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SYNTHESIS AND CHARACTERIZATION OF LPCVD SILICON CARBIDE THIN FILMS FOR X-RAY LITHOGRAPHY

by

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the Faculty of Graduate School of Electrical Engineering

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at

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Approval Sheet

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ABSTRACT

Title: Synthesis and characterization of LPCVD Silicon Carbide thin films for X-ray lithography

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Amorphous silicon carbide thin films were fabricated using low pressure chemical deposition method with a single liquid precursor, di tertiary butyl silane. The films were deposited for a temperature range of $500-850°C$ and at different pressures ranging from 0.05 to 1 torr. The growth rate of the films deposited at constant pressure of 0.2 torr with a flow rate of 60 sccm, was found to follow an *Arrehenius Behavior* in the temperature range of $600-675°C$, yielding an activation energy of 32.5 $k\,cal\,mol^{-1}$. IR spectroscopic study showed an absorption peak centered at 780 cm^{-1}, indicating the presence of Si-C vibrational mode. X-ray diffraction studies confirmed the amorphous nature of the film. Deposition temperature was found to play an important role in the stoichiometry of the film. The carbon atomic composition of the film was increased as the deposition temperature was increased, so that, at $850°C$ the carbon composition was 76%. Addition of acetylene was found to be an alternative method for varying the composition of the film and addition of ammonia resulted in Si-C-N type film. The films were found to be tensile in all cases.

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Chapter 1

INTRODUCTION

1.1 Silicon Carbide as X-Ray Mask Membrane Material

The purpose of synthesis of Silicon Carbide (SiC) thin films is to produce a mask membrane suitable for X-ray lithography. *Lithography* is a process in the Integrated Circuit (IC) fabrication, where a pattern on a membrane, representing miniaturized geometric shapes of electric circuit is reproduced on a semiconductor wafer coated with a thin layer like silicon dioxide or silicon nitride[25].These geometric shapes define various regions in an integrated circuit such as the implantation regions, the contact windows and bonding pad areas. The patterns defined by this process are not permanent elements, but only replica of circuit features. In this process, a photosensitive resist layer covering the wafer is exposed to radiation like optical, UV or x-rays, for a pre-determined time. The radiation is passed through a mask membrane containing the required pattern. The pattern masks the radiation so that the layer beneath the pattern will not be irradiated. The pattern transfer is accomplished by an etching process which selectively removes unmasked portions of the resist which are exposed by radiation.

The pattern is generally formed out of metallic lines of fine line widths. A schematic for lithography is shown in Fig. 1.1.

Optical lithography is a common technique adapted for the pattern transfer. But, as the IC technology advances towards the fabrication of sub-micron electrical devices, this method imposes severe restriction on the resolution of the pattern that is reproduced. This is due to the large wavelength of the optic radiation, resulting in diffraction effects as passed through the membrane, containing pattern of line width of less than a micron. Hence other lithographic methods are being developed, one of which is x-ray lithography, where the wavelength involved will be about 0.5 to 3 nm. Here, diffraction effects are eliminated, resulting in high resolution. Apart from that, x-ray lithography has other advantages. X-rays can penetrate through dust particles of low atomic number[3]. Hence, this method has high immunity to contaminants present in the system. The low absorption of x-rays in the resist material results in uniform exposure and thereby vertical resist profiles can be formed. Scattering effects within the resist layer are minimized in this method. Also, the replicated pattern dimension is independent of the wafer material and the resist thickness. These advantages make x-ray lithography an important process technique in the future ultra large-scale integrated (ULSI) circuit technology.

A viable x-ray mask synthesis is a key component to the success of a submicron lithographic process. The fabrication of x-ray masks requires the use of an x-ray absorber consisting of a material with high atomic

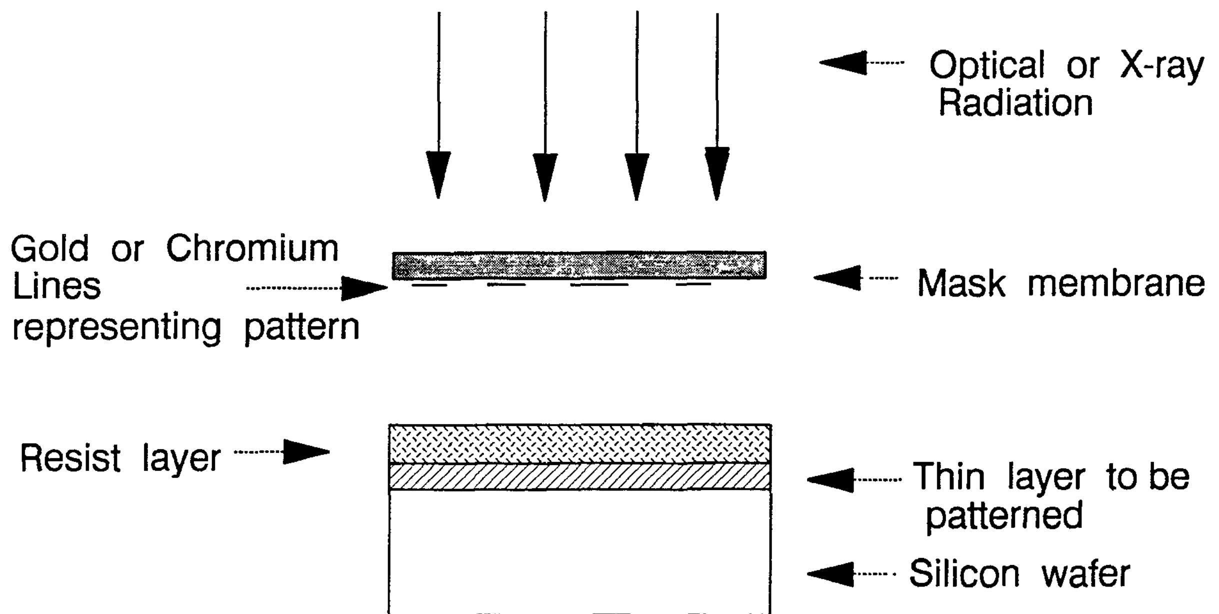

Figure 1.1: A schematic of lithography

number, such as gold to provide good pattern definition contrast. The membrane material for an x-ray mask technology aimed at providing 0.5-0.2 micron feature dimensions must meet several requirements. It should have:[12]

1. High x-ray and optical transmission.
2. High durability for x-ray radiation.
3. High modulus of elasticity to withstand the absorber stress.
4. Low tensile stress for the formation of crack free membrane.
5. Low surface defect density.
6. Long lifetime with mechanical stability so that the process can be repeated several times for increased throughput.
7. Low manufacturing cost.

Among various choices, Silicon Carbide (SiC) is found to be one of the suitable material, as a membrane formed out of this could satisfy the above requirements. The fact that SiC has higher Young's modulus (4.57 $N\,m^{-2}$ [13]) than any material other than diamond makes it a promising selection as a mask membrane material. Also, SiC finds various applications in industry. Its physical and chemical properties make it a suitable material for semiconductor device fabrication.

Recently many papers were published on SiC films, most of them regarding its crystalline form like beta-SiC [19], hetro-epitaxial SiC [16], 3c-SiC [9]

and amorphous hydrogenated SiC:H [23,27], were formed with more than one precursor. In this study, the fabrication of amorphous SiC films with single liquid precursor is presented. The films were deposited by low pressure chemical vapor deposition method.

Successful and economical production of such films involves a set of controlled deposition parameters. Therefore, a systematic study regarding the growth of the film, quality of the film, its electrical and optical properties with respect to these parameters is important. Hence a detailed description of method of deposition and characterization results is presented in the foregoing chapters.

1.2 Outline

In chapter 2, a brief review of literature survey regarding SiC is presented.

In chapter 3, common methods that are commercially used for the thin film fabrication are described. This is followed by the method used in this study, namely low pressure chemical vapor deposition (LPCVD) and its advantages.

Chapter 4, deals with experimental setup, description of LPCVD reactor, experiment parameter details and summary of charectarization studies involved.

In Chapter 5, various results about the nature of the films are discussed, followed by conclusion in the next chapter.

Chapter 2

LITERATURE SURVEY

Many research papers were published regarding the properties of SiC thin films of different types[28,15,13]. Its potential applications as semiconductor device has been extensively investigated. In this chapter a review of research on silicon carbide will be presented.

Non hydrogenated SiC film for x-ray mask synthesis was reported by A.Madouri A.M. Gosnet and J.Bourneix [15] and its properties were investigated. In that study, the stability of the film was found to be related to the strength of the chemical bond. The films were made by triode RF sputtering system. The a-SiC HIP target was sputtered with a RF power of 0.25 W/cm^2 under an argon pressure of 3 to 4 mtorr with substrate temperature of 450^oC. IR spectroscopic study showed a peak near 780 cm^{-1} and annealing studies under nitrogen ambient for 30 minutes, showed increasing stress of the membrane with increasing temperatures. A good quality membrane was achieved with 0.5 micron thickness and an area of 9 mm^2. Hetro-epitaxial SiC was synthesised and studied by M.Yamada, et.al. [16] in a cold wall LPCVD reactor at 1000^oC and at 3.5 torr using a gas mix-

ture of $SiHCl_3$, C_3H_8 and H_2. The stress for such film was found to be $4*10^9 dyn\,cm^{-2}$ and Young's modulus was determined as $4.7*10^{12} dyn\,cm^{-2}$. A carbonization layer was formed before SiC was grown. This was found to be effective in improving crystallinity. The formation of the buffer layer mitigates a lattice mismatch of 20% between SiC and Si and enhances the epitaxial growth. It resulted in decreased film stress.

