DATA ACQUISITION AND CONTROL SYSTEM FOR PULSED LASER DEPOSITION UNIT

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Master of Technology

By

M. S. Ansari

Roll No. 98307403

Under the Guidance of

Prof. A. N. Chandorkar

and

Prof. P. C. Pandey

Department of Electrical Engineering

Indian Institute of Technology, Bombay

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Guide: Arun M. Chandorkar

Co-Guide: R Pandey

Internal Examiner: [illegible]

External Examiner: [illegible]

Chairman: [illegible]

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ABSTRACT

The objective of this project was developing a PC based data acquisition and control system for pulsed laser deposition of thin films. The deposition unit is being set up at the Centre for Advanced Technology, Indore, with an aim to develop semiconductor based electro-optical devices. The data acquisition and control unit measures and controls various parameters under which film growth takes place.

The measurement block includes an in-situ quartz crystal based film thickness monitoring unit. It utilizes the mass loading effect in oscillating quartz crystals. Difference in the acoustic impedance of the deposited film and the quartz crystal is taken into account while determining the film thickness. A water cooled crystal holder and a crystal oscillator with high stability is designed for measurement of the film thickness. Frequency deviation in the quartz oscillator due to mass loading effect is measured by heterodyning principle, by using an external reference oscillator.

Other parameters which, are monitored during the deposition process are chamber vacuum, substrate temperature and laser pulse energy. The control system includes drives for two stepper motors, for rotating the target material and to operate a laser blocking shutter. Sensors and stepper motor drives are interfaced to a PC through an add-on card, consisting of analog-to-digital converter, digital-to-analog converter, buffers, analog multiplexers etc. Data to and from the add-on card are optically isolated by isolation amplifiers and opto-couplers, to minimize coupled noise through common ground. Control unit of the pulsed laser used in the deposition process is serially interfaced to the PC.

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Chapter 1

INTRODUCTION

1.1 OVERVIEW

Pulsed laser deposition scheme is a relatively new technique for deposition of thin films of different materials for use in electronics, opto-electronics and material science. It involves evaporation of a target material in vacuum by thermal energy generated by high power laser pulses. The vapor stream thus generated condenses on a substrate so as to form a homogenous and adherent deposit of desired thickness.

Precise measurement and control of various process variables in pulsed laser deposition system is important from the point of view of controlling the quality and characteristics of the film. Quality of the film depends on the substrate temperature, ambient pressure etc. Uniformity of the film depends, to a large extent on the target to substrate distance. For preparation of repeatable quality of films, it is imperative to have automatic data acquisition, monitoring and control of the substrate temperature, chamber vacuum, film thickness deposited on the substrate, deposition rate, laser energy and repetition rate. Apart from these, a pulsed laser deposition system requires motorized drive to continuously rotate the target material and to operate a mechanical shutter to block the initial laser pulse, till its energy stabilizes to the set value.

1.2 PROJECT OBJECTIVE

The project objective was to design and develop a PC based data acquisition and control system for pulsed laser based film deposition unit. The pulsed laser deposition unit is being set up at the Centre for Advanced Technology, Indore. This unit will be used to study and develop various types of opto-electronic materials and devices. The data acquisition and control system being developed has following measurement and control blocks.

1. An in-situ quartz crystal based thickness monitoring circuit that includes a water-cooled crystal holder, a stable monitor oscillator and circuit for measurement of frequency deviation.
2. Measurement circuit for sensing the chamber vacuum.
3. Scheme for controlling the substrate temperature.

4. Measurement of the laser energy.
5. Control of motorized drives for rotational movement of the target material and operation of a laser-blocking shutter.
6. An add-on card containing Analog to digital converter (ADC), Digital to analog converter (DAC), Counters, Buffers etc. to enable the operation of the above mentioned functions through a PC.

Development of a quartz crystal based thickness monitoring unit for the pulsed laser deposition system was taken up, in-house, instead of using commercially available monitors for the following reasons.

(a) Need for multiple thickness monitors: Some of the planned experiments require information about angular distribution of the target plumes. This may require use of around six sensors placed uniformly around the target. Signals from all these sensors will have to be processed by the data acquisition unit.

(b) Film deposition by simultaneous evaporation of more than one target: For certain applications, the fabrication of films by simultaneous evaporation of two or more targets is required. This necessitates development of specific, monitor crystal holders and separate control of the deposition rate of each source.

Commercial thickness monitors, currently available in the local market do not have provisions for interfacing multiple sensors. Further more, an in-house developed monitoring unit will be more flexible for integration with other blocks in the overall data acquisition system for the pulsed laser deposition setup and for any future up-gradation.

1.3 DISSERTATION OUTLINE

Chapter 2 describes the basic scheme of pulsed laser deposition (PLD) technique. A comparison is made with other methods for film deposition. Specifications and other details of the laser unit being used in the PLD scheme are highlighted. Various process variables to be monitored and controlled are introduced. Sensors and measurement principles for three of the parameter in the PLD process, namely laser energy, chamber vacuum, and substrate temperature are described.

Chapter 3 discusses the techniques for measurement of the film thickness, and describes in detail the quartz crystal based scheme, which was used in the project. Evolution of Z-match technique for determination of film thickness deposited on a quartz crystal is described next. Section 3.2 discusses the design of monitor oscillator and its performance in respect to the frequency stability. Design of instrumentation for film thickness measurement, namely monitor crystal holder and circuit for detection of the frequency deviation due to mass loading are discussed in section 3.3. The experimental results for film thickness is discussed in section 3.4.

Chapter 4 presents the measurement and control issues for the process variables in the deposition set up. It also describes the interfacing of stepper motors for target rotation and for operation of a laser blocking shutter.

Chapter 5 summarizes the project and highlights the issues with scope for further improvements.

Chapter 2

PULSED LASER DEPOSITION UNIT

2.1 INTRODUCTION

Pulsed laser deposition (PLD) is a technique to deposit thin films from a variety of materials such as metals, semiconductors, insulators, dielectrics etc. Fig. 2.1 shows a general scheme of pulsed laser deposition unit. Train of high power laser pulses is focused on the target surface, placed in a vacuum chamber, by a set of optical components. Flash lamp pumped Nd:YAG laser operating at the wavelength of 1.06 μm and excimer lasers in the ultraviolet regime, are the most popular sources used in PLD setups. When the laser radiation is absorbed by the solid target, electromagnetic energy is first converted into electronic excitation and then into thermal energy to cause evaporation. Evaporants form a plume consisting of a mixture of energetic species including atoms, molecules, electrons, ions, micron-sized solid particulate and molten globules. The collisional mean free path inside the dense plume is very short. As a result, immediately after the laser irradiation, the plume rapidly expands into the vacuum from the target surface to form nozzle jet with hydrodynamic flow characteristics. The vapor stream thus generated condenses on a substrate to form a homogeneous and adherent deposit.

Some of the other techniques available for thin film deposition are,

(a) Sputtering of cathode materials in presence of inert or active gases either at low or medium pressure.

(b) Molecular beam epitaxy (MBE).

(c) Chemical vapor deposition (CVD) by pyrolysis, dissociation or reaction in vapor phase.

(d) Chemical deposition from solutions including Electro-deposition, anodic oxidation etc.

(e) Thermal deposition in vacuum by resistive heating.

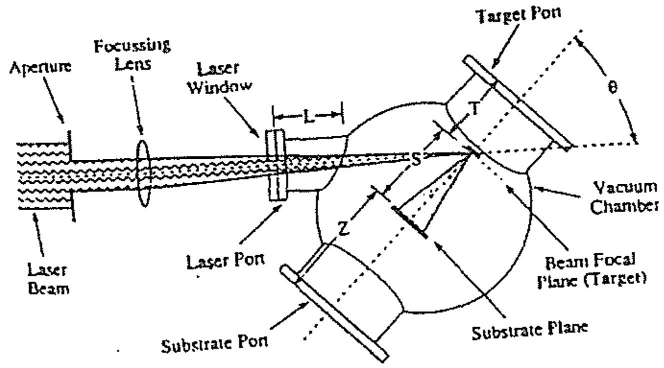


Fig. 2.1 Pulsed laser deposition unit

However pulsed laser deposition scheme has certain advantages as compared to other deposition techniques. Main advantages are,

(i) Dissimilar components of a target can be deposited at the same rate. This property makes the process ideal for deposition of complex multi component super conducting and electro-optical materials.

(ii) Compared to conventional techniques like CVD, in which, the substrate is exposed to a gas mixture that reacts to form the desired film [1], the pulsed laser deposition scheme limits thermal distortion of the substrate and generates a cleaner environment, which results in a considerably purer film deposition [2].

(iii) Localization of heat prevents the gas reaction from occurring in regions remote from the desired deposition sites.

(iv) Pulsed laser deposition units are capable of depositing stoichiometrically correct, highly oriented material without a post annealing process [3].

Disadvantages of pulsed laser deposition scheme are the presence of micron-sized particulates and the narrow forward angular distribution that makes large area scale up a very difficult task [2].

2.2 LASER UNIT

The laser system being used in the deposition system has three main parts: Optical head, power module and control system.

The optical head contains the laser oscillator, the laser amplifier and other optical components. The laser oscillator is an optically pumped, Q-switched, Nd: YAG based system. The Nd: YAG rod is of 6 mm diameter and 115 mm length. It is pumped by a single flashlamp. Its output is coupled to the laser amplifier, which has 9 mm diameter Nd: YAG rod and pumped by two flash lamps.

Power supply module of the laser system contains all the necessary components for controlling and powering the oscillator and amplifier stages. It delivers electrical energy to the oscillator and the amplifier flash lamps. This is accomplished by charging several banks of energy storage capacitors. General specifications of the laser system are,

- Energy per pulse: 150-360 mJ
- Repetition rate: 10 to 50 Shots per second
- Pulse duration: 5 ns
- Energy stability: 2%

The control module offers full operational control of the laser system. The main functions are programmable by an external PC through serial communication port. The communication parameters are:

9600 BAUD, 8 Bit, 1 Stop bit, No parity.

Some of the relevant commands are given below. These are in ASCII string and each of these is terminated, by a carriage return character.

