CHAPTER TWO
THE EXPERIMENTAL PART

2-1 Introduction:

This Chapter will present the experimental setup and the function of each component that was used to deposit a film. In addition to that the material used to fabricate thin film and the procedure of fabrication will be presented as well.

2-2 The Experimental setup:

The experimental setup that was used in this study to fabricate thin films from liquids and measuring their thicknesses is shown schematically in Fig. (2.1)

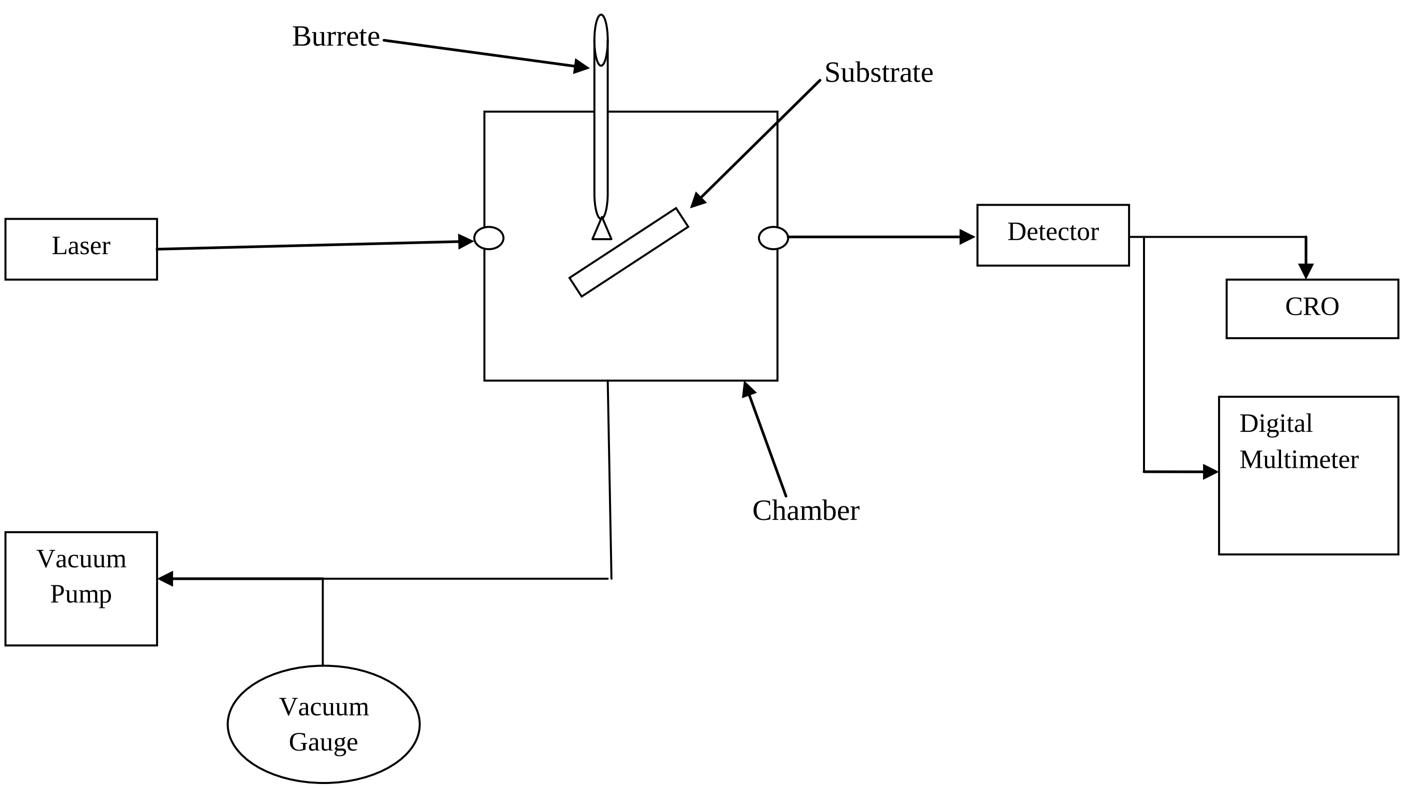

Fig.2.1: Schematic diagram of the experimental setup

2-2-1 Vacuum chamber:

It is a cubic box made of zinc sheets, with dimensions of 22*22* 22 cm, with two circular windows at 3 cm from the center of two faces in the parallel sides of the chamber. Each window is made of glass (1 mm thickness) and 1.5 cm radius. The first window is for the laser input and the other is for the detector.