Amorphous SiC was deposited onto fused silica by CVD using gas mixtures of $SiH_4, CH_4\ and\ H_2$ and its properties were reported by Y.J.Park, Y.W.Park and J.S.Chun [28]. It was found that as the deposition temperature was increased, the structure of the film became more ordered and the amount of hydrogen bonded to silicon atom decreased. The optical band gap was found to be in the range of 2.0 to 2.9 eV and decreased with increase in deposition temperature. At a deposition temperature of 700^oC, the resitivity of the film was found to be maximum. In another study, a-SiC x-ray mask was fabricated for sub-micron devices and pattern displacement test was reported by U. Mackens and others in SPIE [14]. The membrane was found to have high Young's modulus with excellent transperency for synchrotron and optical radiation. Pattern with 0.5 micron thickness had been generated by e-beam lithography as well as mask copying by using synchrotron orbit radiation. Almost similar results were given by H.Luthje, B.matthiessen, M.Harms and A.Bruns[13] regarding x-ray mask fabrication using mask blank processing sequence.

Use of SiC as semiconductor device fabrication material was noted by

H.S.Kong, J.W. Palmour, J.T.Glass and R.F.Davis[19].MESFET's were fabricated in an n-type β-SiC thin film grown by CVD on p-type β-SiC(100) which has been grown on p-type Si(100) substrate. The burried p-type layer allowed the devices to be fabricated several microns away from the SiC/Si interface region which contained numerous defects formed as a result of poor lattice match and different coefficients of thermal expansion between SiC and Si. Saturation of drain currents was achieved at room temperature, for these devices. The maximum transconductance obtained was $1.6mS\,mm^{-1}$. Depletion mode n-channel metal-oxide–semiconductor FET were fabricated on n-type β-SiC (111) thin films epitaxially grown by CVD on the Si(0001) face of 6H α-SiC single crystals and was reported by the same authors [10]. Transconductances as high as 11.9 $mS\,mm^{-1}$ were achieved. Stable transistor was observed at temperatures as high as 650^oC which was the highest temperature for a transistor in any material, at the time of report.

The role of addition of hydrogen in the formatiion of a-SiC from silane and ethylene mixtures was studied by Scott Meikle, Yoshiko Suzuki and Yoshinori Hatanaka [23]. In this study, the deposition rate was found to be increased as the hydrogen partial pressure fraction was increased upto 50%. For further increase of hydrogen concentration the rate was found to be decreasing. The optical band gap was found to be decreased as the H_2 pressure fraction was increased. XPS measurments showed that the density of Si-C bond increased as the slope of the optical edge becomes steeper. As an application to solar cell fabrication, high conductive p-type micro-

crystalline SiC:H was prepared using electron cyclotron resonance plasma CVD. This was done by Y.Hattori, D.Kruangam, T.Toyama, H.Okamoto and Y.Hamakawa [7]. The material with an optical band gap of 2.25 eV exhibits a dark conductivity as high as 10 $S cm^{-1}$. Utilizing this material as a wide-gap hetrojunction contact in amorphous silicon solar cell, a conversion efficiency of 12.0% had been obtained with a large open circuit voltage.

The effects of boron doping on hydrogenated a-SiC films were studied by H.D.Mohring,M.B.Schubert, G.Schumm and G.H.Bauer [17]. Here, these films were p-type doped in alternative process which is based upon plasma enhanced boron diffusion from thin SiB layers on the substrate into the growing intrinsic film. The band gap, refractive index, Penn gap and Urbach energy remain unchanged. Hydrogenated SiC films were fabricated by reactive R.F. sputtering and its nature had been investigated. This was reported by W.Weisweiller, G.Nagel and J.Klepp [27]. The film preparation was done at low temperatures by reactive rf sputtering in acetylene-argon gas. The chemical compositon and structure of these films were highly influenced by ratio of acetylene content in the plasma to the power density used. IR spectroscopic studies showed a absorption band centered at 790 cm^{-1} and the density of such films was 3.2 $gm\ cm^{-3}$. An atmospheric pressure plasma jet generated by inductively coupled RF discharge and containing vapors of Si compounds in argon-carrier gas was used to deposit a-SiC films [4]. Rapid deposition up to 10 $\mu m\, min^{-1}$ could be achieved in this method.

Highly photoconductive hydrogenated a-SiC films were prepared by al-

ternating monolayer deposition and hydrogen passivation by plasma-enhanced CVD in a mutizone apparatus. This was reported by A.Asano, T.Ichimura and H.Sakai[1]. In this, films with photoconductivity of $3 * 10^{-5}\ S\ cm^{-1}$, an optical band gap of 1.91 eV was prepared. In another study, a high-rate depostion around 10 $\mu\,m$ per hour with a optical band gap of 2 to 2.1 eV and with high photosentivity exhibiting a conductivity change more than 10^4 of magnitude under illumination with $50\mu W\ cm^{-2}$ in intensity was investigated using a glow discharge in $SiH_4 - C_2H_2 - H_2$ mixture gas. This was reported by Y.Nakayama, S.Akita, M.Nakano and T.Kawaura [18]. It was showed that C_2H_2 based gas system has high efficiency of the carbon incorporation into the films. A.Raveh, A.Inspecktor, U.Carmi and R.Avni [20] gave a report regarding the properties of SiC films deposited on martensitic stainless-steel plates using rf plasma reactor from a mixture of tetramethylsilane and hydrogen in argon. The film was identified as hexagonal a-SiC with free carbon embedded in it. The hydrogen concentration influenced the structure and composition of the film. The microhardness and density were found to be depend on the total carbon content in the film.

Another potentential application of SiC film in the fabrication of visible light LED was reported by Y.Hamakawa, D.Kruangam, H.Okamoto and H.Takakura [6]. Injection-type electroluminescence thin film diode made of a-SiC was fabricated and it had a basic structure of p-type layer followed by intrinsic and a n-type layer. The emission color was found to be controlled from red to green by adjusting the carbon content of the $ia-SiC$ layer. The brightness of $5\,cd\,m^{-2}$ was obtained for yellow LED with forward injection

current density of 200 $mA\,cm^{-2}$. Films of p^{+} microcrystalline SiC:H films were deposited and studied by B.Goldstein and C.R.Dickson and others[5]. These films grown by conventional rf glow discharge showed conductivites $2 - 2 * 10^{-3}\Omega\,cm^{-1}$ and activation energies 0.05 to 0.1 eV with carbon concentrations 0-6 atomic%, respectively. Increasing carbon content was found to supress the microcrystallinity. Ion implanted p-n junction diodes in were studied by J.A.Edmond, K.Das and R.F.Davis [2].

These studies indicate the significance of SiC films in its various structural forms. Concluding the discussion, it can be noted that the deposition of a-SiC using a single precursor will be worth investigating. In this study, such films were deposited using safe precursor at relatively low temperatures, and by simple method, namely, low pressure chemical vapor depostion.

Chapter 3

METHODS OF THIN FILM DEPOSITION

Thin film fabrication is a complicated process which involves many controlled processing parameters. The time of processing, equipment cost, throughput and quality of films are important factors for a successful commercial production. Many techniques are available in the present technology for film fabrication. But, most common methods that are used for economical production are physical vapor deposition (PVD) and chemical vapor deposition (CVD). This chapter describes these two methods in detail. The reason for choosing CVD method for this study is also explained.

3.1 Physical Vapor Deposition (PVD)

In this method thin film material in gaseous form is allowed to deposit on the substrate directly. No chemical reaction is involved in this method. All PVD process proceed according to the following sequence of steps:

1. The material to be deposited (solid or liquid source) is physically converted in to a vapor phase;

2. The vapor is transported across a region of reduced pressure (from source to substrate);

3. The vapor condenses on the substrate to form a thin film.

Depending upon the method of converting the deposition material to vapor phase, PVD is classified as: PVD by Sputtering and PVD by evaporation.

3.1.1 PVD by Sputtering

Sputtering is a term used to describe the mechanism in which atoms are dislodged from the surface of a material by collision with high energy particles. Here, ions are generated and directed at a target; the ion sputter target atoms; the ejected (sputtered) atoms are transported to the substrate where they condense and form a thin film. The advantages of this method are: (i) Sputtering can be accomplished from large-area targets (ii) film thickness and its quality like step coverage, grain structure can be controlled easily. But, this method suffers from drawbacks such as: (i) high equipment cost; (ii) low deposition rate for some materials (iii) impurity incorporation in the film due to deposition in low-medium vacuum conditions; (iv) inability of some materials to withstand ion bombardment resulting in degradation.

3.1.2 PVD by Evaporation

Thin films can be deposited by applying heat to the source of film material, thereby causing evaporation. If the heated source resides in a high-vacuum environment, the vaporized atoms or molecules are likely to strike the substrates (or chamber walls) without suffering any intervening collisions with other gas molecules. The advantages of this method are: (i) high deposition rate (ii) no substrate surface damage as the energy of the impinging atom is less (iii) high purity films can be achieved due to the deposition occurring in high vacuum. The disadvantages are: (i) accurately controlled alloy compositions are more difficult to achieve (ii) *in situ* cleaning of the substrate surface is not possible with evaporation system (iii) x-ray damages, caused by e-beam evaporation processes in sputter deposition, is avoided in this method.

Considering the principle involved in PVD, it should be noted that the deposition material should be easily changed to gas phase. But, since SiC can withstand high temperature, this method is not suitable for fabrication. Without discussing further, the other common method of film deposition, chemical vapor deposition is considered.

3.2 Chemical Vapor Deposition (CVD)

CVD is a process where one or more gaseous species react on solid surface, forming a solid phase material as one of the reaction products, the others being mostly in gaseous form.

Here, the solid product may form a film or massive bulk on the substrate which enhances the reaction but does not undergo any chemical change. Many parameters like deposition temperature, pressure, flow rate, reactor geometry determine the nature and the quality of the deposit thus produced.

Most of these chemical reactions are endothermic and of different types such as pyrolysis, decomposition, oxidation, reduction, chemical transport reaction etc. The process should have the following sequential steps for completion:

- Mass transport of reactants to substrate
- Adsorption of reactants on substrate surface
- Chemical reaction on the surface
- Desorption of product gases from the surface
- Mass transport of product gases away from substrate

CVD process has several advantages over other methods of film deposition. These advantages make it one of the popular methods adapted commercially. Theses are listed below:

1. Conformal step coverage can be achieved by this method. It is an important factor for the fabrication of electrical devices.
2. Stoichiometric control of the film can be easily achieved by adjusting the processing parameters like deposition temperature, flow rate etc.

