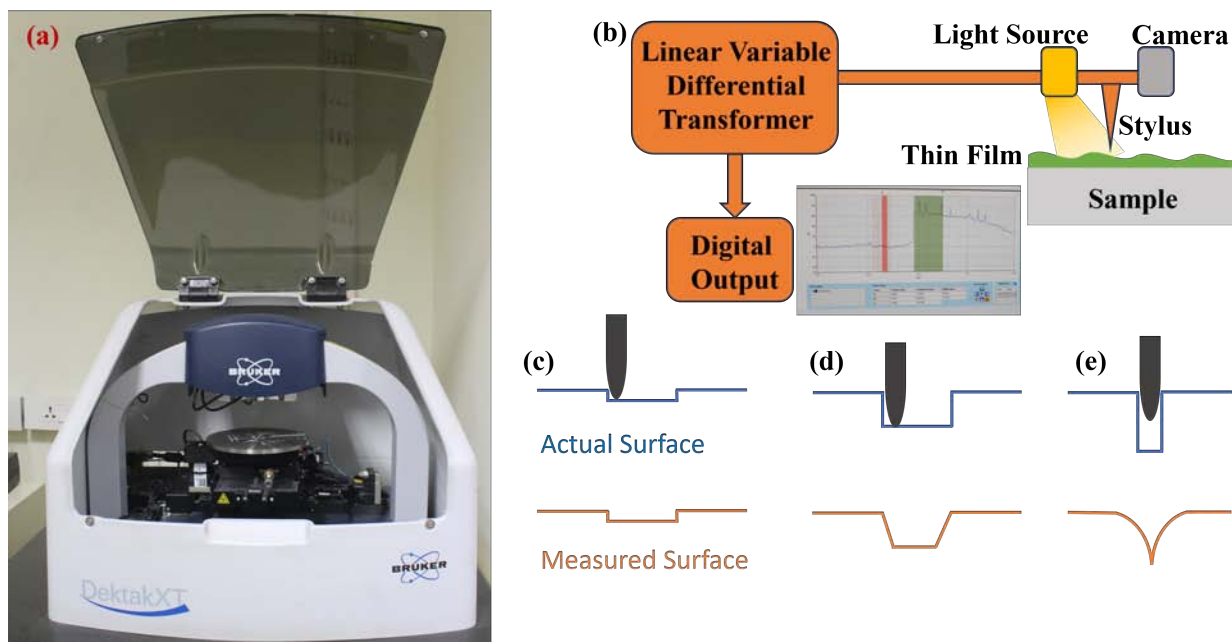


Figure 3.6.1: (a) Digital image of the Bruker DektakXT surface profilometer. (b) Schematic diagram showing important components and working of stylus profilometer. (c-e) Limitation on lateral resolution of the system as the width of valley reaches to stylus dimension.

transformer that generates electrical signal in the response of stylus deflection. Fig. 3.6.1 (a) shows the digital image of Burkert DektakXT surface profiling machine. Fig. 3.6.1 (b) illustrates the basic working and data acquisition components of the system along with a digital image of output screen of Vision64 software. The camera and light source give real time image of the film surface so that stylus can be navigated to the desired location. The shape and size of the stylus plays very important in this system as it is the point of contact that generates data [Song and Vorburger, 1991]. From Fig. 3.6.1 (c-e) shows the variation in output data with respect to original surface as a function of aspect ratio of structure, thus limiting the lateral resolution of measurement [Bennett and Dancy, 1981]. As the system works in contact mode it is prone to wear and tear, and the sample must be examined before being loaded in the system.

3.7 Electrical Characterization

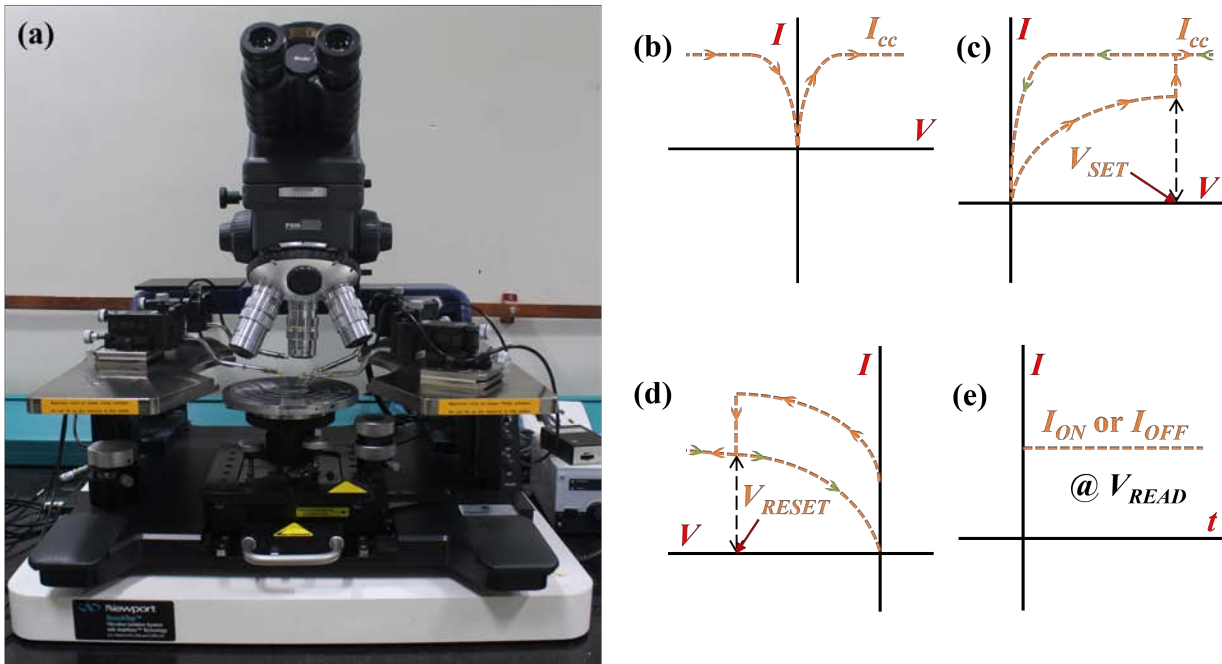


Figure 3.7.1: (a) Digital image of the probe station. Schematic of output characteristics during (b) Short circuit test, (c) SET cycle, (d) RESET cycle, and (e) Retention test.