A small hole in the down side of the chamber is made to connect the chamber to the vacuum pump. It also has a circular exit in the up side, 6 cm radii, which was connected to 10 ml burrete that can be used to flow the sample into the substrate inside the vacuum chamber. In the end of the burrete there was a funnel connected with it by plastic channel, this funnel was used to deposit the sample over large area of the glass substrate. The glass substrate was put with 45 degree angle and it was hold in a stand made from iron. In its end there was a magnet to avoid movements of the substrate. The two windows and the up exit were sealed carefully with silicon rubber before turn on the vacuum pump.

2-2-2 The vacuum pump:

The vacuum pump is a pump that removes gas molecules from a sealed volume in order to leave behind a partial vacuum (Van Atta et al., 1991). Figure (2.2) shows the vacuum pump which was used in this work, it was supplied from Motor division –ST. Louis, Mo. U.S.A., table (2-1) illustrate the pump specifications:

Table 2.1: Specifications of the vacuum pump:

Model	C55JXHJE
HP	R.P.M 1425/1725 S.F
VOLT	110-115/220-250 V
Hz	50/60
PH	1 CODE G
A	5.8-5.3/2.9-2.7 S.F.A
AMB	40 ^{o}C
TIME RATING	CONT

Fig.2.2: Vacuum pump photograph

Pumping speed refers to the volume flow rate of a pump at its inlet, often measured in liters per second, cubic feet per minute or cubic meter per hour. Because of compression, the volume flow rate at the outlet will always be much lower than at the inlet. Momentum transfer and entrapment pumps are more effective on some gases than others, so the pumping speed can be simultaneously different for each of the gases being pumped, and the average pumping speed will vary depending on the chemical composition of the gases remaining in the chamber (Lesker Tech, 2002).

Throughputs refers to the pumping speed multiplied by the gas pressure at the inlet, and is measured in units such as torr-litters/seconds. At a constant temperature, throughput is proportional to the number of molecules being pumped per unit time, and therefore to the mass flow rate of the pump. When discussing a leak, back streaming or out gassing, throughput refers to the volume leak rate multiplied by the pressure at the vacuum of the leak, so the leak throughput can be compared to the pump throughput (Van Atta et al., 1991).

2-2-3The vacuum gauge:

In general, the special devices which measures the vacuum levels are called vacuum gauges. The simplest mechanical gauge you can imagine as a chamber divided by a membrane.

One side of the chamber is connected to the vacuum container and the other to the known pressure tank (it can be simply open, therefore it will be under the atmospheric pressure). The membrane is attached to the pointer mechanism. Depending on the membrane deformation, the pointer will show the vacuum level. A gauge like this is considered to be a coarse or rough gauge.

The vacuum gauge that was used in this work has two ranges, the first one by Bar and the other is by Pascal. It was used it to measure the pressure inside the vacuum chamber (Van Atta et al., 2015).

2-2-4 The samples:

The sample used to deposit a thin film on a glass substrate was $CuSO_4$ with structure shown in figure (2.3), Copper (II) sulfate, also known as cupric sulfate or copper sulphate, is the chemical compound with the chemical formula $CuSO_4$(Andrew et al., 1999).

