Characterizing Etched Grating Profiles With The Alpha-particle Energy Loss Method

by

Christina Kaiser

B.Sc., University of British Columbia, 1999

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Master of Science

in

THE FACULTY OF GRADUATE STUDIES

(Department of Physics and Astronomy)

We accept this thesis as conforming to the required standard

THE UNIVERSITY OF BRITISH COLUMBIA

May 2001

© Christina Kaiser, 2001

In presenting this thesis in partial fulfilment of the requirements for an advanced degree at the University of British Columbia, I agree that the Library shall make it freely available for reference and study. I further agree that permission for extensive copying of this thesis for scholarly purposes may be granted by the head of my department or by his or her representatives. It is understood that copying or publication of this thesis for financial gain shall not be allowed without my written permission.

Department of Physics and Astronamy

The University of British Columbia Vancouver, Canada

Date April 25, 2001

Abstract

The effectiveness of Alpha-particle Energy Loss (AEL) spectroscopy for profiling 1D grating structures was investigated. An analytic simulation of AEL spectra was developed and compared with a Monte Carlo simulation. By a least squares fitting procedure, the analytic simulation was used to extract grating parameters from the experimental spectra of two types of grating structures. A 1D grating of silicon bars on a free standing silicon dioxide support layer was fabricated and profiled by AEL spectroscopy: the grating depth was determined along with a qualitative assessment of the grating sidewalls. AEL profiling of a gallium arsenide grating structure yielded measurement values in agreement with those obtained with a scanning electron microscope.

Contents

. . .

\mathbf{Abstra}	ct	ii
List of	Tables	v
List of	Figures	vi
Chapte	er 1 Introduction	1
Chapte	er 2 Theory : Charged Particle Energy Loss	5
2.1	Nuclear scattering	6
2.2	Electron scattering	6
2.3	Fluctuations	9
Chapte	er 3 Simulation	11
3.1	Simple scenario	11
3.2	Realistic simulations	13
	3.2.1 Monte Carlo simulation	14
	3.2.2 Analytic simulation	14
3.3	Analytic simulation: defining particle trajectories	18
Chapte	er 4 Experimental Procedure and Setup	24
4.1	Setup for AEL data collection	24
4.2	Gallium Arsenide grating structure	26
4.3	Textured membrane fabrication	28

	4.3.1	SOI wafer	28
	4.3.2	Wafer preparation	31
	4.3.3	Thermal oxidation	31
	4.3.4	Patterning the oxide	34
	4.3.5	Anisotropic etch of the bulk silicon	35
	4.3.6	Isotropic etch to deepen SiO_2 grating $\ldots \ldots \ldots \ldots \ldots \ldots$	40
	4.3.7	Transfering the grating pattern to the silicon thin film \ldots .	41
4.4	Implai	ntation	42
	4.4.1	Implantation chamber	43
	4.4.2	Implantated activity	44
	4.4.3	The alpha particle spectrum	46
Chapte	er 5 I	Data	48
5.1	Silicon	$ membrane \ structure . \ . \ . \ . \ . \ . \ . \ . \ . \ .$	48
5.2	GaAs	ridges	54
	5.2.1	Shallow grating depth	54
	5.2.2	Intermediate grating depth	55
	5.2.3	Deep gratings	57
Chapte	er 6 S	ummary and Conclusions	60
3iblio _{	graphy		62

List of Tables

-

,

4.1	Process parameters for the etching of the GaAs grating masked in oxide	
	(sccm \equiv standard cubic centimeters per minute)	27
4.2	RCA clean recipe. Each 10 minute bath was immediately followed by	
	a 5 minute rinse of the wafer in distilled water \ldots	31
4.3	Density and Youngs modulus for quartz and themal oxides. Where	
	Young's modulus values are given for the films the thickness of the	
	thermal oxide film is specified. \ldots \ldots \ldots \ldots \ldots \ldots	32
4.4	Gas flow rates and stage duration for the three stage oxidation process	
	used to form a 420 nm thin film on the surface of the SOI wafer at	
	$1100^{\circ} \mathrm{C}$	33