M: Initiate simmer current in the flash lamp

A: Fires flash lamp in auto mode

Vxxxx: Reads the flash lamp repetition rate

F: Reads the flash lamp shot counter

2.3 PROCESS VARIABLES AND MEASUREMENTS

Properties of the films of given materials depend on their growth conditions as well as on the film thickness. Thus monitoring and control of the growth conditions like substrate temperature chamber pressure etc. become essential, if films are to be made reproducible in every respect. The data acquisition unit being developed monitors following process variables.

a) Laser energy
b) Chamber vacuum
c) Substrate temperature
d) Film thickness

Besides, monitoring of these parameters, motorized drives are required to continuously rotate the target material and to open a laser-blocking shutter. The target material is rotated to avoid evaporation from a single point. The laser blocking shutter is used to allow laser to be incident on the target only when its energy stabilizes to, 2% within the set value.

Pyroelectric detector is used to measure the laser energy. These detectors have the advantages of broad spectral coverage and fast response. These are fabricated from thin slice of a material having a permanent electrical polarization i.e. a ferroelectric. The detector response results from dependence of polarization on temperature. A change in detector polarization will give rise to a displacement current in the detector material and a compensating current flow in the external measuring circuit equal to,

$$i = \int_A (dp/dt)da \qquad (1)$$

Where A is the electrode area, P is polarization and t is time. Equation (1) can be modified for an idealized case in which there is no heat loss to the detector's environment and radiation is uniformly absorbed directly in the detector material, the temperature will increase linearly in response to a constant flux, and the current is given by,

$$i = r_I E \qquad (2)$$

Where, r_I is the current responsivity (in amps/Joule) and E is the total incident energy. The current responsivity, r_I, in turn is given by,

$$\frac{\varepsilon\Lambda}{\rho C_p d} \qquad (3)$$

ε is the absorption co-efficient of the detector, ρ is density of the detector material, C_p is the material specific heat, d is the electrode separation and Λ is defined as the slope of the curve of polarization versus temperature, i.e. $\Lambda=dP/dT$.

The basic equivalent circuit of a pyroelectric detector is a current source in parallel with its own capacitance. To convert the low level current obtained, to a suitable voltage, a high impedance current to voltage pre amplifier is used.

Vacuum of 10^{-8} torr in the deposition chamber is achieved by a turbomolecular pump. Turbomolecular pumps [4] contain a rotor with inclined blades moving at high speed between corresponding stationary blades in a stator. Gas molecules entering the inlet port acquires a velocity and preferred direction, superimposed on their thermal velocity by repeated collisions with the fast moving rotor.

A hot cathode ion gauge is used to measure the chamber vacuum. It works on the principle of ionizing the gas and measuring the collected ion current as shown in Fig.2.2. Ionization is accomplished by acceleration of electrons emitted from a heated filament. The filament is biased at about +50 V so that the electrons will be repelled from the chamber wall, which is at ground potential. The electrons are accelerated towards the cylindrical grid, which is biased to about +180 V. The collected ion current is proportional to the electron emission current, the ionization cross section, and the gas concentration. The ionization cross section varies with gas composition and electron energy. Range of vacuum that the ion gauge can measure is from 10^{-3} to 10^{-10} torr. Sensitivity of the gauge is given by,

$$S = \frac{(I^+)^2}{I^- P} \tag{4}$$

Where, P is the pressure, I^+ is the ion current, I^- is the electron emission current.

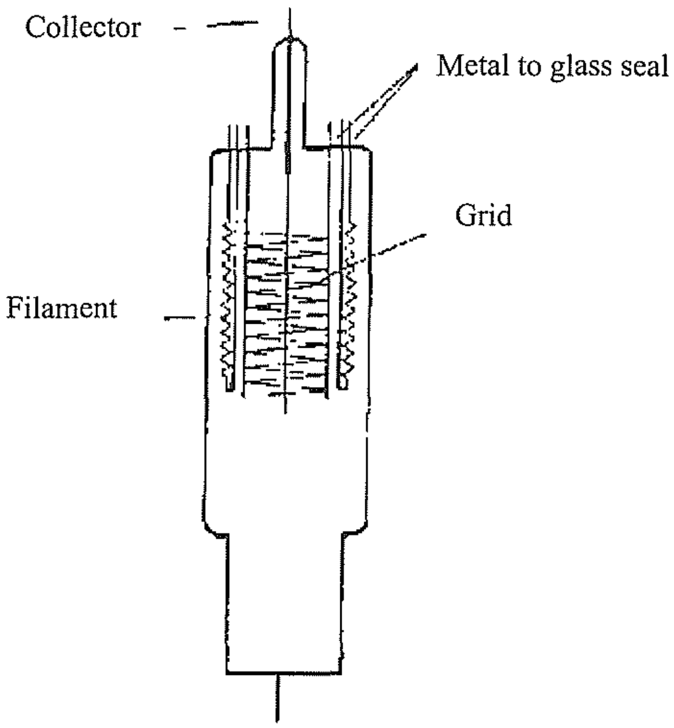


Fig. 2.2 Hot cathode ion gauge

Chapter 3

FILM THICKNESS MEASUREMENTS

3.1 TECHNIQUE FOR FILM THICKNESS MEASUREMENTS

The film preparation by any physical deposition technique requires in-situ monitors for controlling deposition rates and thickness. The deposition process should be automated since, continuous manual adjustment is difficult and cumbersome. The film thickness, which is an important parameter for many applications, should be measured simultaneously along with the rate of deposition. Several methods have been used for determination of film thickness and deposition rate. These are,

- Measurement of the resistance of the deposited metallic films [5].
- Determination of the optical transmissivity of metallic and dielectric films [6].
- Weighing of the mass of the film with microbalances placed in evaporation chamber [7].
- Determination of the change in oscillating frequency of a quartz crystal due to mass loading effect [8].

The quartz sensor, based on mass loading effect best serves our requirement of a reliable and in-situ thickness monitor.

It works on the principle of change in resonant frequency of a quartz crystal due to mass loading. Early investigations in this field [9] and [10] concluded that, for small mass changes of the oscillating quartz in a thickness shear vibration mode, frequency of a crystal upon which a thin film is deposited is linearly proportional to the deposited mass. The theoretical derivation of the mass load versus frequency equation given by Sauerbrey [9], is based on the substitution of the mass density Δm_Q of an assumed additional quartz layer by the mass density m_F of the deposited foreign material. This substitution is valid as long as the elastic properties of the foreign film do not contribute to the resonance frequency. In other words, thickness of the deposited material has to be very small compared with that of the crystal. Under the above-mentioned assumption, we obtain following relation between mass loading and the oscillator frequency change.

$$\frac{m_F}{m_Q} = \frac{f_Q - f}{f_Q} = \frac{\Delta f}{f_Q} \qquad (5)$$

Where, m_F, is mass of the deposited film, m_Q, is mass of the quartz crystal plate, f_Q and f are the resonance frequencies of the unloaded and loaded crystal respectively and $\Delta f = (f_Q - f)$ is the frequency change due to mass loading. Substituting m_F and m_Q in Equation (5) in terms of film density ρ_F, film thickness l_F, quartz density ρ_Q, thickness of the quartz crystal l_Q, and area A, results into,

$$\frac{\rho_F l_F A}{\rho_Q l_Q A} = \frac{\Delta f}{f_Q} \qquad (6)$$

Introducing the substitution $l_Q = (N_{AT} / f_Q)$, where N_{AT} is defined as frequency constant of the crystal, Equation (6) can be rewritten as,

$$l_f = \frac{\rho_Q N_{AT} \Delta f}{\rho_F f_Q^2} \qquad (7)$$

Equation (7) has been shown to be accurate only if $m_F/m_Q < 2\%$ [11]. As a further modification of this equation it was shown [12] that the period τ of the crystal oscillation increases in proportion to the mass loading according to,

$$\frac{m_F}{m_Q} = \frac{\tau - \tau_Q}{\tau_Q} \qquad (8)$$

Where, τ and τ_Q are the oscillation period of the loaded and unloaded crystal, respectively. Equation (8) is known as period measurement technique and it increases the usable mass load range (m_F/m_Q) to around 8%.

The last step in refinement of the proper mass load vs. frequency equation is based upon a one dimensional continuous wave acoustic analysis of the quartz/film composite resonator by Miller and Bolef [13]. It takes into consideration the acoustic impedances of quartz and the deposited material. The basic equation by Miller and Bolef is,

$$Z_Q \tan\frac{f}{f_Q}\pi + Z_F \tan\frac{f}{f_F}\pi = 0 \tag{9}$$

Where, f is composite resonant frequency of the loaded quartz, f_Q is the frequency of oscillation of unloaded quartz and f_F is the mechanical resonant frequency of the deposited film. Z_Q and Z_F are the specific acoustic impedances of the quartz crystal and the film, respectively. Substituting, $f_F=(V_F/2l_F)$ where, $V_F=(Z_F/V_F)$, is the shear wave velocity in the film with respect to the propagation of the piezoelectrically excited acoustic wave [11], Equation (9) results into,

$$\frac{f(2l_F\rho_F)\pi}{Z_F} = \tan^{-1}\left(-\frac{Z_Q}{Z_F}\tan\frac{\pi f}{f_Q}\right) \tag{10}$$

Equation (10) can be rewritten in terms of film thickness and the frequency deviation $\Delta f = (f_Q - f)$, as,

$$l_f = \frac{Z_F}{2\pi\rho_F(f_Q-\Delta f)}\tan^{-1}\left(-\frac{Z_Q}{Z_F}\tan\frac{\pi(f_Q-\Delta f)}{f_Q}\right) \tag{11}$$

Validity of equation (11) is experimentally verified for mass load m_F/m_Q of around 70% [11]. Crystal thickness monitors based on the above equation are known as Z-match technique devices.

An appropriate tooling parameter compensates for geometric factors in the deposition system, which results because of difference between the deposition rate on the substrates and on the sensing crystal.