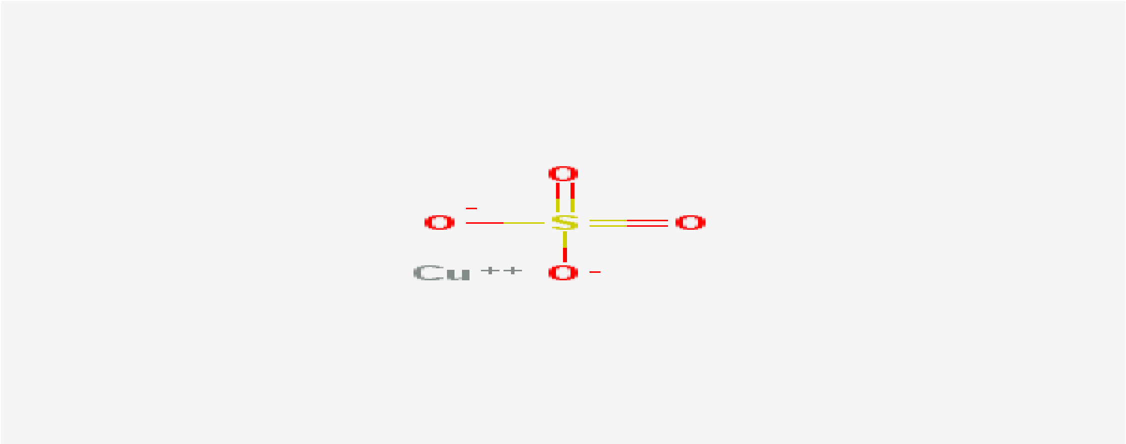

Fig.2.3: $CuSO_4$ structure

This salt exists as a series of compounds that differ in their degree of hydration. The anhydrous form ($CuSO_4$) is a pale green or gray-white powder, whereas the pentahydrate ($CuSO4 \cdot 5H_2O$), the most commonly encountered salt, is bright blue. Copper (II) sulfate exothermically dissolves in water to give the aqua complex $[Cu(H_2O)_6]^{2+}$, which has octahedral molecular geometry and is paramagnetic. Other names for copper (II) sulfate are "blue vitriol" and "bluestone" (Brian Bennett,1998). Copper (II) sulfate has the following properties which are listed in table (2. 2).

Table 2.2: Copper Sulphate properties:

Chemical formula	CuSO4 (anhydrous) $CuSO4 \cdot 5H_2O$ (pentahydrate)
Molar mass	159.609 g/mol (anhydrous)[249.685 g/mol (pentahydrate)
Appearance	gray-white (anhydrous) blue (pentahydrate)
Density	3.60 g/cm3 (anhydrous) 2.286 g/cm3 (pentahydrate)
Melting point	110 °C (230 °F; 383 K) decomposes ($\cdot 5H_2O$) <560 °C decomposes
Solubility in water	1.055 molal (10 °C) 1.26 molal (20 °C)
Solubility	Anhydrous: insoluble in ethanol Pentahydrate: soluble in methanol 10.4 g/L (18 °C) insoluble in ethanol

Refractive index (n_D)	1.724–1.739 (anhydrous) 1.514–1.544 (pentahydrate)

Figure (2.4) shows an absorption spectrum of $CuSO_4$ (and H_2SO not interested in this work).