3. High throughput is possible, especially by LPCVD, due to reactor structure and enhanced mass transfer of the reactant.

4. The quality of the film can be easily reproduced by this method because the processing parameters can be repeated without difficulty.

5. Since the whole area of the substrate is equally exposed to the reactant good uniformity of film thickness can be achieved.

6. Selective deposition of film onto the substrate can be done by masking unwanted portions of the substrate.

7. Unlike in PVD, there is no radiation like e-beam involved, therefore, there no possibility of damage of substrate or the film, by this method.

8. CVD offers an excellent opportunity of impurity doping in device fabrication. This is possible by simply mixing the required dopant along with the reactants.

9. Low maintenance cost of the equipment makes it an attractive method for large scale production.

3.2.1 Types Of CVD Processes

Depending upon the type of energy that applied for the reaction to occur, CVD processes can be classified as: (i) Plasma enhanced CVD (ii) Photo induced CVD (iii) Thermally activated CVD. [11]

3.2.2 Plasma enhanced CVD

In this method, gaseous reactants are allowed in a region of glow discharge created between two electrodes by electric supply. Highly reactive species

are created in this region resulting in interaction between these species thus forming a solid thin film product on the substrate and electrode surfaces.

Glow discharge are usually created at low pressures in the 0.01 to 1 torr range. This causes breakdown of molecules in to reactive species like ions, electrons. Electric field due to ac, dc or microwave sources across two electrodes creates plasma region between the electrodes.The molecules themselves can be near to the ambient temperature but the breakdown electrons will be at higher temperature causing the reaction. Thus this method can be employed at relatively low temperature and it is useful for temperature sensitive materials.

Film deposition rates are substantially higher in this method than in thermally activated LPCVD. Also, conformal step coverage can be achieved. But the disadvantage of this method is the complex process that occurs in the plasma state makes the synthesis of stoichiometric films difficult. The low deposition temperature of film formation results in gases trapped in the film, which frequently causes thermal instability due to outgassing.In a newly developed method, high density plasma is created using electron cyclotron resonance ion source. The main feature of this method is low deposition temperature that is needed for high growth rate. [11]

3.2.3 Photo induced CVD

Short wavelength UV radiation is used to activate the reactants in gaseous phase forming the product material. A selective absorption of photonic

energy by the reactant molecules or atoms initiates the process. Typically, mercury vapor is added to the reactant gas mixture as a photosensitizer and is being activated by the radiation from a high intensity quartz mercury resonance lamp (253.7 nm wavelength). The advantage of this method is low deposition temperature needed for films like SiO_2 and absence of radiation damage like the previous method. The limitation of this method is unavailability of effective production equipment.

In another type, laser beams are used for activating the reactants. In pyrolysis type reaction, a highly localized heating of the substrate that induces film deposition by CVD surface reactions, and can be exploited for the direct writing of patterns on a substrate. In evaporation method, the laser simply acts as an energy source to vaporize atoms from a target to the substrate. In yet another type, the reactant atoms or molecules absorb a specific wavelength of the laser energy applied resulting in chemical gas phase reaction that are very specific, leading to highly pure film deposits. But, these methods are still in developing stages.[11]

3.2.4 Thermally activated CVD

This process uses direct thermal energy for the chemical reaction. The simplest type of this method is conventional atmospheric pressure CVD where, the reactant gases are allowed in to the chamber at normal atmospheric pressure. Energy is supplied by heating the substrate directly. The temperature and reactant flow rate determine the film growth rate. The advantage of APCVD is it simplicity that it needs no vacuum pumps. The

disadvantage is the tendency for homogeneous gas phase nucleation that leads to particle contamination, unless special optimized gas injection techniques are used.

The deposition rate and uniformity of films created by all CVD process are governed by the rate of mass transfer of reactant gases to the substrate and the rate of surface reaction of the reactant gases. In atmospheric CVD, these two rates of same magnitude. Lowering the gas pressure enhances the mass transfer rate relative to the surface reaction rate. This makes it possible to deposit films uniformly in a highly economical close spaced positioning of the substrate wafers kept vertically inside the chamber. Thus low pressure CVD (LPCVD) is a widely used method in cost competitive semiconductor industry. Another advantage of this method is gas phase nucelation is very much reduced. This is a suitable method for the SiC deposition.

Depending upon the supply of energy, CVD can be further classified as hot wall and cold wall reactor system. In hot wall reactor system, the reactor is heated to high temperature and the gas molecules hitting the wall receive the thermal energy. Here wafers are not heated directly. The advantage of this system is a temperature gradient can be provided to the chamber which results in uniform thickness. In the other type, the wafers are heated to high temperatures directly. The reactants that are adsorbed on the surface undergo chemical change due to the temperature of the wafer. But, controlling the wafer temperature is difficult and hence uniform depo-

sition is also difficult.

3.3 Summary

The common methods of film deposition, namely PVD and CVD, were described. Their various classification, advantages and disadvantages were mentioned. The LPCVD was found to be suitable method for its advantages compared to APCVD. Therefore, in this study, hot wall low pressure chemical vapor deposition system was employed.

Chapter 4

EXPERIMENTAL SETUP AND CHARACTERIZATION TECHNIQUES

In this chapter details of LPCVD reactor, experimental procedure,summary of experiments and the theory involved in film characterization techniques are discussed.

4.1 LPCVD Reactor

The schematic for hot wall low pressure chemical vapor deposition reactor is shown in the Fig 4.1. The reactor consists of fused quartz tube of 5 inch in diameter and about 50 inch in length. The tube is kept inside a *Lindberg* three zone furnace. The zone temperatures are controlled by manual settings. A maximum temperature of 1200^oC can be reached using this furnace and a gradient of temperature can also provided inside the quartz tube. Heating is provided by Lindbar silicon carbon heating elements. It is equipped with Platinel II thermocouples which senses the temperature of

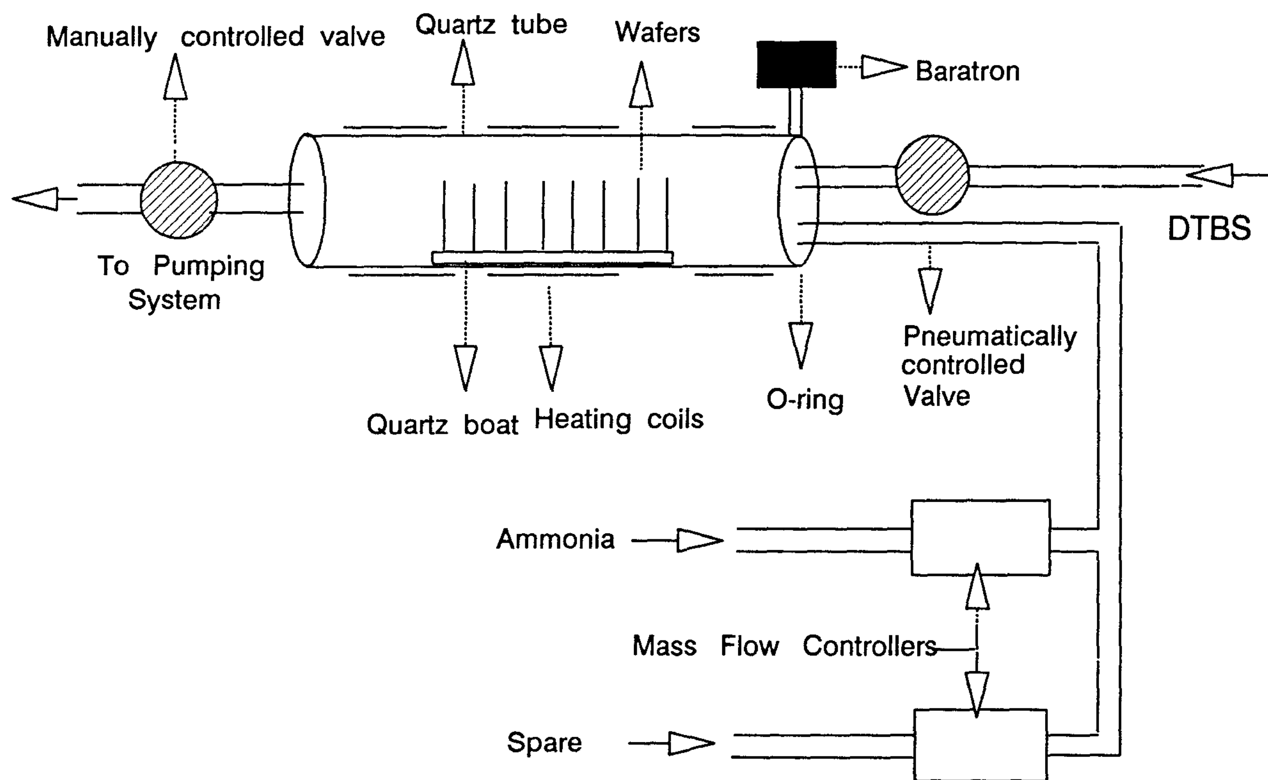

Figure 4.1: A schematic for LPCVD reactor

the zone and the voltage developed by it is used for automatic control. The tube and the coils are covered with ceramic enclosure. The tube is sealed on both ends by end caps and metallic lids. During heating process, thermal expansion of O-rings may cause leakage in the system. To avoid this problem, water cooling is arranged by cold water circulation (not shown in the figure). Apart from this, additional cooling is done by fans when the temperature of the furnace is increased more than $400^{o}C$. A MKS Baratron gauge with a range of 10 torr is used to monitor the pressure at the input end. The monitored pressure is displayed by MKS display unit. The input seal consists of three provisions for gas inlet, so that, if more than one precursor are used, they will mix together and diffuse inside.