3.2 CRYSTAL OSCILLATOR CIRCUIT

The monitor crystal is excited by an oscillator circuit based on the single chip clock generator/driver IC 8284A. The chip contains a crystal controlled oscillator. Oscillator circuit of the 8284 is designed primarily for use with an external series resonant fundamental mode crystal. Schematic diagram of the oscillator circuit is shown in Fig. 3.1. The 6 MHz monitor crystal is connected across pins X_1 and X_2 of the oscillator, to generate a stable clock source. Buffered output of the oscillator is available at the pin 12 (OSC.) of the 8284A. The monitor oscillator is protected against voltage surges by connecting diodes D1 to D4 across the crystal. Power for the oscillator is derived form a regulated 5V dc supply with load regulation of 0.01%. The monitor crystal is placed in close proximity to the substrate inside the deposition chamber to receive the evaporant. The crystal is a flat circular plate approximately 1.40 cm in diameter and 0.028 cm thick, AT cut with an angle of 35^0 10'. Cross section of the crystal vibrating in thickness shear mode is shown in Fig. 3.2. AT-cut crystals have better temperature co-efficient and the cumulative effect of heating by radiation from an evaporation source is generally small compared to the frequency shift due to the mass deposited in the same time. Distance between monitor crystal and the evaporation source is 25 cm. The crystal is water cooled and temperature is not allowed to exceed 50 ^{0}C. Drift in the oscillator circuit is tested with a digital counter. Stability of the oscillator is an important parameter as it decides the accuracy of measurement of film thickness measurement. To study the stability, oscillator is kept ON continuously for 24 hours in an environment where the temperature is maintained in the range 20 to 25 ^{0}C. It is connected to a digital counter with a 100 cm long shielded cable. Frequency is measured every hour. It has been found from this experiment that there is only 1.7 Hz variation in 24 hours duration for the 6 MHz monitor crystal oscillator.

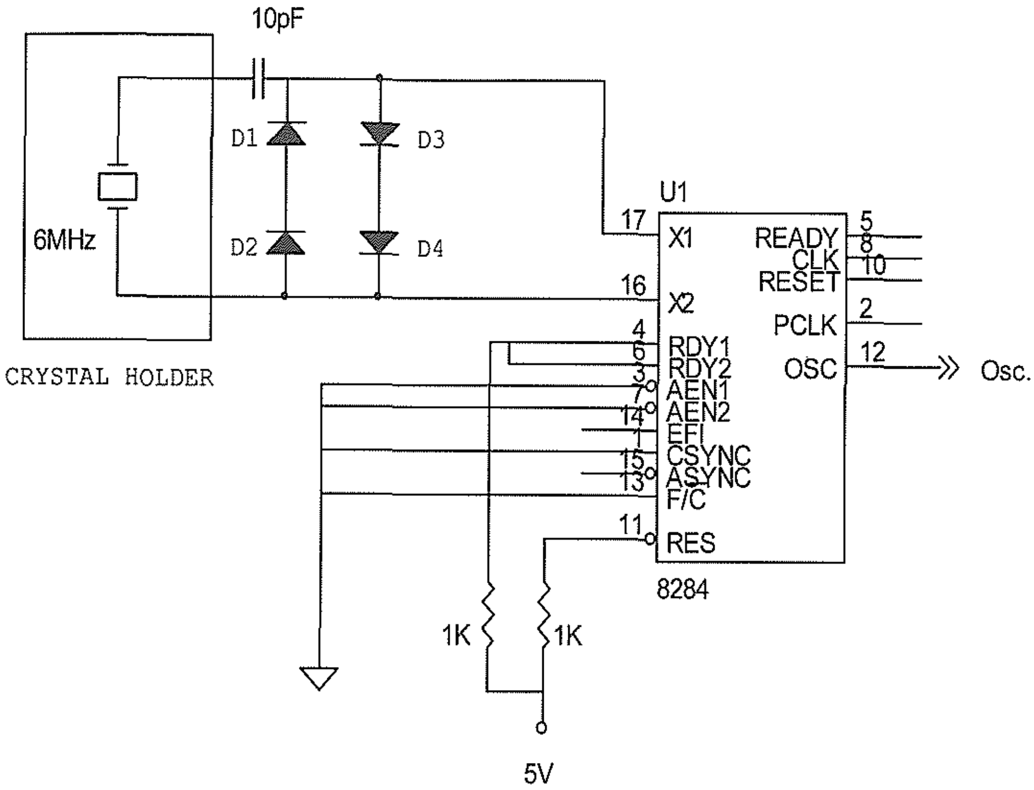


Fig. 3.1 Oscillator circuit

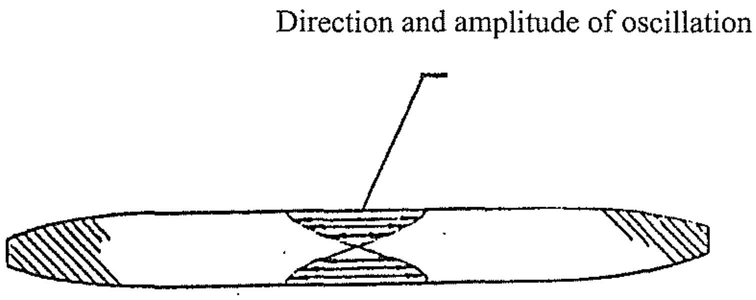


Fig. 3.2 Cross-section of crystal vibrating in thickness shear mode

3.3 INSTRUMENTATION FOR FILM THICKNESS MEASUREMENT

The instrumentation developed for the thin film thickness measurement consists of two main parts, namely, a water cooled crystal holder and electronics for measurement of the frequency deviation in the monitor oscillator. Water cooled crystal holder is required to minimize the effect of temperature on the frequency of oscillation. Electronics for measurement of the frequency deviation due to deposition of evaporated target material on the crystal surface is based on heterodyning principle. These are described in the following sub sections.

3.3.1 CRYSTAL HOLDER

The crystal holder is shown in Fig. 3.3. It consists of a water cooled shield soldered to a copper block with suitable cuts for the crystal. A teflon insulated cable connects the sensor head to the electrical feedthrough. The feed through is installed in the vacuum chamber through base plate hole. The co-axial cable from monitor crystal is connected to the feed through's external BNC connector. Water line connections to the feedthrough are accomplished by brazing the copper tubes to the steel base plate.

Sufficient cooling is obtained by water flow of approximately 0.5 litre per minute. Temperature of the circulating water is kept between 20 to 25 ^{0}C. Excessively cold water may result in condensation of water on the crystal, when the system is vented. Excessive moisture may cause the crystal to cease oscillation.

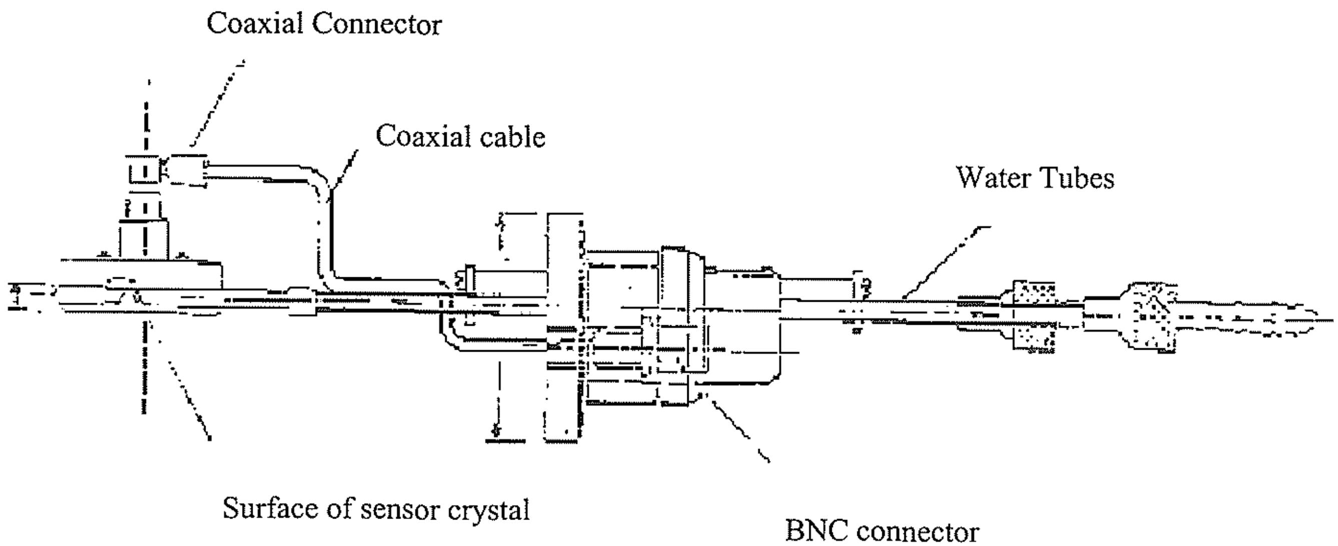


Fig. 3.3 Crystal Holder

3.3.2 FREQUENCY DEVIATION MEASUREMENT

Frequency deviation in monitor crystal due to mass loading in accordance with Equation (11) is measured by heterodyning principle. For this purpose a reference crystal oscillator placed outside the deposition chamber is used. The reference oscillator is a 6 MHz commercial oscillator having an operating temperature stability of 1x 10^{-7}. The instantaneous monitor frequency f_m-Δf, where f_m is the fundamental frequency of the monitor crystal and Δf is the frequency change due to mass loading, is mixed with the stable reference oscillator frequency f_r. The integrated circuit balanced modulator LM1496 is used for this purpose. Salient features of LM1496 are,

- Fully balanced output
- Bandwidth 80 MHz
- Input offset current 5.0 μA
- Output offset current 30 μA
- Differential output swing 8.0 V_{p-p}

The modulator output contains the sum and difference of the two input frequencies. The higher frequency component f_r+ (f_m-Δf) from the balanced modulator is filtered out by an opamp (741) configured as an unity gain buffer. Circuit diagram of the balanced modulator is shown in Fig. 3.4. The carrier signal is applied to pins 8 and 10 in common mode to a set of cross-coupled differential amplifier. The bias voltage applied to pin 5 determines the amount of current through the amplifiers. The resistor connected to pins 2 and 3 sets the modulator gain with a smaller resistor resulting in higher gain. The DC voltage difference between pins 1 and 4 will balance the differential amplifiers for complete carrier rejection by equalizing the current in each differential amplifier. When the signal is applied to pins 1 & 4, current through the amplifier alternately increases (or decreases), to output the sum and difference frequencies. The output is taken from pin 6. This circuit offers a high degree of carrier suppression (–60 dB).