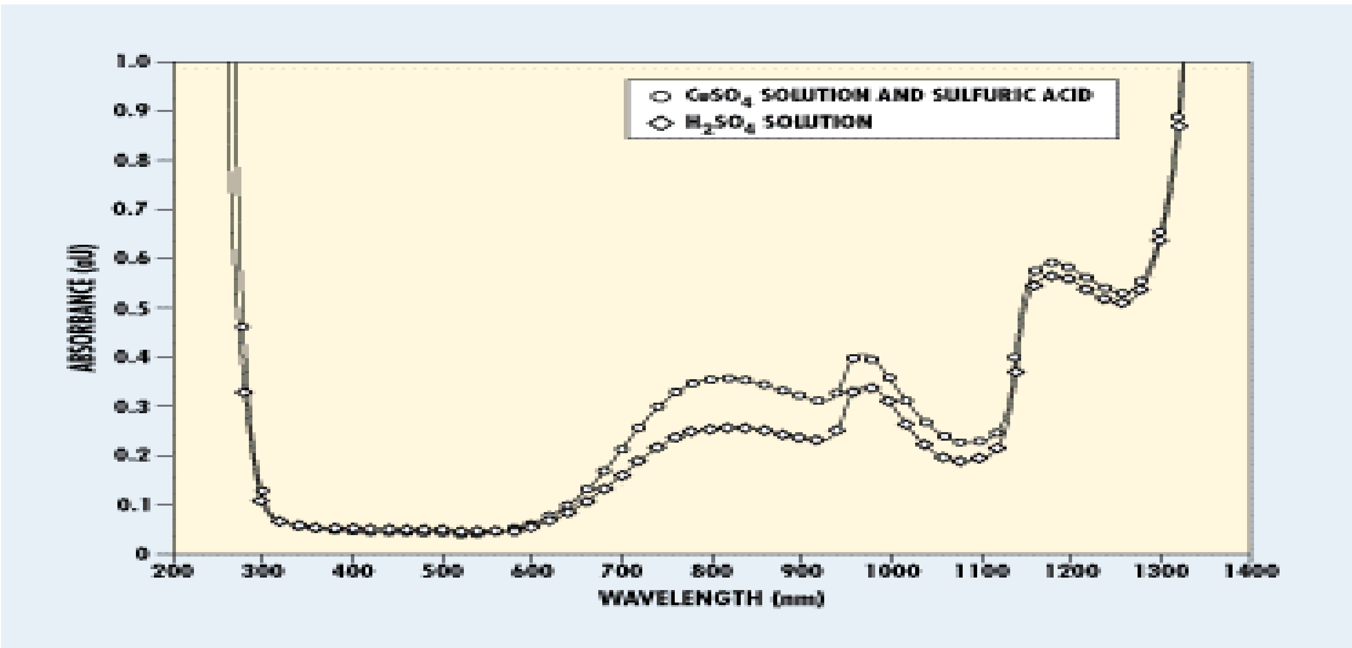

Fig.2.4: Absorption spectrum of $CuSO_4$ (it's not important in this work the Spectrum of H_2SO)

2-3 Lasers and light sources:

Different laser and light sources were used to measure the thicknesses and the optical properties of the deposited thin films.

2-3-1 Helium –Neon (He-Ne) laser:

The most common and inexpensive gas lasers the He-Ne laser .Laser emission in the He-Ne laser occurs from excited levels of neutral Ne atoms that have been populated by collision with excited He atoms. The relevant energy levels are shown in Figure (2.5) (Walter, 1983).

