CHAPTER 3

Experimental Setup

3.1 Experimental Setup

A lot of methods are used to synthesis ZnS nano particles. Chemical precipitation is widely used for the preparation of colloidal nanoparticles. Cluster formation is very less in this method compared to the other methods. Simple thermal chemical reaction vapor transport deposition method is another method to produce nanoparticles. In this experiment the two methods were used.

3.1.1 Synthesis of ZnS nano particles chemical precipitation method.

In this work ZnS:Mn nanoparticles were synthesized by using three chemical compounds which are Zinc sulfate, Sodium sulfide and Manganese sulfate. The solutions were prepared in distilled water with concentrations of 0.5126M, 0.069M and 1M respectively. The previous concentrations were obtained by dissolved 22.11g of $ZnSO_4$ in 150 ml of distilled water, 0.47g of Na_2S in 50ml of distilled water and 1.7g of $MnSo_4$ in 10ml of distilled water. The solutions were first refluxed for an hour separately. So, 150ml of $ZnSO_4$,50 ml of Na_2S and 10 ml of $MnSO_4$ were prepared.

Three kinds of different samples had been synthesized in the test. Sample number one was prepared by adding 50 ml of $ZnSO_4$ to 6 ml of Na_2S which was continuously refluxed to get a colloidal form of ZnS. The colloidal sample was refluxed for 20 min at 80°C for uniform distribution of the particles. 1 ml of $MnSO_4$ was then added into 50 ml

ZnS colloid (which is already prepared). The colloidal sample was again stirred for another 30 min at 80°C. Then this colloidal was filtered out and washed with distilled water and ethanol for removing the additional impurities formed during the preparation process. The filtrate was dried at 100°C for 4 h. The previous steps for sample number one were repeated two more times for other samples but with $MnSO_4$ concentration of 2M and 3M.

. The replacement reaction can be written as follows:

$$ZnSo_4 \leftrightarrow Zn^{2+} + So^{2-}$$

$$N(Zn^{2+} + Na_2S) \leftrightarrow (ZnS)_N + 2N(Na^+)$$

The prepared product was characterized and analyzed using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDX).

3.1.2 Synthesis of ZnS nanoparticels by vapor transport deposition method

ZnS nanostructures are grown on silicon (100) wafers by using of a simple thermal chemical reaction vapor transport deposition method by heating the zinc and sulpher powders. There is no other metal catalyst in the process. The experimental system consists of a horizontal tube furnace (110 cm long), temperature controller, gas supply and control system. One side of the horizontal tube was connected to a vacuum pump and its other end is linked with gas supply and control system (fig 3.1). The source materials are high-purity of Zn (98%) and S (98%) powders in molar ratio of 1:1. In the experimental process, small quartz boat (2.5 cm diameter, 10 cm long) was placed into the horizontal tube and pushed to the center of the furnace (which has the maximum point of temperature). Silicon substrate pieces (Si [100] were place 2 cm downstream from the center. The reaction was heated at $700^{\circ}C$ in the center and the temperature of the substrate region was about $500^{\circ}C$ due to the temperature gradient. The Ar gas (99.9%) had been introduced as the carrier with flow rate was 60 standard cubic centimeters per minute (s.c.c.m). The heated at $700^{\circ}C$ was continued for 15 min.

Finally the furnace was turned off and the quartz tube was cooled down to room temperature. White color products were formed on the surface of the silicon wafer.

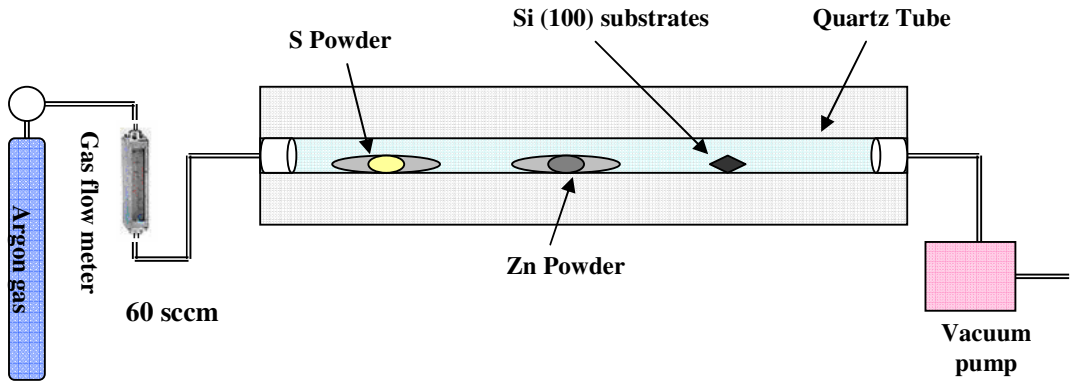

Fig. 3.1 Schematics showing the synthesis of ZnS nanoparticels by vapor transport deposition method