CHAPTER VII

SUMMARY AND CONCLUSIONS

The chemical deposition methods are low cost processes and the deposited films are found to be of comparable quality to those obtained by more sophisticated and expensive deposition process. Generally, acquiring good quality films in CBD method depends on chemicals, their concentration, and temperature of hot bath, pH value of the solution, dipping time and withdrawing speed of the substrate. In CBD method, the film formation on substrate takes place when ionic product exceeds solubility product by heterogeneous nucleation and also results in unnecessary formation of precipitate in the solution by homogeneous nucleation that leads to wastage of material. The wastage of material is avoided by modifying CBD into SILAR method. By keeping these factors, a PC controlled dip coating machine has been designed and fabricated. This machine can be used for a simple CBD which utilizes only the up/down control.

We employed SILAR method for the preparation of ZnO films by using both up/down and rotational control of the machine. The dipping speed of glass substrates into chemical bath, dipping time of the substrates in the chemical bath, withdrawing speed of the substrates from the chemical bath, time taken to move from one chemical bath to other and number of dipping cycles were properly controlled by user interactive programs developed in C++. Four experimental methods were followed for preparation of ZnO films to change the adsorption rate and reaction rate. Totally 64 film samples were prepared in all four methods. Among them, we carefully have chosen 8 samples i.e, 4 samples with different dipping cycles and same annealing temperature & 4 samples with different annealing temperature and same dipping cycles in each method for characterization. These samples were undergone the weight deposited, thickness measurements, transmission and absorbance for bandwidth calculation, XRD, SEM and EDAX studies.

The deposited weight in the substrate varies with dipping cycles, annealing temperature and the method. It is observed that the deposited weight increases with increasing the dipping cycles in each method. But the deposited weight decreases with
increasing annealing temperature in all four methods. It also depends on the methods in which the films are prepared. The method II records minimum deposited weight 1.57E-05 g/cm² per dipping cycle. The method III shows the maximum deposited weight 3.18E-05 g/cm² per dipping cycle because it has more adsorption rate and reaction rate due to the presents of EDTA and hydrazine hydrate. Thickness of the film formed on the glass substrate depends on the dipping cycles, annealing temperature and method. Thickness of the samples increases with increase in dipping cycles and decreases with the annealing temperature of the samples in all four methods. The sample with minimum thickness (0.7 µm) is formed out of the method II and the maximum thickness obtained (1.4 µm) in the method III. The increase in the thickness the method III is due to the presents of chelating agent.

The XRD studies reveal that the ZnO thin films prepared in all four methods show hexagonal wurtzite structure. And also observed that the crystallization increases with the dipping cycles and annealing temperature in all four methods. The SEM micrographs show that the spherical along with the spongy clusters at low annealing temperature. When the annealing temperature increases the clear cylindrical grains are observed in all four methods. The method III and IV shows the formation of clear grains at higher annealing temperature. EADX study reveals that the film prepared in the method II has the maximum Zn mass percentage (72.85%). The method I records the maximum atomic percentage of oxygen (62.3%).

In optical study the absorbance and transmittance spectrum were taken for all the samples. It reveals that the absorbance increases with increase in dipping cycle and remains unchanged with annealing temperature in all four methods. The sample with 20 dipping and annealing temperature 673K has minimum absorbance and for 50 dipping and annealing temperature 673K has maximum absorbance. The band gap of samples varies with the dipping cycles and annealing temperature. The decrease in band gap of ZnO film may be attributed to the improvement in crystalline quality along with the reduction in the porosity and increase in grain size.

Thermal study shows that variation in resistivity, TCR and the figure of merit among the samples. The dipping cycles and annealing temperature are the main factors
which changes the resistivity of the samples. The sample with the minimum dipping cycles and minimum annealing temperature has the maximum resistivity in all four methods. The TCR is high for the samples with minimum dipping and minimum annealing temperature in all four methods. The figure of merit of the samples also varies with the dipping cycles and annealing temperature in all four methods. The sample with 50 dipping cycles and 673K annealing temperature in method IV shows minimum figure of merit.

By examining the thermal behavior of selected films which are more suitable for the fabrication of the temperature sensor are identified. A temperature sensor was fabricated using a small glass air tight bottle with copper connecting wire. Using the temperature sensor, op-amp linearizing circuit and digital voltmeter, a digital thermometer based on ZnO films was constructed and their behavior was analyzed. During the testing period it is observed that ZnO films were also sensitive to the humidity. With very little modification, we can fabricate humidity sensor and suitable circuit can also be constructed for identifying the atmospheric humidity.

The fabricated instrumental setup is very useful for film fabrication with different dipping time, dipping speed, position of substrate in the chemical bath, with-drawing speed and dipping cycle. The same setup can also be used for optimizing the film fabrication by changing the dipping length and dipping time of the substrate into chemical bath. The High voltage or high magnetic field circuits can also be attached to the machine to influence the film formation with slight modification in the machine setup. Three or four chemical solutions can be used for film formation by changing the data of the user interactive programs and they can be placed appropriately on the rotating base. The audio indicators such as buzzer and visual indicators like LED can be attached to machine, which are very useful during film fabrication. The additional attachments can be added with up/down moving pulley and rotating base upto 7 Kg and 3 Kg respectively.