Voltage signal obtained from the block consisting of mixer and the unity gain buffer contains the information about film thickness in terms of varying frequency. This signal is shaped by a zero crossing detector comprising of the comparator LM311. The TTL compatible output from the comparator is fed to 8253 timer counter on the PC add-

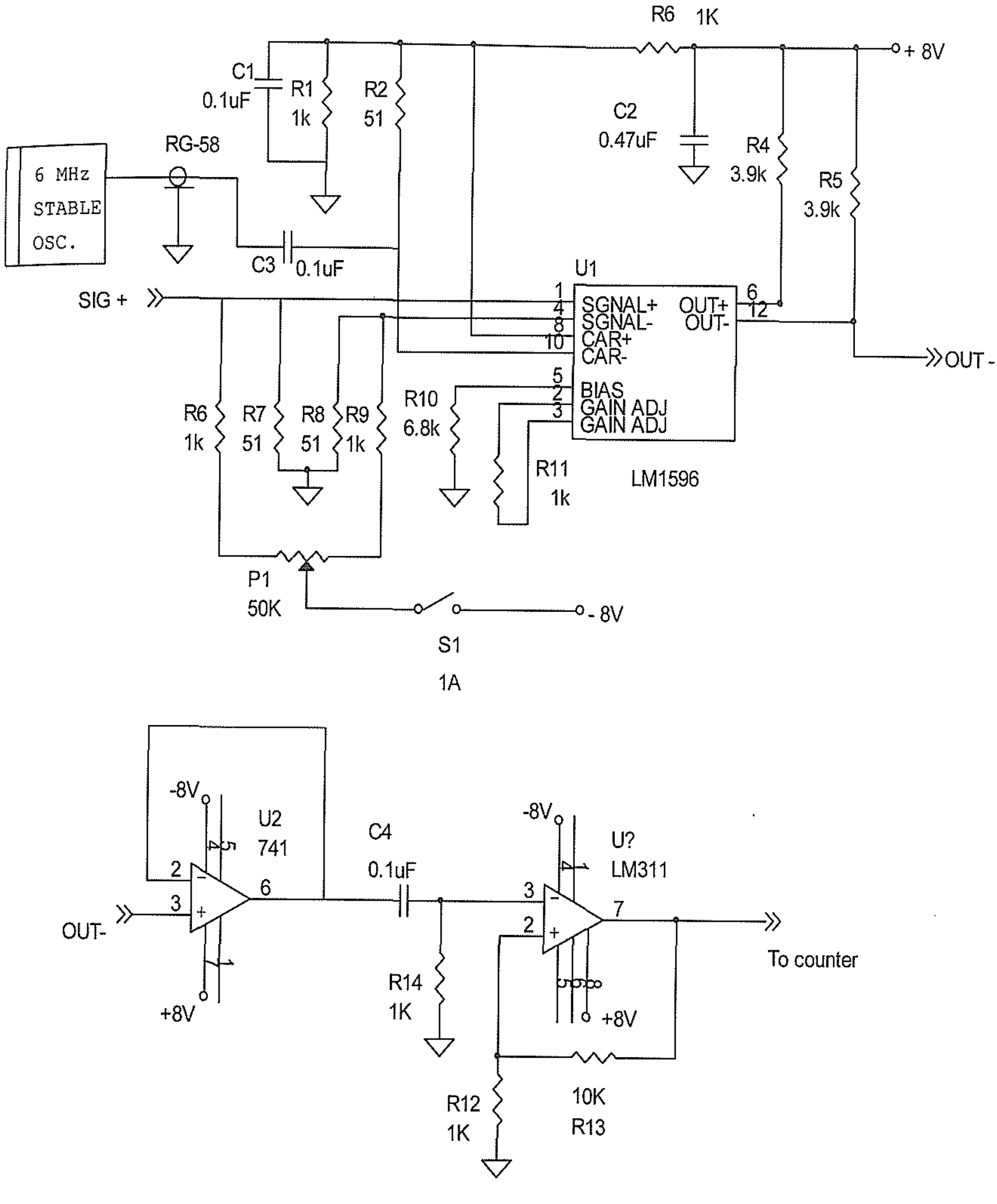


Fig. 3.4 Balanced modulator circuit

on card. This is configured as a 16 bit counter. A program written in ANSI C reads the counter value to compute the frequency.

3.4 TEST RESULTS

The thickness measurement unit was tested for deposition of ZnO film on Al_2O_3 substrate. The deposition chamber vacuum and substrate temperature were maintained at 10^{-8} torr and 500 ^{0}C respectively. Laser was operated at 10 shots per second. The laser pulse energy was set at 200 mJ. Films of different thickness were deposited by controlling the number of laser shots i.e. by setting the time duration for which the laser was fired. After each deposition, the corresponding change in oscillator frequency, Δf, corresponding to the deposited thickness, l_f, was monitored. The film thickness, l_f, was calculated by Z-match technique, as given by equation (11). Other parameters used for calculation of the film thickness are,

Fundamental frequency of oscillation of the monitor oscillator, f_Q=6x10^6 Hz

Density of the quartz crystal, ρ_Q=2.648 gm/cm^3

Acoustic impedance of the quartz, Z_Q= 8.801x10^6 kg/m^{2}sec

Density of the film material, ρ_F=5.61x10^3 kg/m^3

Acoustic Impedance of the film, Z_F=15.7x10^6 kg/m^2 sec

In the first experiment three deposition runs were carried out on independent sets of target, substrate and monitor crystal. In each run 5000 laser shots were fired and deviation in the frequency of monitor oscillator Δf, was used to calculate the deposited film thickness. It was observed that frequency of oscillation of the monitor crystal did not stabilize to a final value immediately after the required number of shots had been fired. It was noted that during the deposition period the frequency falls steeply. However after stopping the laser shots and hence the deposition process, there exists a transient period of about ten minutes during which the frequency continues to decrease. The transient period is caused by the heat form the laser generated plasma plume in front of the target, or due to the bombardment by plasma ions from the plume [14]. In all the three depositions performed under identical conditions, the final deviation in the frequency, Δf, is nearly equal. This is shown in Fig. 3.5. The calculated values of film thickness were compared by ex-situ measurements with a surface profilometer at TIFR, Mumbai. The result is shown in Table 3.1. It is to be noted that for the same number of laser pulses there is a variation in the measurements of film thickness by the surface profilometer, for the three sets.

Table 3.1 Results for experiment I. Thickness measured by Δf and surface profilometer for three different samples with same number of laser pulses (5000 shots)

Run No.	Δf (in Hz)	Film thickness calculated from Δf (in A^0) (l_f)	Thickness obtained by surface profilometer (in A^0) (l_s)	Percentage difference $100(l_s-l_f)/l_s$
1.	5400	1188	1300	9
2.	5480	1205.6	1345	10
3.	5430	1194.6	1315	9

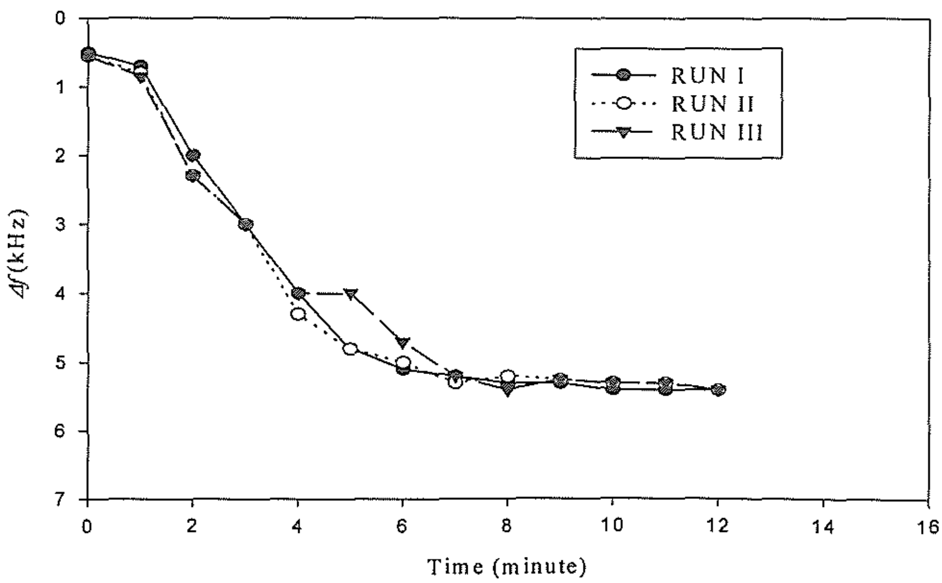


Fig 3.5 Frequency variation vs. time for film deposition in experiment I

In the second experiment, the deposition runs were carried out for three different time durations. For each run the deviation in monitor oscillator frequency, Δf, during the post-deposition period were observed. In the three runs the laser shots were fired for 6, 10 and 16 minutes respectively. Results for this experiment are shown in Table 3.2.

Table 3.2. Readings for experiment II. Thickness measured using Δf for different numbers of laser shots

Deposition time (in minute)	No. of laser pulses	Stabilized Δf (in Hz)	Calculated film thickness (in A^0) (l_f)
6	3600	4235	923
10	6000	7025	1533
16	9600	11100	2423

It is noted from these observations that the steady state change in the frequency of oscillation of the monitor oscillator is linear with respect to the number of laser shots. Variation in, Δf, with respect to time during the post-deposition period is plotted in Fig. 3.6.

In the third experiment, five deposition runs were carried out on different sets of target, substrate and monitor crystal. For each run the number of laser shots fired, was varied. Thickness were also measured ex-situ by surface profilometer. Calculated values of the film thickness, l_f, were compared with reading of the surface profilometer, l_s . The result is shown in Table 3.3. Film thickness as calculated from the monitor oscillator frequency deviation and the corresponding readings of the surface profilometer are plotted against the number of laser shots for each run and shown in Fig. 3.7.