Performance of the fabricated devices were evaluated by doing electrical characterizations. As the fabricated device is a two terminal device, a two probe setup was utilized to take electrical measurements. Fig. 3.7.1 (a) shows a digital image of a Cascade Microtech PM5 probe station that is utilized to connect the top and bottom electrode of the device to characterization unit. The Keithley 4200 semiconductor characterization system was used to obtain all the electrical characterization results. Fig. 3.7.1 (b) the schematic of output $I - V$ after short circuit test. This is an initial test performed by shorting both the probes to verify that all electrical connections are fine. The compliance current (I_{cc}) is applied to limit the maximum current flow as the connection are shorted. Fig. 3.7.1 (c) shows the SET cycle curve of a resistive memory device with set voltage (V_{SET}). Similarly, RESET cycle is shown in Fig. 3.7.1 (d) with reset voltage (V_{RESET}). All these characterizations are performed in voltage sweeping mode in which voltage is varied from some initial value to final value. The retention test of the device is performed in sampling mode where

a specific read voltage (V_{READ}) is applied after certain time interval and current is recorded, as shown in Fig. 3.7.1 (d). A low voltage is often selected as V_{READ} so that it does not change the current state of the device.

3.8 Model Parameter Extraction and its Utilization

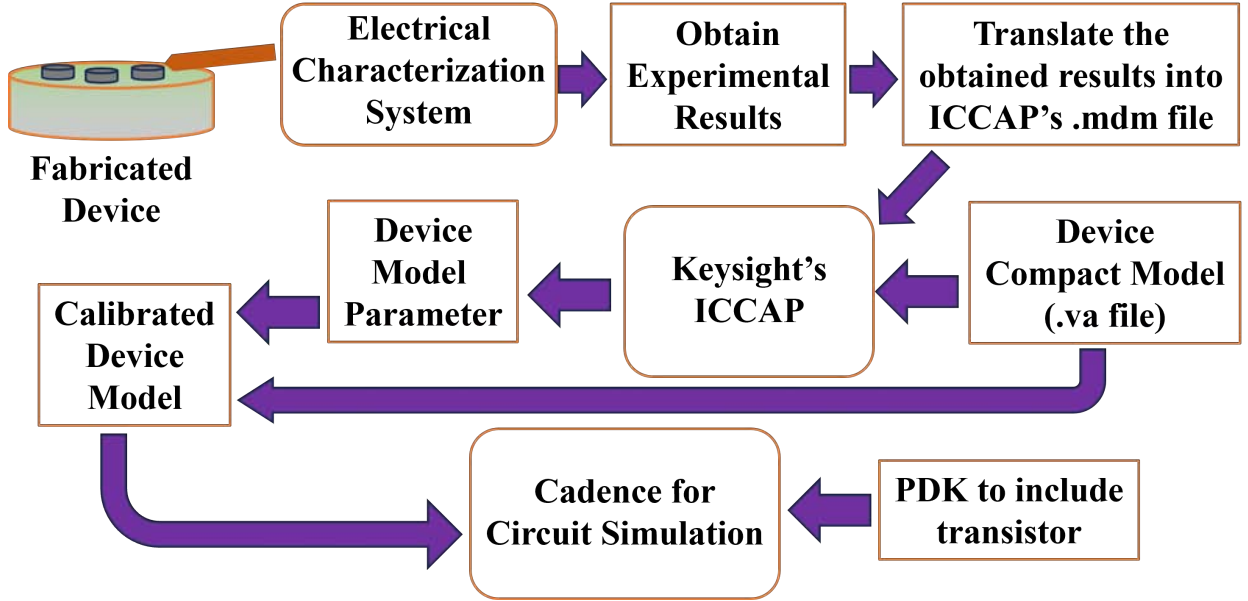


Figure 3.8.1: Process flow to achieve circuit simulation from device results.

Once the device has been fabricated and its performance has been observed, the next step is to utilize the device in a circuit. However, before investing in fabrication of actual circuit it is always recommended to simulate the circuit beforehand. The process flow to achieve circuit simulation from a device is illustrated in Fig. 3.8.1. The experimental results are obtained by electrical characterizations of the fabricated device. The experimental result and the device model is utilized for extraction of fitting parameter which is done by the Keysight Technologies' IC-CAP (Integrated Circuit Characterization and Analysis Program). IC-CAP is a customizable semiconductor device modeling tool which accepts experimental data and input model files in Verilog-A, an industry standard modeling language for analog circuits. Moreover, IC-CAP allows to vary the model parameters so that simulated data fits well with the experimental data for efficient extraction of correct model parameters. These parameters when integrated with device model gives calibrated device model. The calibrated model can then be used for device and circuit simulation related studies. The model can be imported in Virtuoso Cadence as va file to design any proof of concept circuit by utilizing the fabricated device performance.