The system will be kept at low pressure by vacuum pumps. This system uses a booster pump and a mechanical backing pump. Booster pump is used to enhance the flow of gases and thereby pumping speed. Mechanical backing pump does the real pumping and this combination provides a vacuum of low as a milli torr. Booster pump is a *Ruvac* single stage roots pump operated at 220 V supply and that the backing pump is a *Trivac* dual stage rotary vane pump. Nitrogen ballast gas is used in the pump to dilute any hazardous outgoing gas. An oil filtration system is also used to separate the micron size dust particles that are accumulated during pumping process.

Wafers are loaded inside the tube using a quartz carrier boat. Wafers are kept vertically in the slots provided on the boat. The boat is kept inside

the quartz tube and the tube is sealed by inlet lid. A manual control valve is provided at the output end to control the rate at which gas is removed from the reactor and therefore controls the pressure inside the reactor. Precursor is allowed through a pneumatic control valve provided at the input end. Unloading of wafers is done by bringing the reactor to atmospheric pressure. This is done by closing the output valve and passing a controlled flow of nitrogen, into the chamber.

4.2 Experiment Procedure

Silicon Carbide thin films were deposited using single liquid precursor, namely Di tertiary butyl silane (DTBS). This chemical was patented by Olin Hunt Inc., in 1989. Its chemical formula is $SiH_2(C_4H_9)_2$. It has a molecular weight of 144, vapor pressure of 20.5 torr at 20^oC and boiling point of 120^oC at 760 torr. This vapor pressure of the liquid enables a easy vapor transfer of the liquid in to the chamber at room temperature.

Films were deposited on $< 100 >$ oriented single and double sided polished silicon, and fused quartz wafers. Six to ten wafers were used for each experiment. Wafers were weighed before loading, by electronic weighing balance. After loading, the furnace was brought to low pressure by pumping down the chamber for about an hour. Then the temperature was raised to desired level in steps of 200^oC. The flow rate of the gas was fixed by setting the output valve at a particular fixed position and adjusting the valve of the liquid container to get a pressure of 0.2 torr at 600^oC. This

was repeated for every experiment. This procedure was adapted due to the unavailability of proper flow meter.

After the run was completed, wafers were weighed once again. The difference of weight gives the mass deposited. Knowing the time of run, the deposition rate was determined. Thickness of double sided and single sided polished silicon wafers are measured using nanospectrometer. IR spectroscopic analysis was done using PERKIN ELMER Infrared Spectrophotometer-580. Optical microscopic observation and x-ray diffraction studies were performed for selected wafers from each run. This was followed by etching studies. Attempts were made to develop a circular membrane of about 2 cm diameter, on single side polished wafer. This was done by etching the film deposited on rough side and also the wafer over the circular region, leaving behind the film deposited on the polished side, as a membrane. The back side film of the wafer was etched by plasma etching. PLASMA II AMP 3300 reactor was used to etch the film. This was done with 250 W rf power and at base pressure of 0.007 torr. CF_4 with 8% of O_2 was used as process gas. Substrate etching was done using KOH, iso-propanol and water mixed in a ratio of 1:1:1 and at a temperature of 70°C. Finally, samples from each run were sent to Fort Monmouth and Olin Hunt Inc., for compositional analysis.

4.3 Factors Affecting The Quality Of The Film

In a CVD process, the parameters that affect the rate of growth and the nature of the film are as follows:

- Deposition temperature
- Flow rate of the reactant gas
- Pressure of the chamber
- Wafer spacing inside the chamber

These parameters were noted down for each run including the time of run. The time of run determines how thick the film will be. The above factors were considered for the film analysis purposes.

4.4 Summary Of Experiments

Totally 21 runs were performed. The range of deposition temperature was from 500 to 850$^\circ C$. These runs were performed at different pressures ranging from 0.05 to 1 torr. Experiments from 500 to 700$^\circ C$ were done with a flow rate of 30 sccm and at a pressure of 0.2 Torr. Experiments from 750 to 850$^\circ C$ were done with a flow rate of 15 sccm. Three experiments were performed with addition of ammonia and one with acetylene. Wafers deposited at 750$^\circ C$ were annealed at 900$^\circ C$ for 45 minutes. The thickness of the film thus produced was different for various runs ranging from 400 Å to 17000 Å per side.

4.5 COMPOSITIONAL ANALYSIS TECHNIQUES

The compositional analysis is essential as it gives important information like stoichiometry of the film, the presence of other unwanted elements which affect the quality of the film etc. Here, two important techniques which were used for the analysis of silicon carbide films are discussed.

RUTHERFORD BACKSCATTERING is a surface analysis technique that can give quantitative information on elemental composition and depth profiling without the use of standards. In this method high energy ions are allowed to collide with atoms and the energy of the recoiled ions are measured. If it is a perfect elastic collision, the energy of the backscattered ion depends upon the energy of the atom with which it collides. Typically doubly ionized helium ions accelerated to 2 Mev are used for this purpose. This procedure is sensitive to detect only heavier atoms and cannot be used for detecting hydrogen. The probability of a scattering collision occurring is larger for heavier atoms. Therefore the count, that is, backscattering yield will be more for heavier atoms and will be less for lighter atoms. It is easy to detect the presence of two atoms with large difference of atomic number. Apart from that, this method can provide the concentration of atoms versus depth. As the ion traverses the sample, it loses energy because it undergoes many grazing collisions before it hits an atom and recoils back. Similarly, on its way back also, it loses some energy. Thus the energy of the

returning ion indicates the depth of collision. The depth resolution that can be achieved by this method is 10-200 Å. RBS is a non-destructive method, since there is no sputtering effects involved. Crystallographic information like location of atoms on lattice sites can also be obtained by this method.

ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS is another technique which was followed by Olin Hunt Inc., for the composition analysis. In this technique a beam of low energy x-rays like the K_{alpha} line of aluminum at an energy of 1.487 keV is used to bombard the sample. The x-ray photon energy removes an inner shell electron from an atom when it is absorbed. This electron is emitted with a kinetic energy characteristic of the difference between the x-ray and the binding energy of the electron. The energy of the emitted electron defines the type of the atom and the number of electrons at this energy is related to the density of atoms present. This method can be used to examine several top monolayers of thin film and it also offers information regarding the chemical bonding of the elements. But due to focusing difficulties of x-rays, this method has been generally done over several mm diameter samples. This method cannot detect hydrogen or helium.

INFRA-RED SPECTROSCOPY is a technique used to determine the chemical nature of thin films. Some materials will absorb certain frequencies in the infra-red region (wavelengths 2 to 25 microns) because of the excitation of vibrational energy transitions in molecular species. In the same way that electronic transitions in atoms can absorb radiation of specific

frequencies, the vibration of a molecule (stretching or bending) will have a resonance value, and it will be excited by any radiation of this frequency. When IR radiation of a particular frequency impenges a sample containing molecular species, it may or it may not be absorbed. If all frequencies are passed through, some will be absorbed to varying degrees depending on the molecular species involved. The intensity of the return radiation therefore depends upon the vibrational mode of the molecules. The presence of a particular molecular vibration can be detected by an absorption peak in the spectrum.

Chapter 5

FILM CHARACTERIZATION RESULTS

Various results regarding the nature of the film will be discussed here. Film growth rate analysis, density analysis and compositional analysis are explained in detail. The problem encountered in the formation of membranes during etching process is also described.

5.1 Film Growth Rate Analysis

One of the important aspects of nature of the film is the rate of film growth, with respect to deposition temperature. Since a chemical reaction is involved in the formation of the film, the rate of chemical reaction is a key factor for deposition rate. The rate of chemical reaction depends upon the deposition temperature, if the reaction is endothermic. SiC films were deposited at different temperatures, from 500-850^oC. Other parameters like pressure, flow rate and wafer spacing were kept constant. For every experiment the growth rate, that is, thickness deposited per hour is calculated

for each wafer and average was taken.

Generally, the rate of most reactions increases as the temperature is raised. It can be stated approximately that the rate doubles for every 10^oK increase in temperature. But the temperature dependence of rate has been found to fit the expression proposed by Arrhenius:

$$GrowthRate(G.R) = A\ exp(-Ea/RT). \tag{5.1}$$

where: A is pre-exponential factor (nearly independent of temperature) and Ea is Activation Energy, which has to be supplied for the reaction to occur. Activation energy depends upon the chemicals that take part in the reaction. R is the gas constant and is equal to 1.98717 $cal\ K^{-1}mol^{-1}$ and T is the absolute temperature of the reaction.

Taking natural logarithm (Ln) on both sides of the above equation, and plotting a graph of Ln growth rate versus reciprocal of temperature, leads to a straight line with negative slope. This type of curve is called *Arrhenius plot.* The negative slope of the curve gives the ratio between Activation Energy and Gas constant. By determining the slope of the curve, the activation energy for the reaction can be calculated.