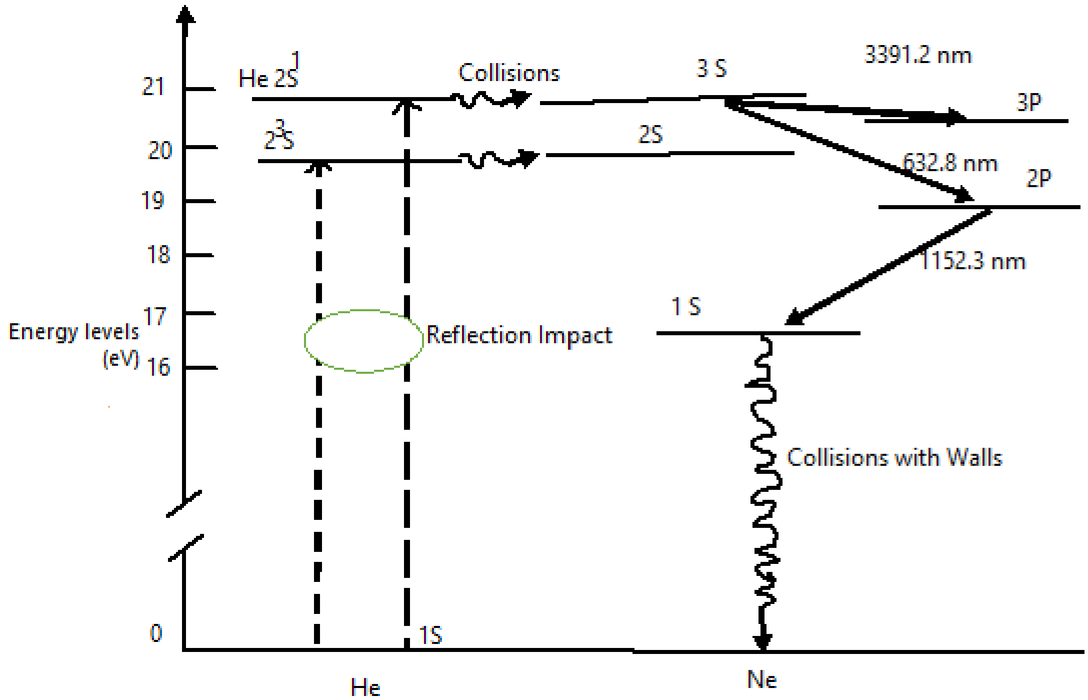

Fig.2.5: Outline of the energy levels involved in the He-Ne laser. Although single states are shown for Ne, the levels actually contains many states.

The three dominant laser lines are at 632.8, 1152.3, and 3391.2 nm. The line at 1152 nm was the first observed from a neutral gas laser. Nowadays most He-Ne lasers are constructed to emit at 632.8 nm. The wavelength at which oscillation occurs can be controlled by variation of the coating applied to the laser mirrors or by application of an external inhomogeneous magnetic field (Walter, 1983). It is particularly necessary to suppress oscillation on the 3391-nm transition if operation at 632.8 nm is desired since a high cavity intensity in the 3391-nm line tends to deplete the population in the upper state for the 632.8-nm line. The He-Ne laser is usually operated in a mixture of He and Ne with proportions of 10: 1 and at a total pressure of up to several torrs. Under these conditions, the spontaneous linewidth of the 632.8-nm line is limited by the Doppler width at ~ 300 K, the ambient temperature of the laser tube.

Helium-neon gas laser used in this work was manufactured by PHYWE SYSTEM GmbH Company, Germany, and its specifications are listed in table (2.3).

Table 2.3: Technical specifications of He-Ne laser:

Wavelength	632.8 nm
Power energy	1 mW
Type	CW
Beam diameter	0.5- 1.7 m rad
Divergence & Efficiency	< 0.1%
Classification	Class I

2-3-2 Omega laser XP:

Omega laser is a diode laser with different probes, each provides certain wavelength and power. The omega XP is housed in a rigid metal case. Soft rubber pads are designed to prevent the unit from slipping on hard smooth surfaces. The omega XP is managed by a microprocessor and displays the latter on an alphanumeric screen.

Omega laser used in this work is supplied with three probes, the first with wavelength 675 nm, CW mode of 30 mW output power, the second probe with wavelength 820 nm, CW mode of 200 mW output power, and the third probe with wavelength 915 nm, CW mode of 200 mW output power. It was manufactured by Omega Company-England, and its specifications are listed in table (2.4), (Biotechhealth, 1996).

Table 2.4: Technical specifications of Omega XP:

Size:	210 X 130 X 130 mm
Pulsing frequencies:	2.5 Hz, 10 Hz, 20 Hz, 73 Hz, 146 Hz, 700 Hz, 1 KHz, 5KHz, 10 KHz
Battery life:	Cluster probe 4 hours; single probes up to 10 hours
Battery:	Rechargeable Metal Nickel Hydryde 4.1 Ah

Specifications of the visible red (675 nm) probe:	
Wavelength:	675 nm
Coherence:	Laser
Power:	30 mW
Power Density:	0.24 W/cm^2
Classification	Class III-B
Specifications of infrared probe 820 nm:	
Wavelength:	820 nm
Coherence:	Laser
Power:	200 mW
Power Density:	1.60 W/cm^2
Classification	Class III-B
Specifications of infrared probe 915 nm	
Wavelength:	915 nm
Coherence:	Laser
Power:	200 mW
Power Density:	1.60 W/cm^2
Classification	Class III-B