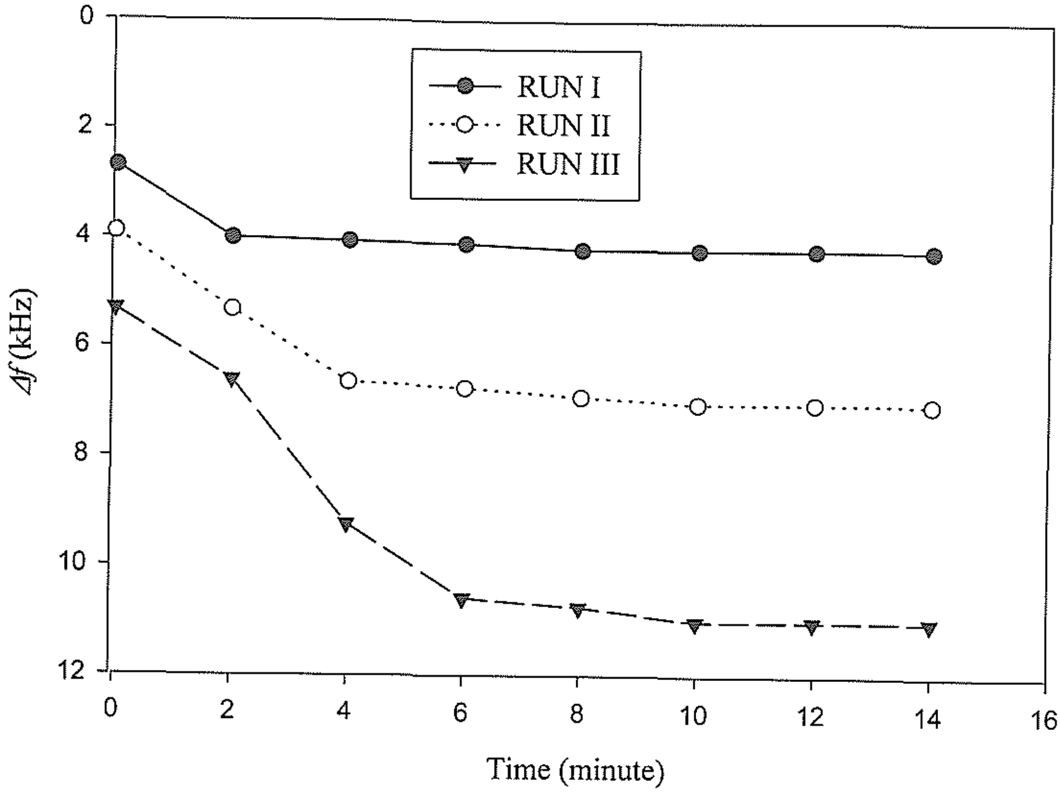


Fig. 3.6 Frequency variation vs. time after firing of the laser shots for film deposition in experiment II

Table 3.3 Readings for experiment III

Run No.	Total number of laser shots	Δf (in Hz)	Film thickness calculated from Δf (in A^0) (l_f)	Thickness obtained by surface profilometer (in A^0) (l_s)
1.	1960	2115	461	1000
2.	3000	3260	711	900
3.	4800	5220	1138	1200
4.	6000	6525	1423	1520
5.	9000	9770	2132.3	2250

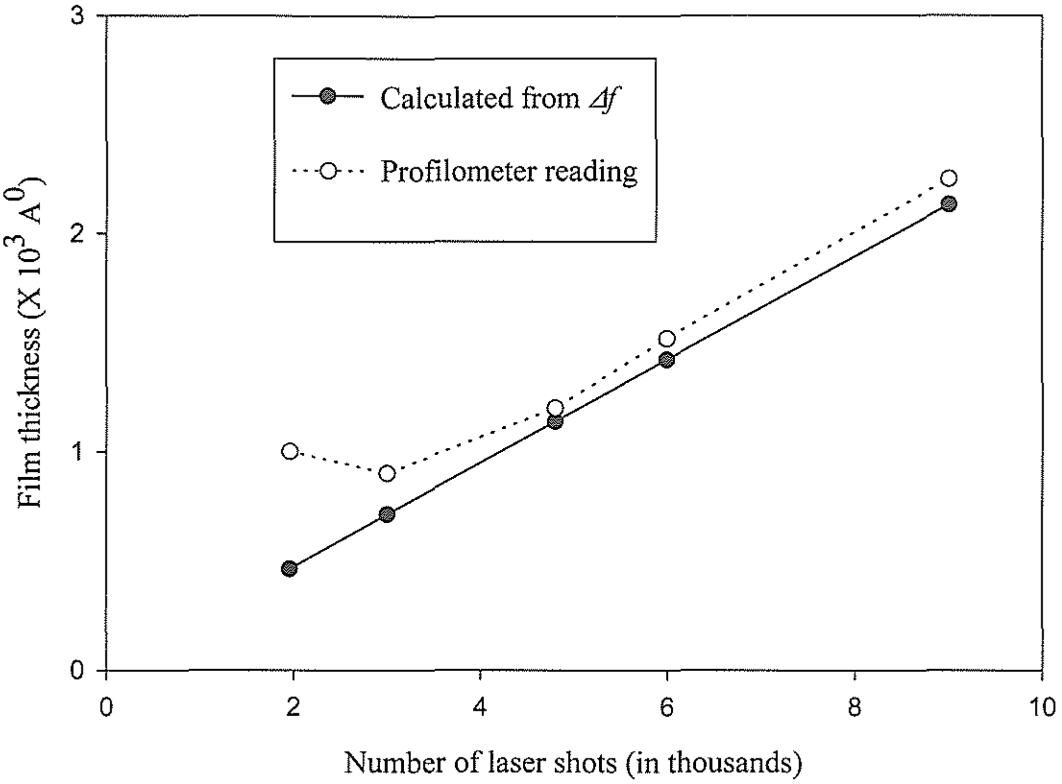


Fig. 3.7 Film thickness calculated from Δf and readings of surface profilometer vs. number of laser shots

In the fourth experiment, ten different deposition runs were carried out with same set of target, substrate and monitor crystal. Values of film thickness as calculated form f were compared with the linearly interpolated values of the profilometer readings. These are shown in Table 3.4. The values of film thickness as calculated from variation in monitor oscillator frequency, Δf, and estimated from the linearly interpolated values of the profilometer readings are plotted with respect to the number of laser shots in Fig. 3.8.

Table 3.4 Readings for experiment IV

Run No.	Total number of laser shots	Δf (in Hz)	Film thickness calculated from Δf (in A^0) (l_f)	Thickness obtained by linearly interpolated value of the profilometer reading (in A^0) (l_s)
1.	2000	2120	462	490
2.	3000	3270	713	750
3.	4800	5215	1137.3	1200
4.	6000	6535	1425	1500
5.	7200	7855	1713.8	1800
6.	9000	9740	2125.7	2250
7.	12000	13000	2839	3000
8.	2000	2115	461	490
9.	3000	3265	711.8	750
10.	6000	6545	1427.6	1500

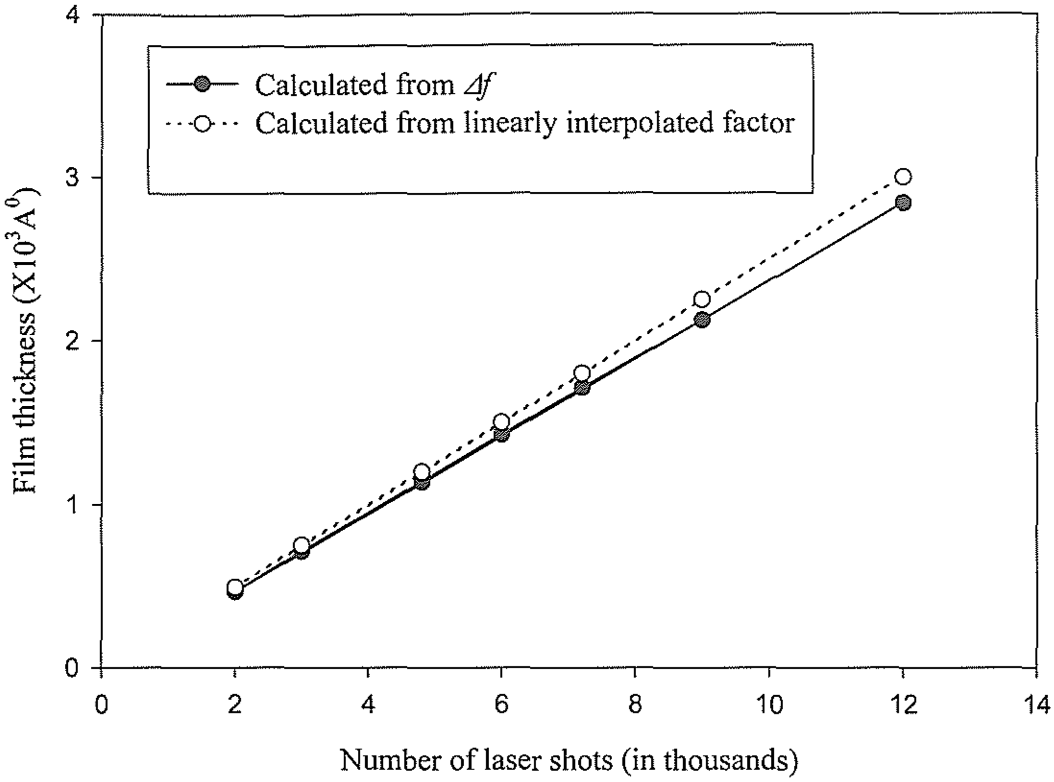


Fig 3.8 Film thickness as calculated from Δf and from the linearly interpolated factor vs. no. of laser shots

From the readings obtained by the above mentioned experiments it is observed that the values calculated from Δf measurements are repeatable and for thickness greater than 700 A^0 the calculated values are linear with respect to the number of laser shots, and varies between 5 to 10 percent in comparison with the corresponding surface profilometer readings.

Chapter 4

PROCESS VARIABLE MEASUREMENTS AND CONTROLS

4.1 SYSTEM DESCRIPTION

Schematic of the data acquisition and control unit for the pulsed laser deposition set up is shown in Fig. 4.1. Laser beam from the optical head of the laser system is incident on a beam splitter marked BS. This reflects part of the laser beam for energy measurement. S1 is the shutter, which blocks the initial laser pulses. Only when the laser energy stabilizes to a threshold value of 200 mJ, stepper motor M1 opens this shutter and the laser is incident on the target T. Phase sequence for the stepper motor is generated by the digital output stage of a PC add-on card. The add-on card consists of a timer, ADC, DAC and buffered digital I/O stage. Another stepper motor M2 continuously rotates the target, to avoid crater formation. The quartz crystal based sensor accomplishes the task of thickness monitoring of the deposited film. The monitor crystal is placed near substrate, which is marked S in the diagram. Chamber vacuum is measured by a hot cathode ion gauge. A thermocouple marked TC in the diagram measures the substrate temperature which, in turn is placed in the feed back loop of a programmable temperature controller.