Fig 5.1 shows Ln of growth rate (Angstrom per minute) plotted against reciprocal of deposition temperature in Kelvin scale. From 600 to 675^oC the deposition rate follows Arrhenius behaviour. After 700^oC the G.R saturates and showed no rise. There was no or little deposition below 600^oC, which

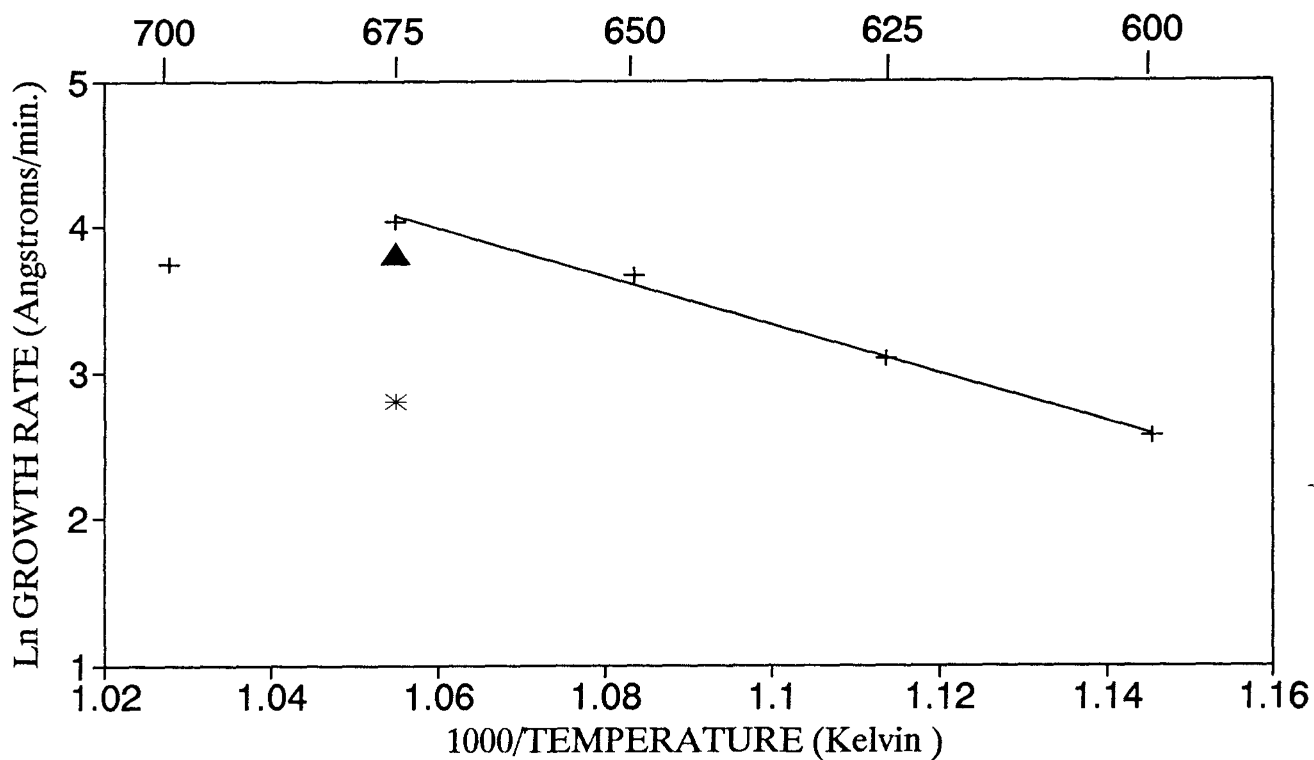

Figure 5.1: Effect of temperature on deposition rate, showing the Arrhenius behavior between 600 and 675°C.

was evidenced by negligible deposition at 500^oC. The slope of the curve was found to be -16.25 giving an activation energy of about 32 $k\,cal\,mol^{-1}$. The points in the line shows the experiments conducted at same pressure of 0.2 torr and at a flow rate of 60 sccm. At 675^oC, the experiment was repeated with different pressures of 0.1 and 0.05 Torr. The G.R was found to decrease as the pressure is decreased. This is shown by two different points on the same 675^oC line. The deposition rate was found to be maximum at 675^oC of about 40 $\AA$ per minute.

5.2 Density analysis

The density of the deposited film was found to be independent of deposition temperatures. A graph plotted by taking thickness of the films deposited at various temperatures and for various length of time, against the mass deposited on the same wafer, is shown in Fig. 5.2. Double side polished wafers were taken into consideration for this analysis. The thickness of the film deposited on each side is measured using *nanospec* at six different points. A refractive index of 2.7 was taken for calculation as per the experimental verification using ellipsometer. The average thickness for each side was added together and was taken for plotting the graph. The mass deposited on the film was measured using electronic weighing balance. The graph shows the mass of the film deposited for a various deposition temperatures in the x-axis. The y-axis shows the corresponding thickness deposited on both the sides. The linearity of the curve indicates that the density is independent of temperature. Density is calculated from the fundamental

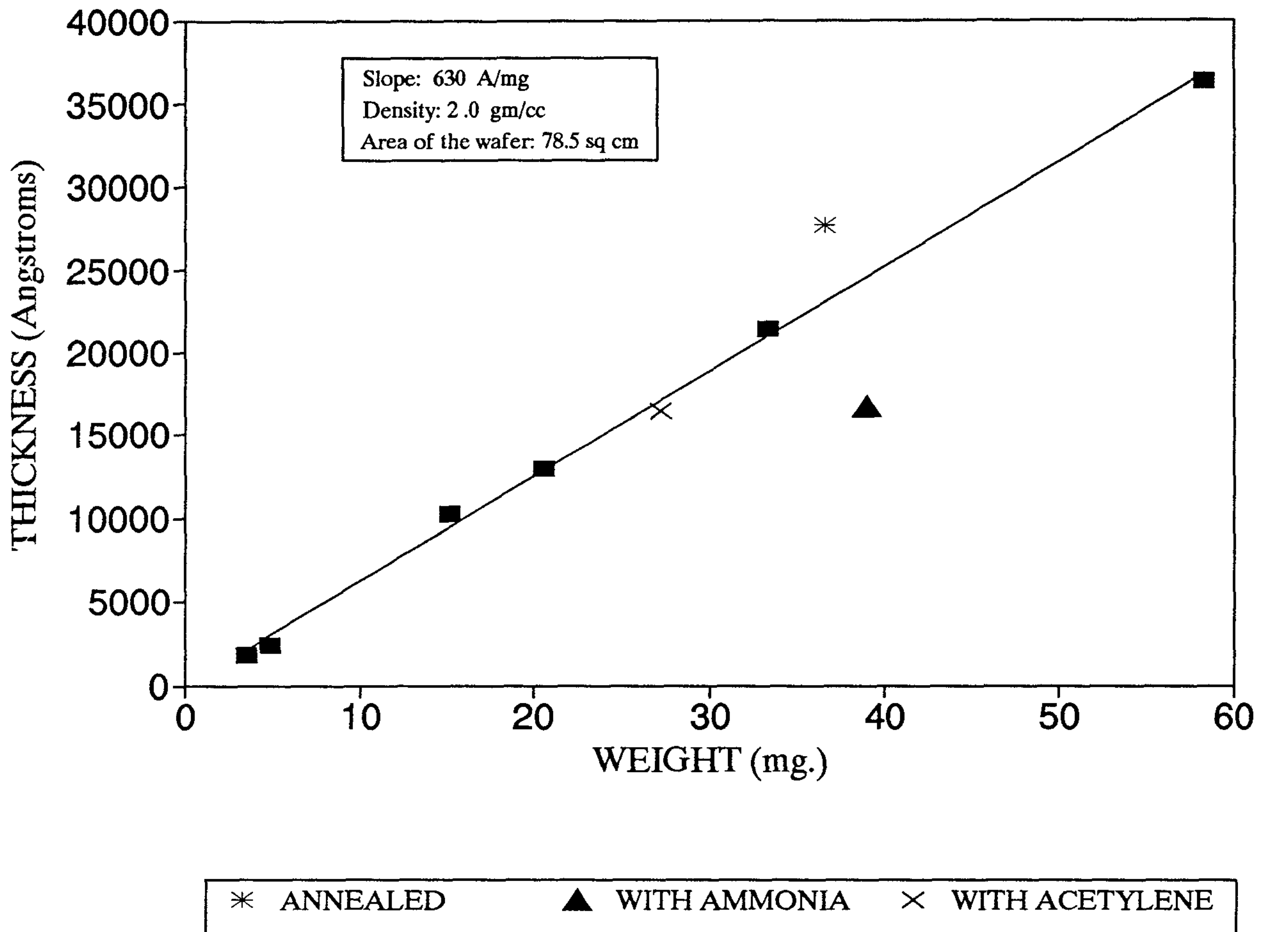

Figure 5.2: A plot of thickness of the film versus mass deposited on the wafer, showing that the density of the films is independent of temperature.

mass-volume relation. As the area of the wafer is constant, density can be calculated from the slope of the curve. The slope of the curve was about 630 $\AA$ per gm. and the density thus estimated was around 2.02 $gm\ cm^{-3}$. Regression analysis was carried out for the plot and the extrapolated points in the curve were well within one standard deviation. Points with different shapes correspond to the thickness of the film deposited with different conditions. When acetylene was added to the precursor, carbon content was increased but the film density did not deviate much from other films deposited without addition of any other gas. This was evidenced from the curve. But, when the wafer was annealed (at $900^{o}C$;for 45 minutes) the film became very tensile developing lot of cracks and this may be a reason for the low density of the produced film. Addition of ammonia showed an increase in density due to the incorporation of heavier atom of Nitrogen, in the film. The density of the films was found to be constant irrespective of the change in the stoichiometry of the films at higher deposition temperatures, due to the corresponding change in volume of the films.

5.3 Compositional Analysis

Compositional analysis of samples deposited at various temperatures showed a marked change of stoichiometry of the film. Independent analysis of samples for the percentage of silicon and carbon present were carried out at two different places, Fort Monmouth and Olin Hunt Inc. Fort Monmouth Rutherford Backscattering (RBS) analysis and Olin Hunt used electron spectroscopy for chemical analysis (ESCA) and Auger analysis The results obtained from both of them showed that the deposition temperatures play

an important role in the stoichiometry of the film.

For the analysis of SiC samples, doubly ionized helium (He^{+2}) with energy of 2 to 3.4 Mev, depending upon the film thickness was used. Fig 5.3 shows the RBS spectrum for a sample deposited at 675°C. The x-axis shows the energy of the backscattered beam and the y-axis shows the normalized yield. The sample thickness was found to be 4739 $\AA$. The estimation of percentage composition of silicon was determined by comparing the amplitude of the peak corresponding to the silicon energy level to that of the amplitude of the peak corresponding to the energy level for standard 100% silicon. For this sample the atomic composition was determined to be $Si_{1.08}C_{0.92}$. (Si 55%, C 45%).

Fig 5.4 shows a ESCA survey of a sample deposited at 850^oC. The sample was etched for 100 $\AA$ before analysis. This is to disregard any oxide incorporated when the film is exposed to atmosphere. The x-axis shows the binding energy of the emitted electrons. The y-axis represents intensity level. Silicon and carbon percentage was estimated by the level of peaks corresponding to its binding energy levels. For this sample the atomic composition was estimated as:Si-24%, C-70%, O-5%, N-1%.