2-3-3 Diode Laser 532 nm:

A diode laser, is an electrically pumped semiconductor laser in which the active laser medium is formed by a p-n junction of a semiconductor diode similar to that found in a light-emitting diode. The laser diode is the most common type of laser produced with a wide range of uses that include, but are not limited to, fiber optic communications, barcode readers, laser pointers, CD/DVD/Blu-ray Disc reading and recording, laser printing, laser scanning and increasingly directional lighting sources (Walter, D.W., 1983); (K. Thyagarajan & Ajoy Ghatak, 2010).

Table (2.9) lists the technical specifications of this diode laser, at 25 ^{o}C:

Table 2.6: Specifications of laser diode 532 nm:

Wavelength	532 nm
Beam size type $1/e^2$	1.2 mm
Beam circularity	≤ 1.3:1
Beam divergence	≤2.0 m rad
Mode	TEM_{00}
Operating voltage	4-6 V DC
Operating Current (25 ^oC)	< 300 mA

2-3-4 Aiming beam (LED) of Q- switched Nd: YAG laser:

This is a light emitting diode (LED) used to aim the beam of Nd: YAG laser and to simplify the alignment of the beam to the desired destination, there are various aiming beams for Q-switched Nd: YAG lasers some of them are diode laser with wavelengths (635 nm), (655 nm), etc. This device is very benefit since the energy supplied by the Q-switched Nd: YAG are in the IR mainly.

The aiming beam used in this work was manufactured as the aiming beam of the Q-switched Nd: YAG laser (Model Name HS 220 E) manufactured by Shanghai Apolo Medical Technology Co. ltd (Shanghai Apolo Medical Technology Co. ltd, 2011).

The aiming beam of this device which was used in this study is a LED emitting 660 nm wavelength, with spot size from 1-3 mm .

2-3-5 Sodium vapor lamp:

A sodium-vapor lamp is a gas-discharge lamp that uses sodium in an excited state to produce light. Low-pressure sodium lamps only give monochromatic yellow light and so inhibit color vision at night .

Sodium emits many lines in the visible region of the E.M. spectrum. The dominant yellow emissions they are probably referring to the doublet located at: 588.9950 nm and 589.5924 nm having a relative intensity of about 2:1(David Latchman, 2015).

Sodium lamp that was used in this work emits (589.2) nm and was manufactured by Phillips, some of it is specifications are listed in table (2-7) (Phillips, 2010).

Table 2.7: Specifications of sodium lamp 93122:

TECHNICAL DETAILS: **Product type**: Lamp/Bulb **Technology**: HID/ HIGH INTENSITY DISCHARGE **Type**: Sodium Spectral Lamp	
ELECTRICAL PROPERTIES: **Watts**:18 W **Volts**: 50 V	
PHYSICAL CHARACTERISTICS: **Shape**:T9 1/2 **Base:** E26/E27/ MEDIUM SCREW **Ends**: Single ended **Diameter (mm/''):** 30 MM	
LIFE: 500 H	**PRODUCT NUMBER**: 93122

2-4 The Photo-detector:

Photo-detector is a device that converts the incident optical radiation into an electrical current or voltage which is proportional to the light intensity. One example is the PIN photodiode.

The PIN photodiode uses an extra high-resistance layer, I, between the p and n layers to improve response time. The light penetrates through the thin P^+ layer to the intrinsic layer, I, where it is absorbed. The speed of movement of the generated charges is high due to the electrical field of the depletion region. The response time of the PIN photodiodes is of the order of several nanoseconds (Pavel Ripka &Alois Tipek, 2007).

A silicon pin photodiode (PHYWE Naturwissenschaft und Technik- Germany, 2007) was used in this work for detecting light from laser sources.