4.2 MONITORING OF PROCESS VARIABLES

A hot cathode ion gauge measures the chamber vacuum. Sensitivity of the gauge used in the system is 10 mA per torr. Thus the operating Vacuum of 10^{-8} torr yields a current of 0.1 μA. A current to voltage converter as shown in Fig. 4.2, is used to convert this current to an equivalent voltage. The current to voltage converter uses a low noise and low bias current operational amplifier LH0052. The 100K feed back resistor is shunted with a 0.1 μF capacitor to reduce the high frequency noise. The current to voltage converter generates 10 mV form 0.1 μA gauge current. A 12 bit ADC, AD574, on the PC add-on card digitizes this voltage. Reference voltage for the card is 5V. This gives ADC resolution of 1.2 mV, which in terms of the vacuum is equal to 10^{-9} torr.

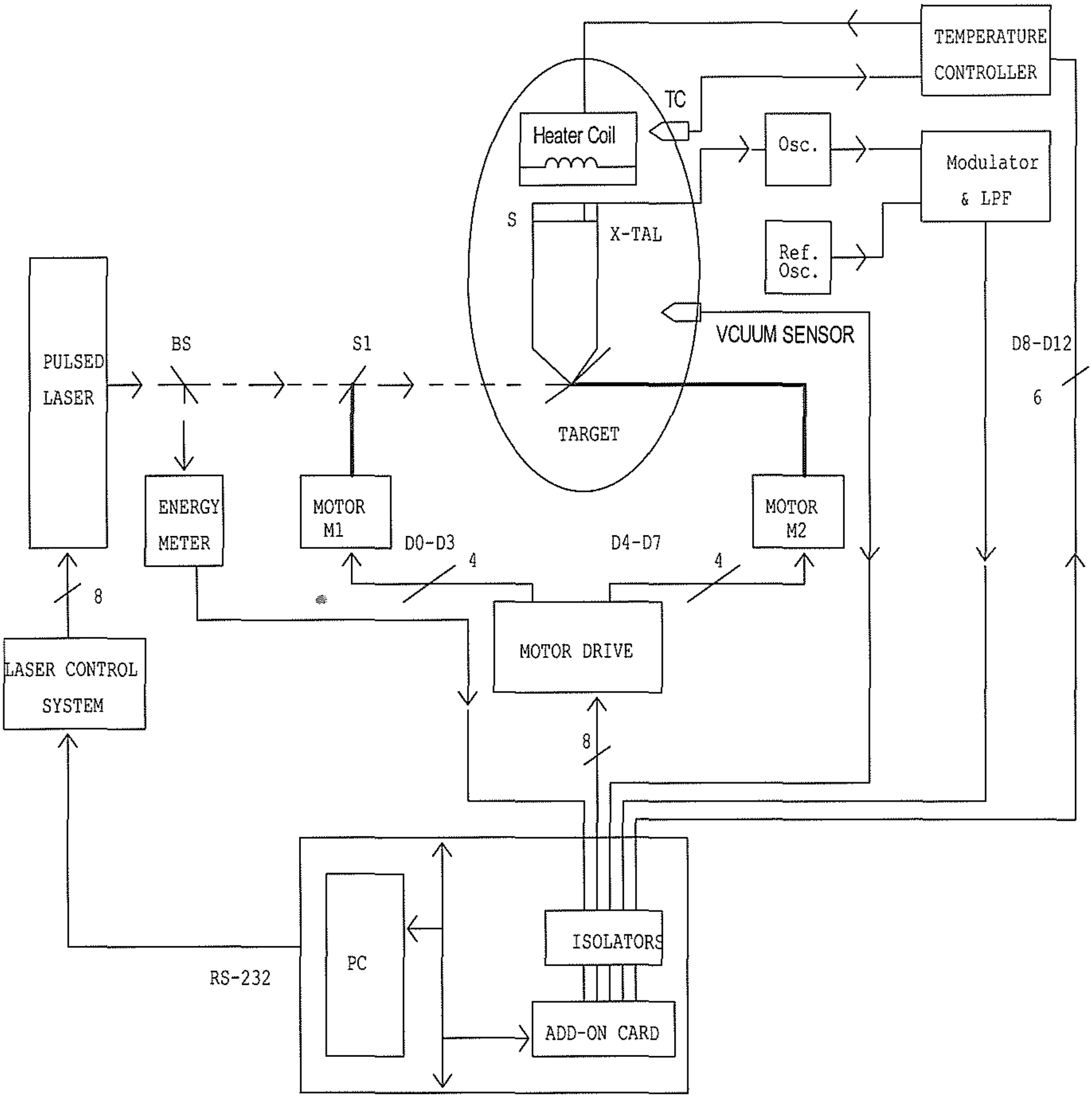


Fig. 4.1 Data acquisition and control unit

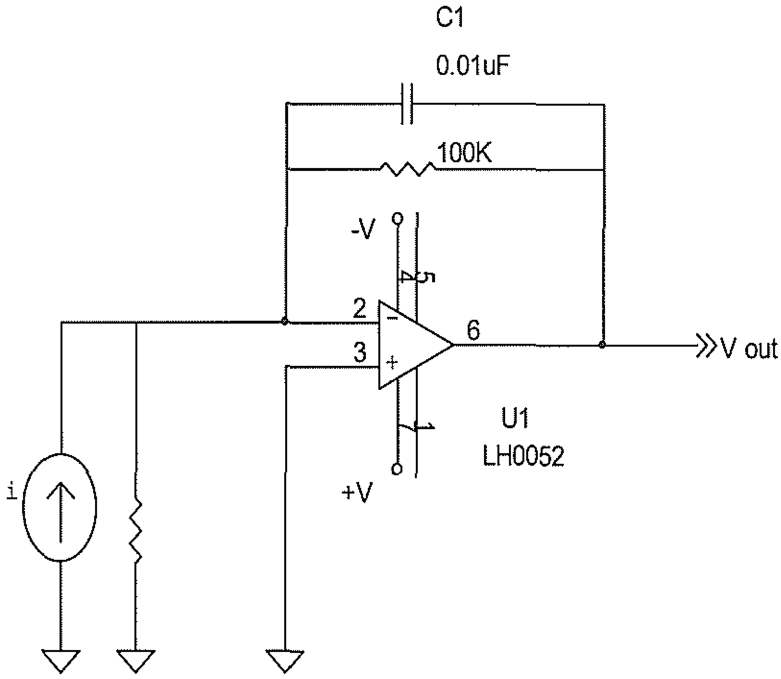


Fig. 4.2 Current to voltage converter

An Eurotherm make temperature controller in combination with a K-type thermocouple controls the heater. The temperature is ramped to 500 ^{0}C with a rate of 30 ^{0}C per minute. This is achieved by programming the temperature controller through its digital inputs. Six digital lines D8 to D13, from the PC add-on card is used for this purpose. These buffered lines are isolated by opto-couplers 6N138.

The pyroelectric detector used to measure the laser energy has a sensitivity of 5V per Jule. The optimum laser energy for PLD process is 200 mJ which, corresponds to a signal strength of 1V. This is interfaced to the ADC on the add-on card through an isolation amplifier ISO 100.

4.3 CONTROL OF MOTOR DRIVES

The system requires two motors to rotate the target material and to operate the shutter placed in laser path during the deposition process. The motors and the loads i.e. shutter and the target material are marked M1, M2, S1 and T respectively in Fig. 4.1. The target material has to be continuously rotated at 10 rpm to avoid evaporation from a single point. Shutter S1 placed in the laser path is used to block the laser radiation till laser energy stabilizes to 200mJ, i.e. the laser pulses before the laser system stabilizes are to be blocked.

For the operations described above, stepper motors of torque 800 gm-cm is used. These are four phase motors with bifialer windings. Voltage requirement is 12V. And the required current per phase is 0.15 Amps. Fig. 4.3 shows the motor winding scheme. The phase sequence is given in Table 4.1.

Table 4.1 Phase sequence for stepper motor drive

	Step	Q_1	Q_2	Q_3	Q_4
CW Rotation	1	ON	OFF	ON	OFF
	2	ON	OFF	OFF	ON
	3	OFF	ON	OFF	ON
	4	OFF	ON	ON	OFF

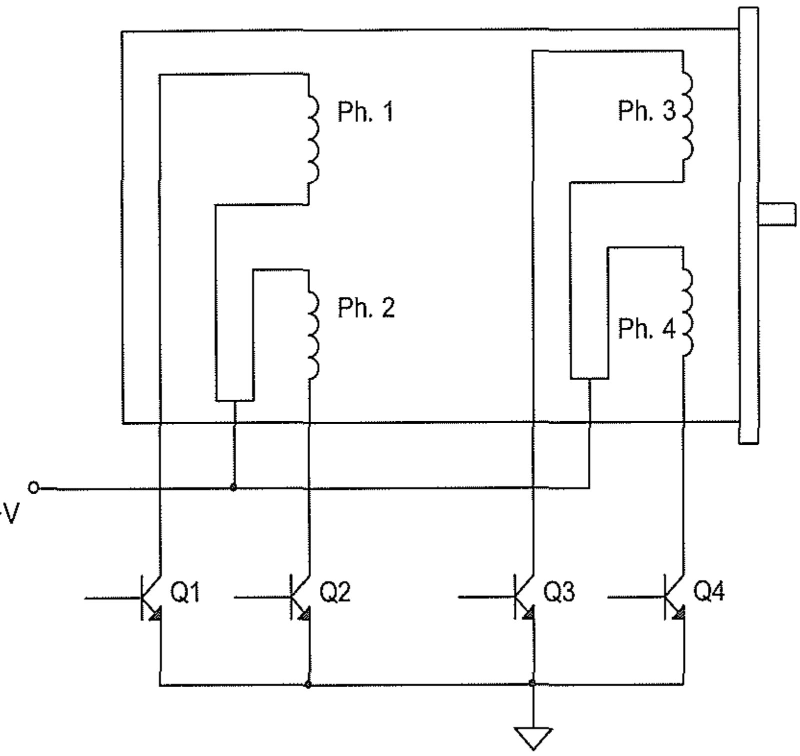


Fig. 4.3 Stepper motor winding diagram

The motor is driven from one step position to the next by switching a dc supply from one set of stator winding to another. Phase sequence for the motors M1 and M2 are generated from the digital outputs D0 to D3 and D4 to D7 respectively of the PC add-on card. These digital outputs are optically isolated by Opto-isolator 6N138. A monolithic driver IC L298 drives the motor coils. Circuit diagram of the motor winding along with driver IC is shown Fig. 4.4. Each L298 drives four phases of a stepper motor. Diodes 1N4007 are used for protection of the output stage of L298 during switching off the respective motor winding.