The stoichiometric change through the temperature range of 600°C to 850°C is shown in the Fig 5.5. Here, both RBS ESCA results are depicted. The graph was plotted by taking the ratio of atomic composition of silicon to that of carbon, of films deposited at various temperatures. Lower the

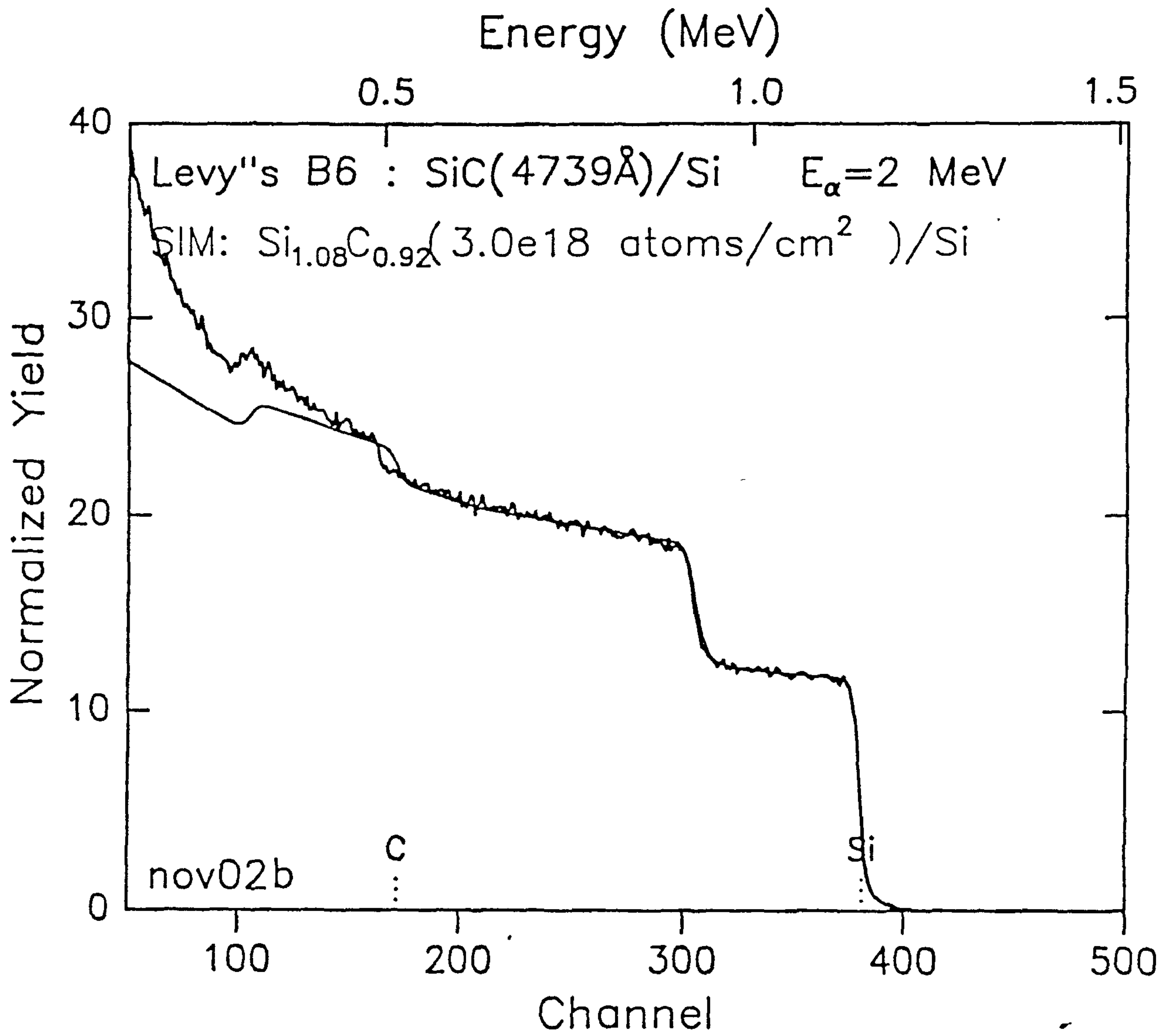

Figure 5.3: RBS spectrum for a sample deposited at 675°C.

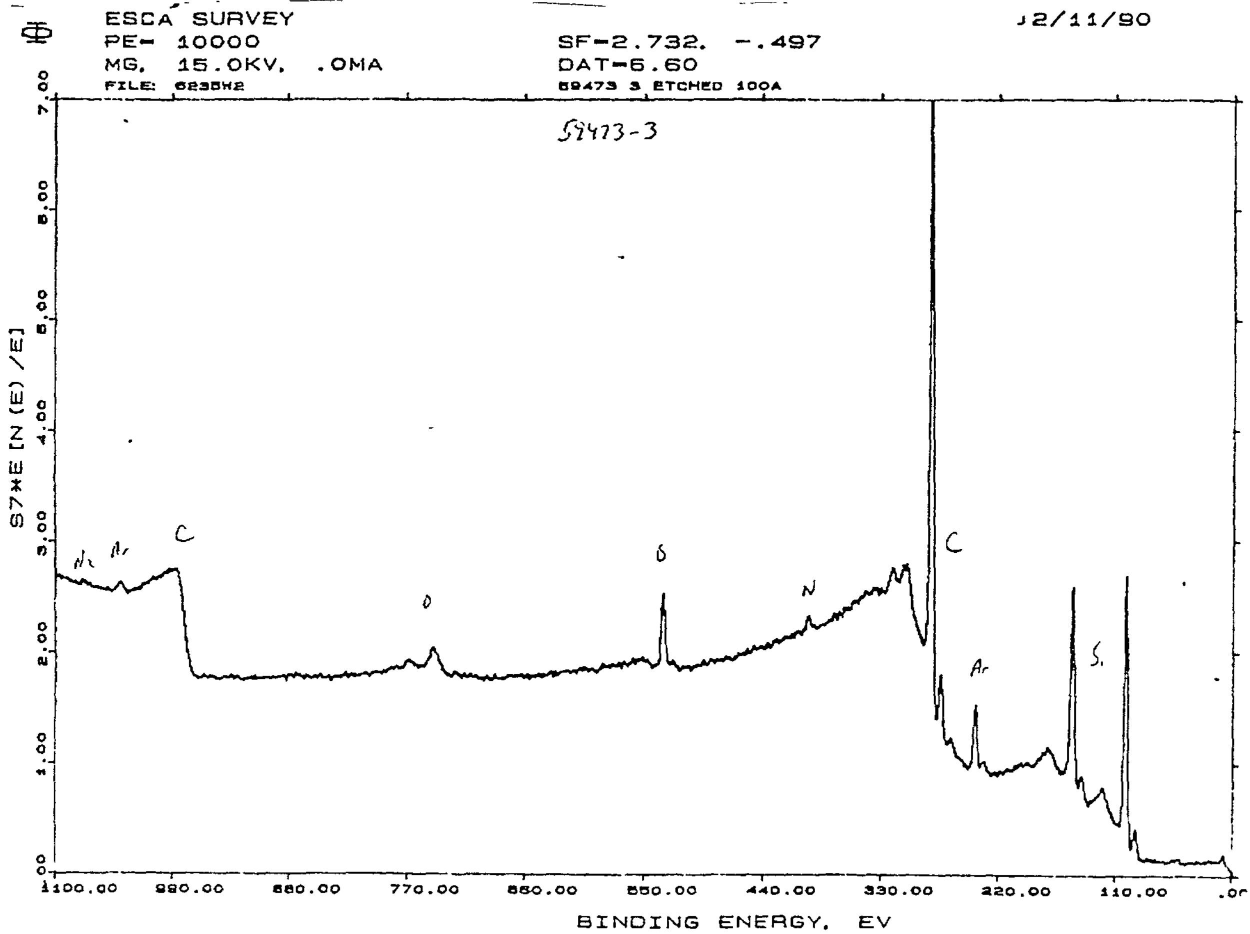

Figure 5.4: ESCA survey for a sample deposited at 850°C.