The pin photodiode respond over the approximate range of 0.4 to 0.75μm. The frequency response of pin photodiode is of order of 1010 Hz. The high resistance incident layer assists greatly in the production of photocurrent from incident photons (Ready-1997).

2-5 The Digital Oscilloscope (CRO):

One of the most important application of the electrostatic focusing and motion of the charged particles in electric and/or magnetic field is cathode ray oscilloscope. This oscilloscope is one of the most important tools for the developments of electronic circuits and for modern electronics (Uma Mukherji-2007).

C.R.O is used as a tool for display of the waveforms as well as for making many kinds of measurement quickly and almost accurately. It is not particularly a very accurate measuring instrument but is best used where only approximate and quick measurement is required (Uma Mukherji, 2007).C.R.O. that was used in this work was manufactured by Tektronix Company, and its specifications were listed in table (2.8) (Tektronix, 2007).

Table 2.8: The specifications of C.R.O.:

Voltage range	Frequency	Power max.	VA max.
100 - 120 V	45 - 440 Hz	25 Watts	35 VA
120 - 240 V	45 - 66 Hz		

2-6 The Digital Multimeter DT-700D:

DT-700D is new style mini –type pocket size digital multimeter and it is widely used in our life for Multi-use with AC/DC voltage and resistance measurement. The DT-700 D used in this work was manufactured by Globalmarket, China (globalmarket.com, 2015). Table (2.9) Lists The DT-700 D Basic Specifications.

Table 2.9: DT-700 Multimeter basic specifications:

Measuring Current Range	2000 μA-10A
Measuring Voltage Range	DCV:200mV-500V,ACV:200-500V

Measuring Resistance Range	200Ω-2MΩ
Product size	126*70*26 cm

2-7 Setup arrangement:

Figure (2.6) shows a photograph for the setup arrangement for the deposition of liquid, and monitoring their thickness.