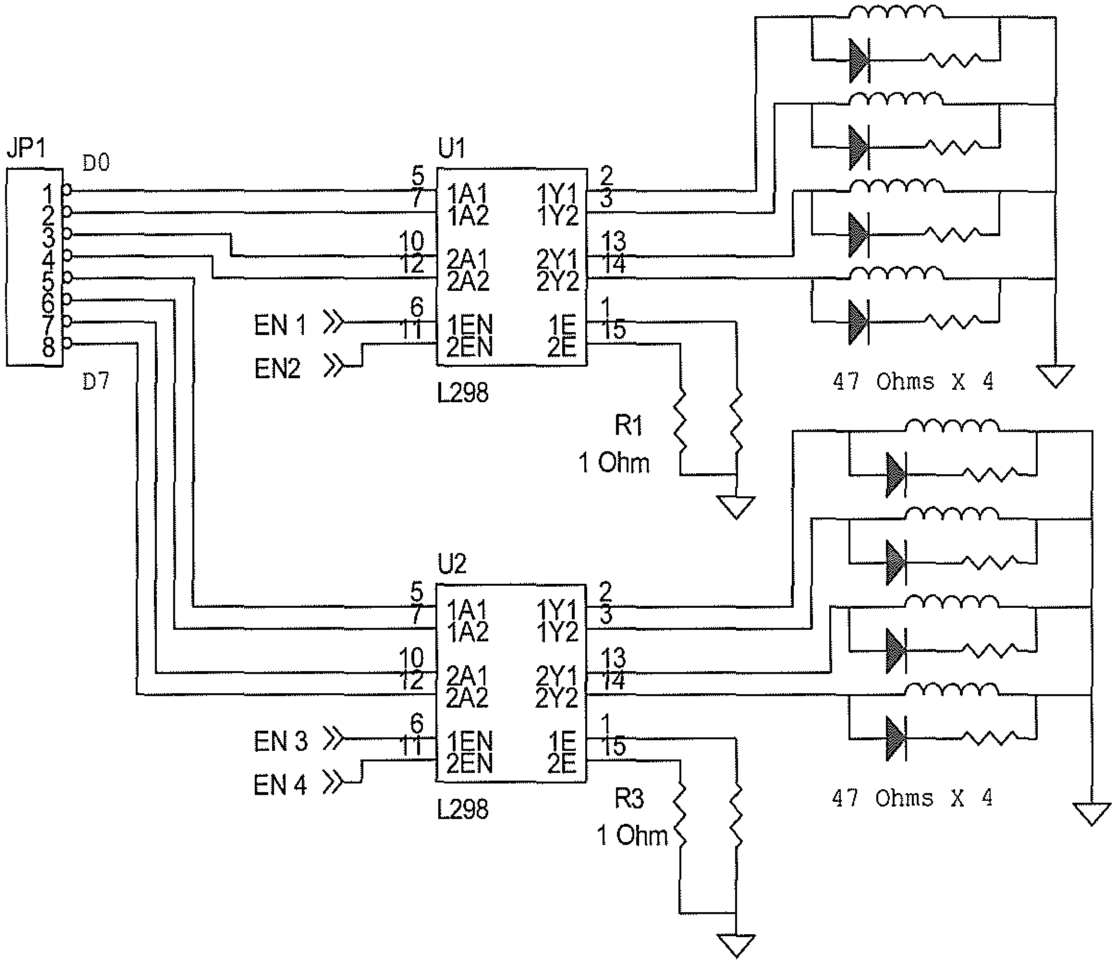


Fig. 4.4 Stepper motor driver circuit

4.4 CONTROL PROGRAM

Flow chart for the control program is shown in Fig. 4.5. Input parameters to the control program are density of the film material ρ_F, its acoustic impedance Z_F and the desired film thickness l_f. On start up, the system reads vacuum sensor through one of the analog inputs of the PC add-on card. On achieving the required vacuum level of 10^{-8} torr, the Eurotherm temperature controller is programmed by digital output D8 to D13 of the PC add-on card to ramp the substrate temperature to 500 ^{0}C at the rate of 30 ^{0}C per minute. Under the above mentioned conditions of chamber vacuum and the substrate temperature, the laser is started by sending appropriate command through the PC serial port. However, the shutter S1 does not allow it to be incident on the target material unless the laser energy reaches 200mJ. Laser energy is being read by a pyroelectric detector and is interfaced to the PC through one of the analog inputs of the add-on card. When the laser energy is stabilized to the required value, the stepper motor M1 opens the shutter S1. Also the target is continuously rotated at 10 rpm by activating the stepper motor M2. Phase sequence for these motors are generated by programming the digital output of the add-on card. The digital out signals D0 to D7 is used for this purpose. Plumes start forming after the laser radiation is allowed to be incident on the target. These are subsequently deposited on the substrate as well as on the quartz crystal. Because of the mass loading effect, frequency of the crystal oscillator begins to decrease. This is heterodyned with a reference oscillator and the frequency difference, Δf, which is proportional to the deposited film thickness, is measured by an 8253 based counter in the PC add-on card. It takes some time, for the monitor frequency to stabilize after the deposition process. Therefore, an appropriate cooling time will have to be given before calculation of the film thickness from Δf is carried out. The laser pulses are stopped from being incident on the target by activating the laser blocking shutter, after firing an estimated number of shots. The monitor frequency is allowed to stabilize and film thickness is calculated, based on the stabilized value of Δf, using Equation (11). If the calculated value of film thickness is equal to the entered value, the process is terminated by turning off the laser unit and the substrate heater. If the calculated film thickness is not equal to the required value, the laser blocking shutter is opened and again an estimated number of laser shots, based on the difference of calculated value and the required value are fired.

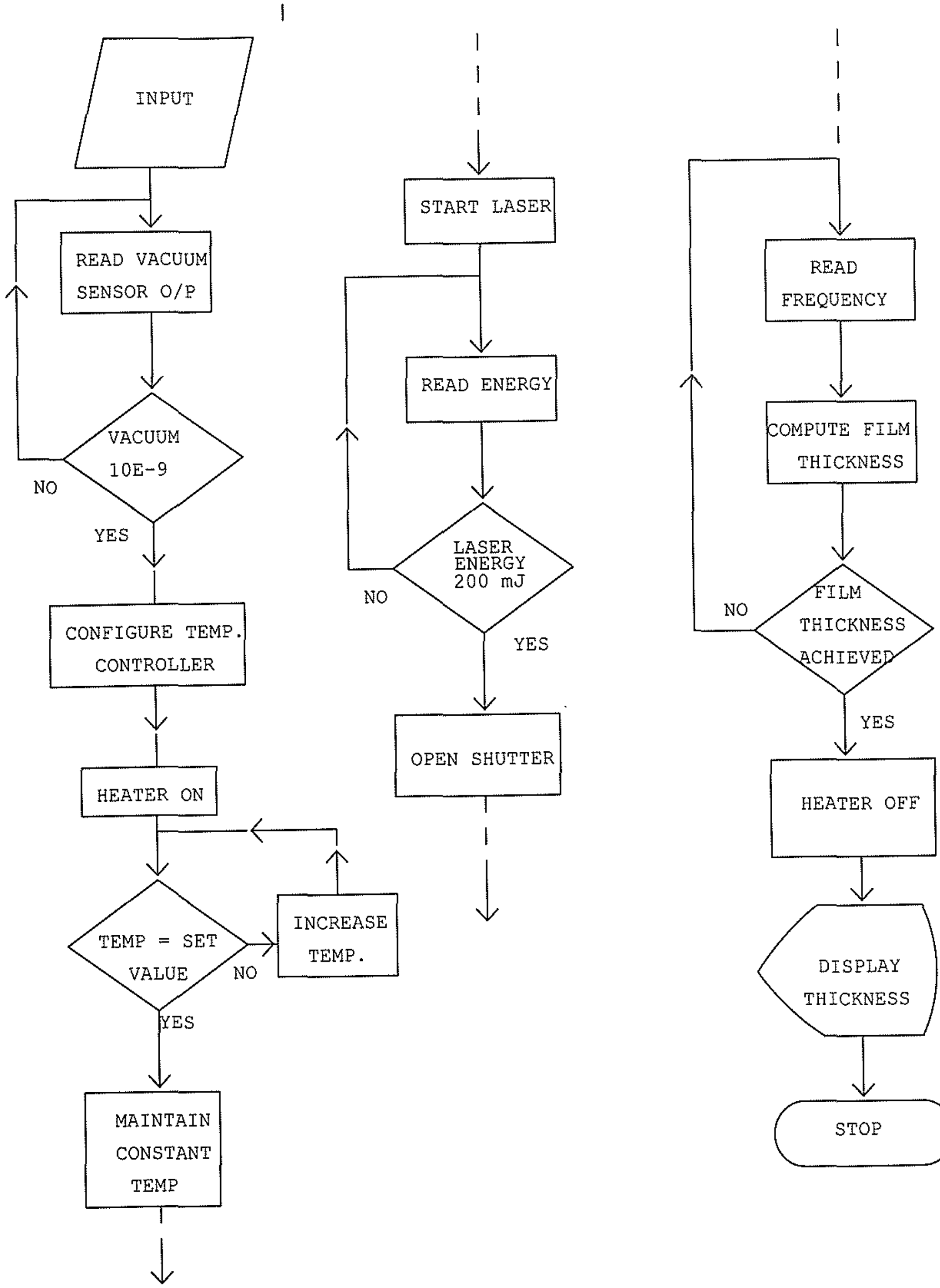


Fig. 4.5 Flow chart of the control program

Chapter 5

CONCLUSION AND SCOPE FOR IMPROVEMENTS

The objectives of the project namely, development of a PC based data acquisition and control system for various process variables in a pulsed laser deposition scheme has been completed. The design and development included (a) a stable crystal oscillator and circuit for detection of frequency deviation. (b) pre-amplifiers and other necessary electronics for measurements of vacuum and laser energy. (c) programming and use of a PC add-on card (developed earlier) for interfacing various sensors, drives and control circuit for stepper motors (d) interfacing of PC with the laser control system. Overall specifications of the system are,

1. Resolution of film thickness measurement : 10 A^0
2. Crystal frequency : 6 MHz
3. Controlled parameter : Rate of deposition and film thickness (Selectable)
4. Resolution of energy measurement : 0.2 mJ
5. Resolution of vacuum measurement : 10^{-9} torr
6. Speed of target rotation : 10 rpm

Further improvement in the system may be carried out to reduce the post deposition transient period, during which the monitor frequency continues to decrease. At present cooling time has to be provided to observe a stable frequency change due to mass loading. Using a vacuum compatible, thermally conducting paste between the monitor crystal and the heat conducting surface in the crystal holder may provide better coupling for heat transfer and may reduce the duration of the transient period.