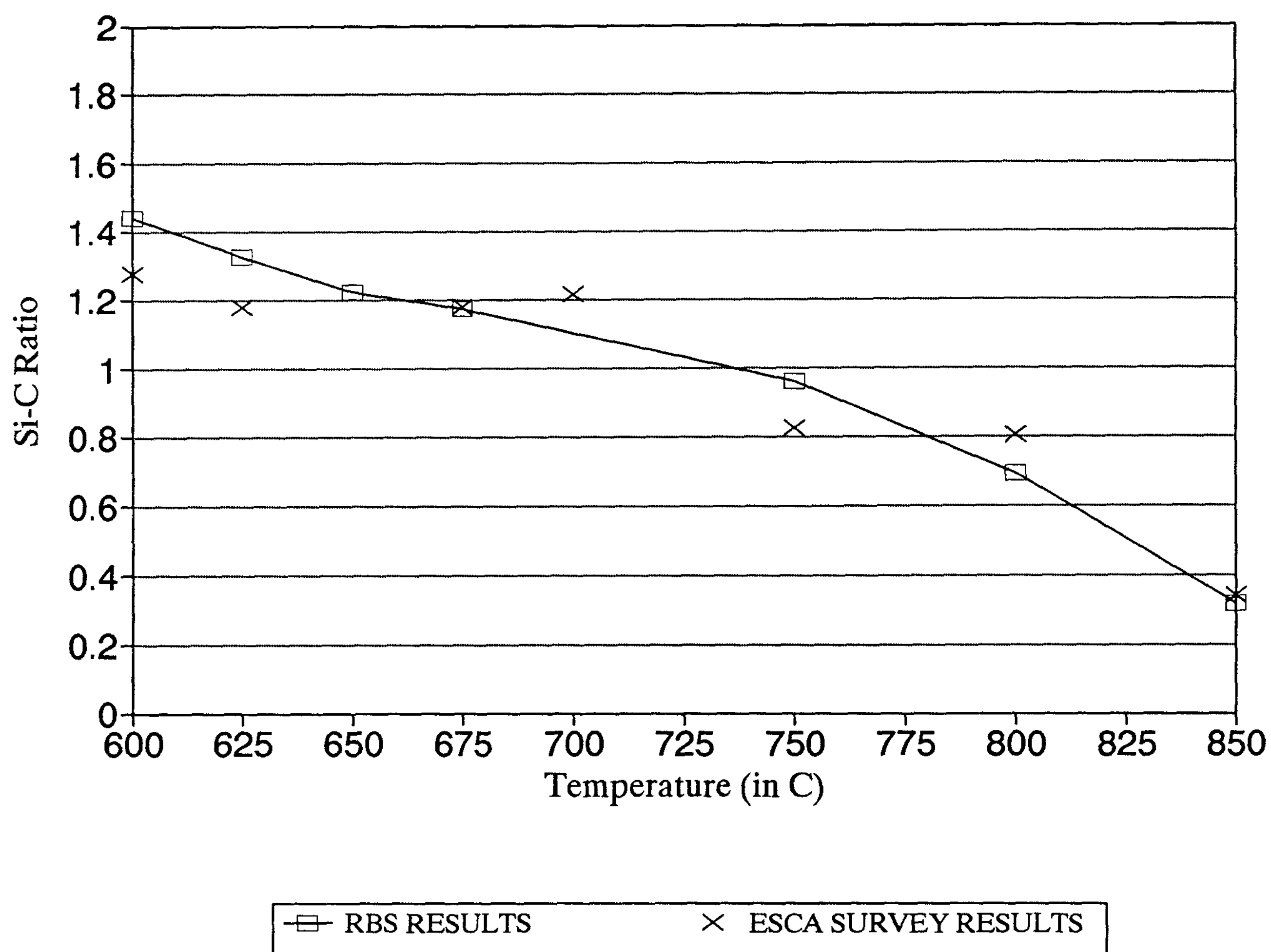

Figure 5.5: Plot of Si-C atomic composition versus deposition temperature.

ratio means higher the carbon content. A ratio of 1 corresponds to soichiometric film. The percentage composition of silicon is found to be decreased as the deposition temperature is increased. From ESCA analysis it can be seen that the stoichiometry of the film was almost the same (Si-55% and C-45%) between 600-700^oC. After that temperature the percentage of silicon dropped steadily and at 850^oC, it was 24%. At a deposition temperature of 750^oC nearly stoichiometric film was achieved. The addition of acetylene was shown to be an alternative mean of varying the stoichiometry of the film. At a deposition temperature of 675^oC and pressure of 0.1 torr, the addition of 30 sccm of acetylene to 15 sccm of DTBS resulted in films with a composition of Si-40% and C-60%.

5.4 IR Spectroscopic Analysis

A plot of energy level of the reflected IR spectrum versus wavenumber (reciprocal of wavelength) can be used to detect the presence of vibrational mode of a particular molecule. IR spectroscopic analysis were carried out for various samples. The absorbed wavelength was shown by the peak which is centered at 780 cm^{-1}. This is in good agreement with the standard SiC absorption peak. Fig 5.6 shows such an IR spectrum for two samples deposited at 675^oC and at 650^oC one below the other. Both the spectrum show the absorption peak centered at the above mentioned wavenumber. IR spectroscopic study also revealed that there was no incorporation of hydrogen in the film. Also, addition of ammonia with DTBS produced a Si-C-N type film. This was evidenced by an additional shoulder peak in

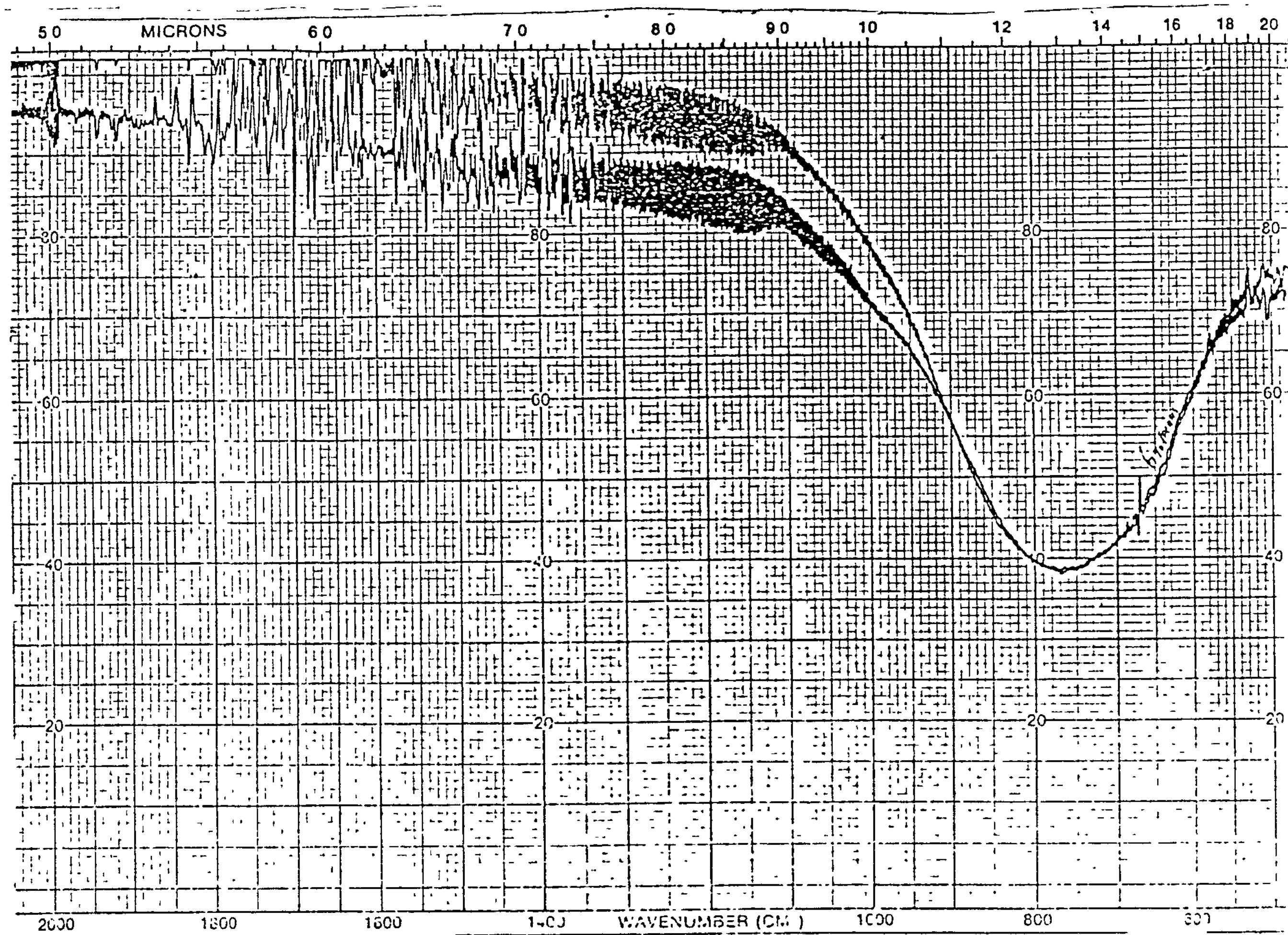

Figure 5.6: IR spectrum for samples deposited at 675°C and 650°C, showing the presence of Si-C vibrational mode.

the IR spectrum centered around 830 cm^{-1}, indicating Si-N vibrational mode.

5.5 X-Ray diffraction studies

X-ray diffraction studies of these samples showed that the films were amorphous in all cases. X-ray diffraction analysis were carried out using Rigaco x-ray diffractometer, with the K_{alpha} lines from a copper target used for the analysis. Fig 5.7 shows the diffraction pattern obtained for the sample deposited at $750°C$. This is a plot showing the normalized diffracted energy level versus twice the angle of incidence of x-rays on the sample. In the figure, peaks correspond to the silicon substrate and the curved line indicates the amorphous nature of the film.

5.6 General appearance and optical microscopic observation of films

Films with less deposition showed colored rings around the edges of the wafer. The films were susceptible to scratching and peeling. Films with more than two microns started flaking off the wafer when they were left for few days. Generally the films were found to be tensile as evidenced by presence of microcracks observed through optical microscope. No microcracks could be observed for the films around $1000\AA$ thickness. But, films deposited at $650°C$ or more having thickness of more than 1000 $\AA$ have this problem. Fig 5.8 shows the of the film magnified 110 times. Film on silicon substrate and quartz substrate are shown in the order. It is inter-

esting to note that the microcracks on silicon substrate follow a pattern. It is believed that stress release along $< 111 >$ or $< 110 >$ plane may be the reason for the formation of such cracks. This problem was aggravated during etching process. The etching process could not be completed as the film developed more cracks and flaked off. The development of cracks due to etching is shown in Fig 5.9. The method to avoid the formation of such cracks is yet to be solved.