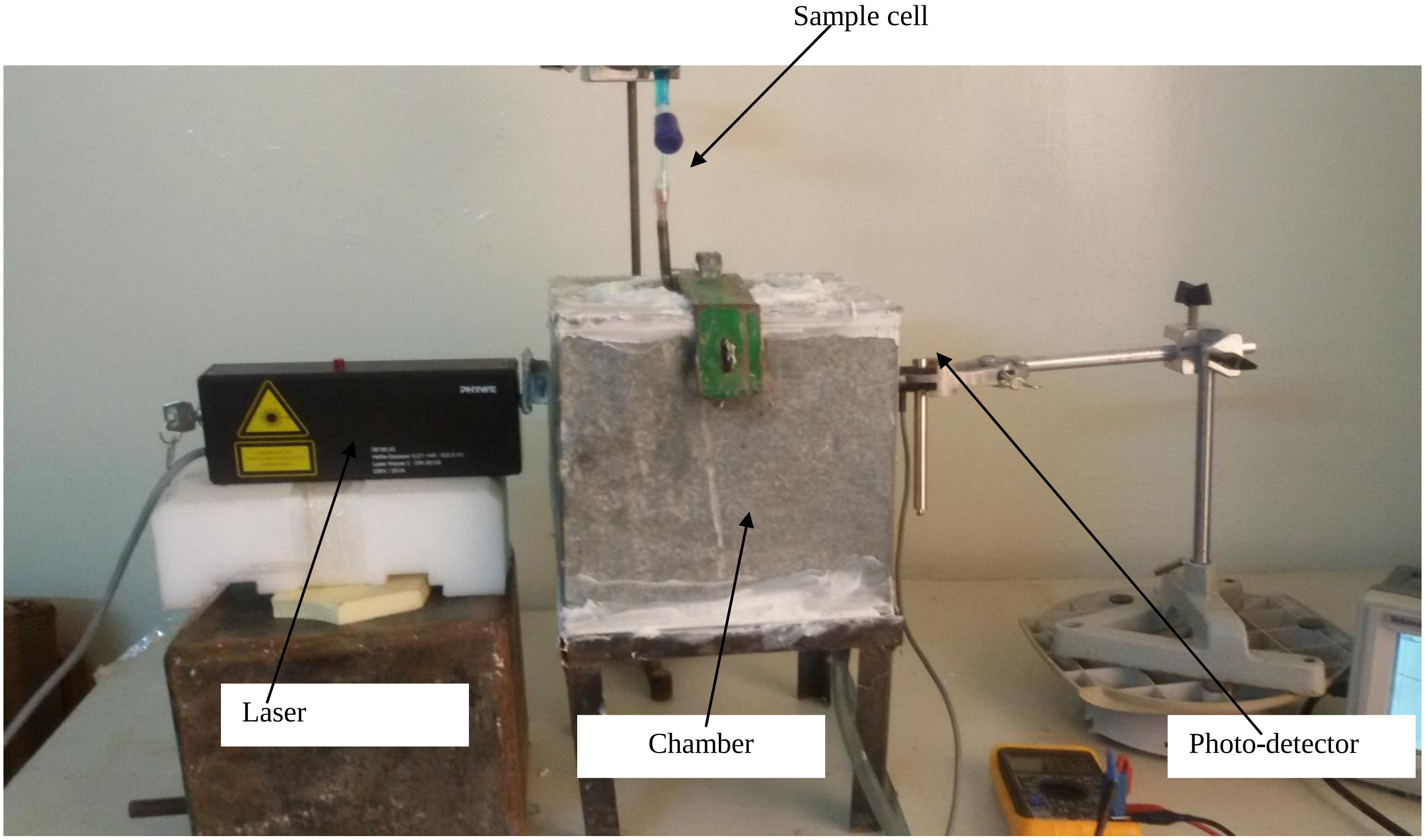

Fig.2.6: All components and the experimental setup for He-Ne assisted deposition

2-8 The Experimental procedure:

The experimental procedure will be summarize as follows:

1- According to molecular weight 12.6 gm of $CuSO_4$ powder was dissolved in 50 ml distilled water in order to get a 1M concentration of the solution.

2- Then, the experimental setup was arranged as shown in figure (2-4) for He-Ne assisted liquid deposition and as in figure (2.5) for green laser assisted deposition.

3- The sample holder was inserted in the vacuum which hold the glass substrate at an angle of 45^o, and was closed carefully after that.

4- The vacuum pump was turned ON to evacuate the chamber and the pressure was measured inside the chamber after 10 minutes by the vacuum gauge.

5- Helium-Neon laser was turned ON and then ($\mathbf{I_o}$) was measured by digital multimeter and the signal was detected by C.R.O.

6- The sample was deposited slowly from the burrete on the glass substrate, and then the laser intensity was detected by the C.R.O. during deposition until the appearance of the interference fringes, which indicate the desired thickness. The transmitted intensity of laser (**I**) was measured .

7- Thicknesses of thin film was calculated from interference fringes, that equal half multiple of the wavelength of the laser source wavelength, either in case of the green laser or He-Ne laser.

8- The transmission spectrum of the fabricated thin film for each thickness were tested using different laser sources, sodium light, and aiming beam (LED 660 nm) of the Q-switched Nd: YAG laser.

9- The refractive index for the thin films (μ) at a specific wavelength of different thicknesses were calculated using the measured reflectivity R and the glass refractive index μ_s according to: (Elhadi, 2007).

$$\mu = \left(\frac{\mu_s[1+\sqrt{R}]}{1-\sqrt{R}}\right)^{\frac{1}{2}}$$
$$\mu_s = \frac{1}{T_s}\left(\frac{1}{T_s^{\,2}} - 1\right)^{\frac{1}{2}} \qquad (2\text{-}1)$$

where T_s represents the transmission of glass substrate.

10- The absorption coefficient of the films at specific wavelength was deduced from the measured value of reflectivity R, transmittance T, refractive index ns, and thickness t according to :(Elhadi, 2007); (Mousa & Ponpon, 2006).

$$\alpha = \frac{1}{t}\mu\frac{(1-R)^2}{T} \qquad (2\text{-}2)$$