Many of the parameters of films grown by pulsed laser deposition technique are still not well understood. These are highly non-linear with respect to the laser energy, substrate temperature and vacuum level. The data acquisition unit described in this thesis may be used to characterize and model the film growth under various conditions.

APPENDIX A
PC ADD-ON CARD

A.1 SPECIFICATIONS AND SALIENT FEATURES OF THE PC ADD-ON CARD

The PC add-on card has following general specifications and features.

- Two single-ended analog input channels
- A 16 bit counter channel based on Intel 8253 programmable Timer/Counter
- A 12 bit successive approximation analog to digital converter AD574.
- 16 digital output channels

The analog inputs are used to digitize signals from vacuum sensor and laser energy meter. These analog inputs are isolated by isolation amplifier ISO100 and selected by multiplexer PMI Mux-08. The MUX-08 from Precision Monolithics Inc. is an eight-channel analog multiplexer, which connects a single output to one of the eight analog inputs depending upon the state of a 3-bit binary address.

The A/D data are stored in two registers located at addresses BASE+4 and BASE+5. The lower byte AD0 to AD7 is stored in BASE+4 and the higher byte data AD8 to AD11 is stored in BASE+5.

The digital outputs are isolated by Opto-coupler 6N138. Six of the digital outputs are used for programming the Eurotherm temperature controller. Eight digital outputs are used for driving the stepper motor.

The 16 bit programmable interval timer/counter (Intel's 8253) is used for measurement of frequency deviation.

A.2 ANALOG TO DIGITAL CONVERTER

This is a 12-bit successive approximation type AD574. Conversion time is 35 μ Sec.

A.3 DIGITAL TO ANALOG CONVERTER

The DAC used is Burr Brown' s 7541. Resolution of the digital to analog converter is 12-bits.

A.4 ISOLATION AMPLIFIER ISO 100

ISO 100 is used to interface analog signals from vacuum sensor and laser energy meter to the PC-add on card. It is an optically coupled amplifier. Main specifications of ISO 100 are,

Isolation voltage : 750 V

Input offset : 500 μV

Input offset current : 10 nA

A. 5 OPTO-COUPLER 6N138

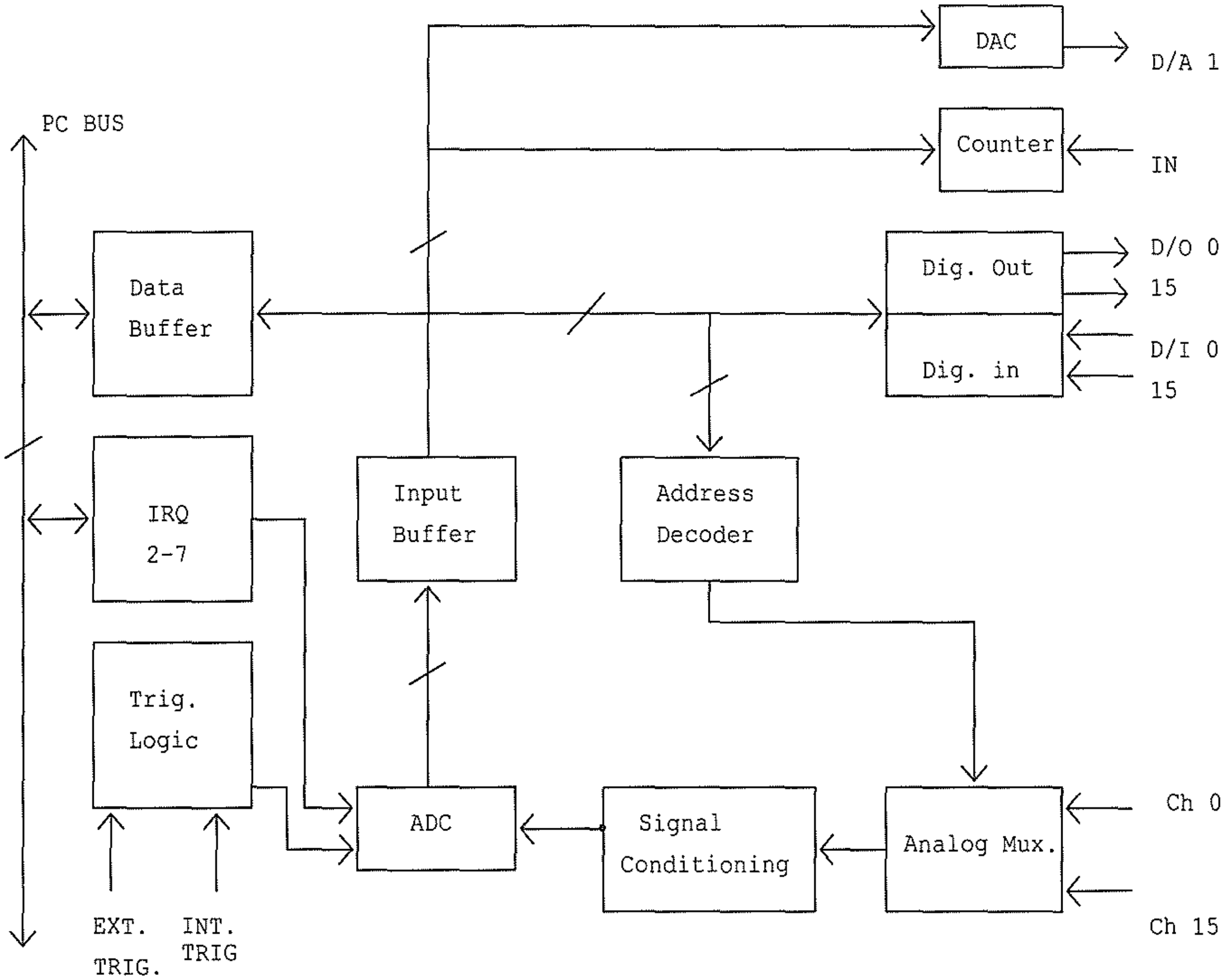
Digital out signals (D_0 to D_{15}) used to program the temperature controller and to drive the stepper motors are isolated from the stepper motor with the help of Opto-coupler 6N138. These isolators have high current transfer ratio, typically 800% and gives TTL compatible outputs.

A.6 ANALOG MULTIPLEXER

Analog multiplexer is used to select one of the eight analog channels for processing. MUX-08 from PMI is used for this purpose.

A.7 COUNTER 8253

The PC add-on card has an 8253 counter. It is used to measure the frequency deviation in monitor oscillator. Its frequency response is 2 MHz.



PC ADD ON CARD

APPENDIX B
DATA SHEETS

B.1 OPERATIONAL AMPLIFIER LH0052

This is used in current to voltage converter circuit for the vacuum sensor. LH0052 is a low bias current (10pA) precision FET input operational amplifier. Its other features are

Low input offset current: 500 femto amps.

Low input offset drift : 5μV/C°

Low input offset voltage : 100 micro Volts

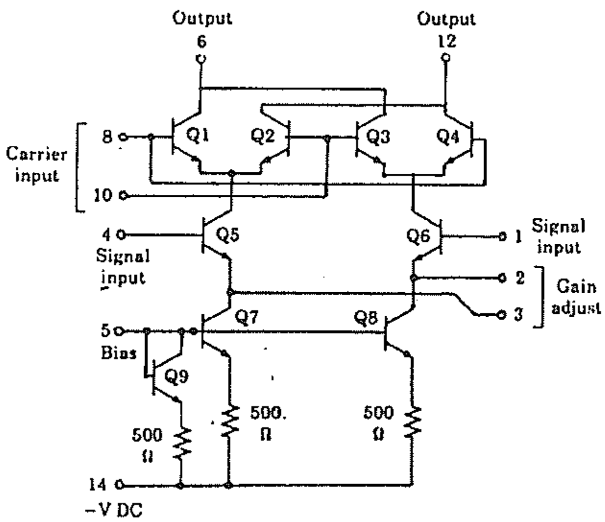
High open loop gain: 100 dB

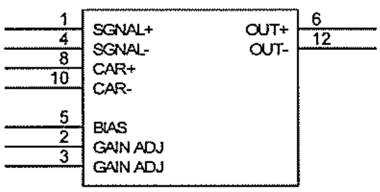
B.2 CLOCK GENERATOR 8284

The 8284 is a single chip clock generator driver. The oscillator circuit of the 8284 is designed primarily for use with an external series resonant, fundamental mode crystal. Working with a single +5V supply, it can generate 10 MHz signal.

B.3 BALANCED MODULATOR LM 1496

LM1496 is a balanced modulator, which is used to produce two side bands produces an output voltage proportional to the product of an input voltage and a switching (carrier) signal. It has frequency response up to 100MHz. Schematic diagram of LM1496.is shown below,



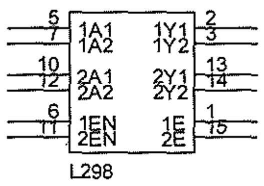


LM1496

Q1 with Q4 and Q2 with Q3 form a set of cross-coupled differential amplifiers. Transistors Q7 and Q8 serve as constant current generator for the differential amplifiers. Bias voltage applied to pin 5 determines the amount of current through the amplifiers. With application of the signal to pins 1 and 4, transistors Q5 and Q6 will alternately increase (or decrease) the current through their associated amplifiers to output the modulated signal.

B.4 STEPPER MOTOR DRIVER IC L298

L298 from Sprauge is a dual full bridge driver designed to accept standard TTL logic levels and drive inductive loads. It can provide total DC current upto 4A. Two inhibit inputs are provided to disable the device independently of the input signal.



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