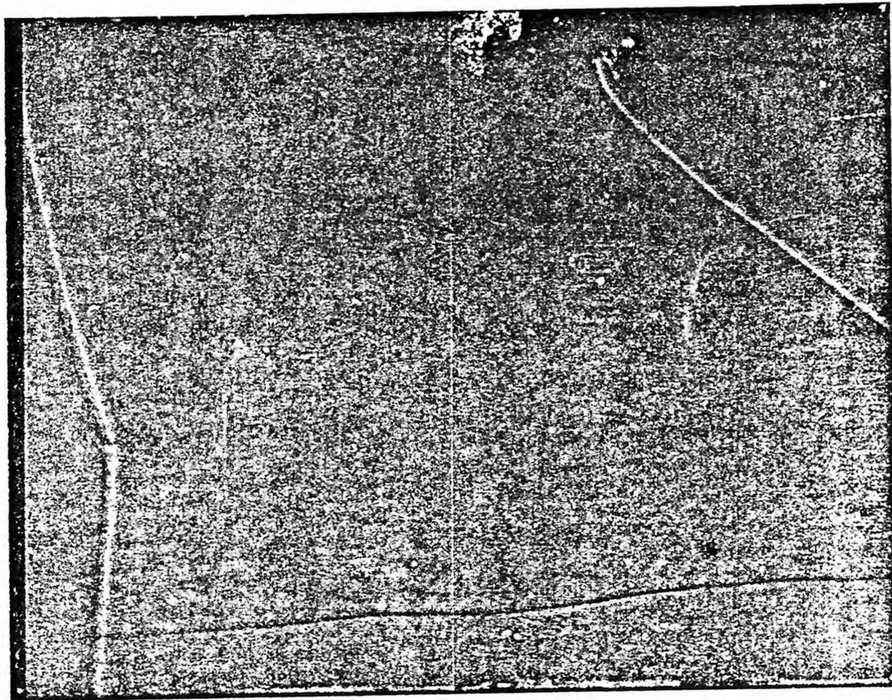

Figure 5.10: SiC film on fused quartz wafer magnified to 110 times.

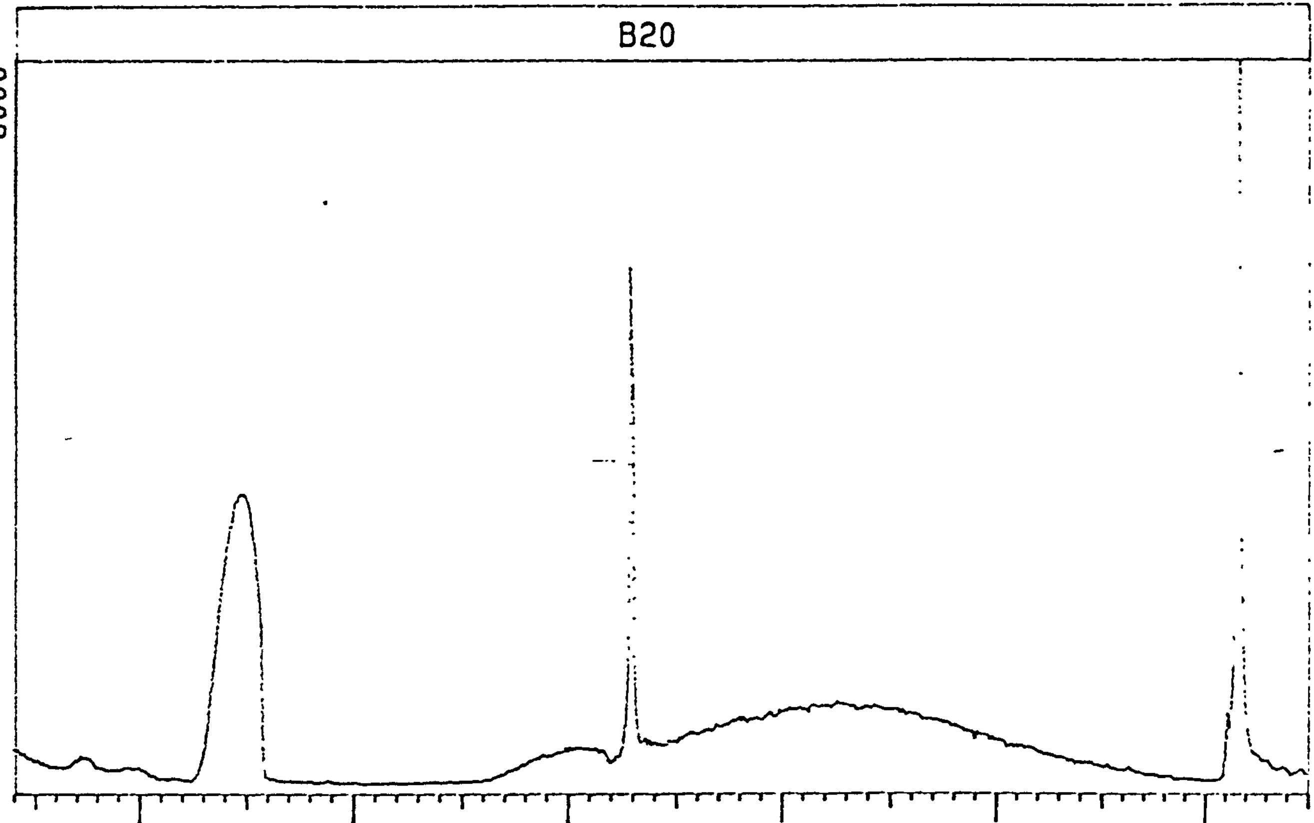

Figure 5.7: X-diffraction pattern shows the amorphous nature of the film.

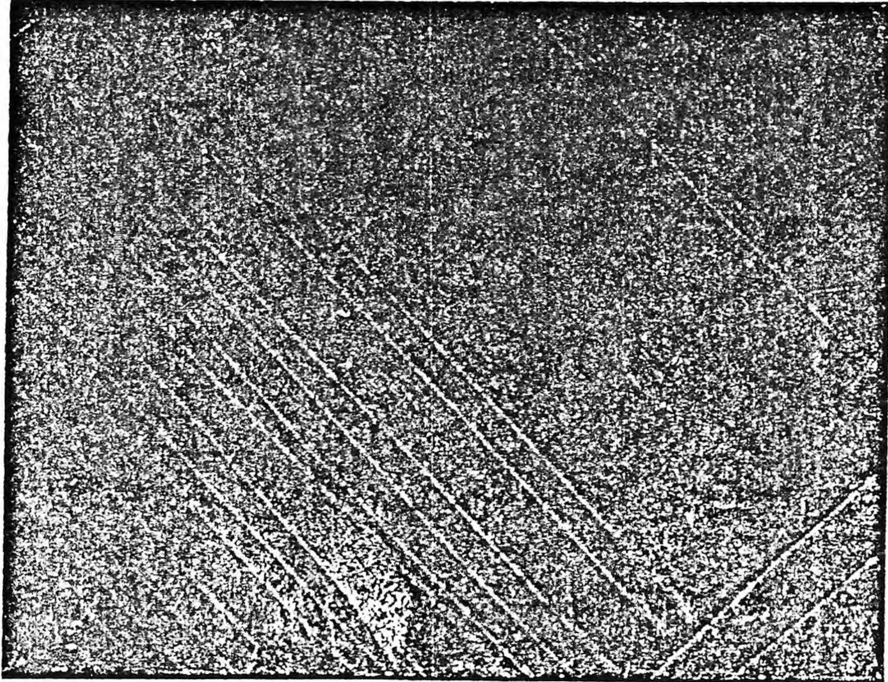

Figure 5.8: Microscopic view of SiC film on Si < 100 > substrate magnified to 110 times showing the presence of microcracks.

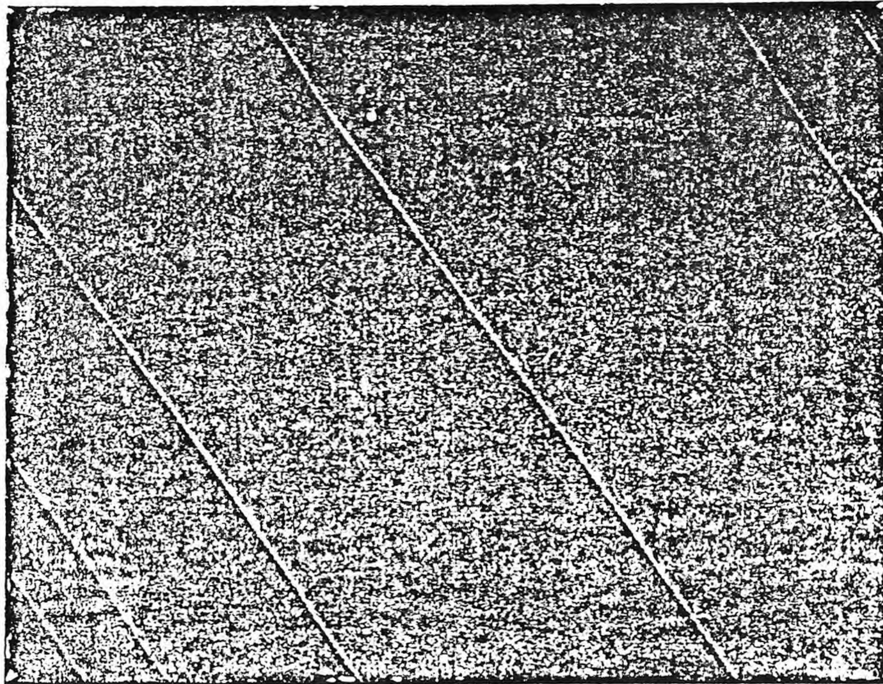

Figure 5.9: SiC film on Si < 111 > substrate depicting microcracks.

Chapter 6

CONCLUSION

6.1 Concluding Remarks

A suitable material for x-ray mask membrane fabrication was investigated. The properties of silicon carbide justify the choice of this material for the membrane preparation. Of various methods available for film preparation, low pressure chemical deposition method was found to be suitable. Amorphous SiC films were synthesized by this method, using a liquid precursor, Ditertiary butyl silane. Films deposited on silicon and quartz substrates were subjected to various characterization techniques. Temperature was found to be an important factor for the film stoichiometry. The kinetic growth regime was found to be from $600°C to 700°C$. Infra-red spectroscopic analysis confirmed the presence of Si-C vibrational mode. X-ray diffraction studies confirmed the amorphous nature of the films. In all cases, the films were found to be tensile as evidenced by presence of micro-cracks.

6.2 Further Scope Of This Study

This study can be extended further for the investigation of formation of micro-cracks and methods to avoid them. Deposition of these films on different substrates like sapphire and on silicon substrates with other orientation may be significant. Other properties of the film like refractive index, stress measurement, optical and x-ray transparency can be studied. The knowledge of above mentioned properties may lead to successful commercial fabrication of x-ray mask.

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