List of Figures

2.1	Depiction of the scenario for deriving Bohr's classical energy loss cal-	
	culation.	7
3.1	Idealized profile of a 1D grating structure with period A. \ldots .	11
3.2	Idealized alpha particle energy spectrum for a parallel beam of alpha	
	particles that pass through a 1D grating structure at normal incidence.	
	The source energy is centered at energy E_0	12
3.3	Energy dispersion due to the presence of a collimator. The height of	
	the peaks is approximately 600 000 counts.	16
3.4	Parameters descriptive of alpha particle trajectories	18
3.5	A beam of alpha particles at angle θ and arbitrary angle ϕ is incident	
	on a cylindrical aperture of the collimator. The collimator thickness is	
	${\bf t}$ and the aperture radius is r. The "effective" aperture, illustrated as	
	region Σ , is a geometric function of t, r and θ	19
3.6	Cross-sectional view of the grating and alpha particle trajectories that	
	pass though the grating surface at points where the surface slope is	
	discontinuous. The trajectories are indicated by dotted lines. Note	
	that for $\phi \neq \frac{\pi}{2}$, the trajectories and the labeled distances, t_{A1} and	
	t_{A2} , are not confined to the cross-sectional plane	20
3.7	Normalized analytic and Monte Carlo spectra for three surface geometries	22

3.8	Monte Carlo data and analytic data, with and without 20 keV detector	
	resolution broadening. In the Monte Carlo simulation the acceptance	
	angle cutoff of the collimator was 26.5 °. For the analytic data, normal	
	incidence was assumed. Δ is the difference between the Monte Carlo	
	and the simulated data.	23
4.1	Schematic drawing of the experimental setup used for AEL imaging of	
	the GaAs and membrane grating structures.	25
4.2	Structure of the gallium arsenide sample	26
4.3	Membrane fabrication	29
4.4	Fabrication of SOI bonded wafers	30
4.5	Microscope image of the silicon bars at an edge of the patterned region.	
	Also, schematic representation of the bars from three points of view.	36
4.6	Arrangement of the inverted funnel and thermometer with the beaker	
	that held the sample and the EDP solution $\ldots \ldots \ldots \ldots \ldots \ldots$	38
4.7	Schematic drawing of the SOI sample in the teflon holder. The teflon	
	holder does not extend across the base of the sample so that the upward	
	flow of the hot etchant over the etch region is not obstructed. \ldots .	39
4.8	A free standing SiO_2 thin film, 420 nm thick $\ldots \ldots \ldots \ldots \ldots \ldots$	40
4.9	A free standing SOI membrane grating structure	42
4.10	View of the implantation chamber as seen from above with both source	
	holders extended. The aperture plate in the upper left corner of the	
	image has a hole diameter of 7 mm	44
4.11	Schematic side view of the implantation chamber with the Thorium	
	source extended and a wafer in place for implantation	45
4.12	Alpha particle spectra of an implanted silicon wafer before and after	
	the growth of 490 nm of Al. The greater height of the "pre-deposition"	
	spectra is due to the longer collection time associated with it. \ldots	47
5.1	Grating viewed with an optical microscope at 5X magnification	49

5.2	Underside of grating viewed at 5 X magnification	50
5.3	Cross-sectional view of the membrane grating overlaid with a sketch of	
	the simulation profile that best fit the spectral data	51
5.4	AEL spectrum (discrete points) for the membrane structure of Fig. 5.3	
	with best fit simulation (continuous line)	52
5.5	As in Fig. 5.4 except that the best fit simulation was found under the	
	constraint that the grating sidewalls were perfectly vertical. \ldots .	52
5.6	Simulation for effective etched depths of 100, 115 and 130 nm shown	
	with GaAs grating spectral data. Δ is difference between the actual	
	and simulated data. The Gaussian linewidth convolved with the sim-	
	ulated data is 44 keV FWHM	55
5.7	Image of GaAs grating etched to 415 nm depth with the profile of the	
	best fit simulation superimposed	56
5.8	Best fit simulation with GaAs grating spectral data for an etch depth	
	of 415nm	56
5.9	Image of GaAs grating at an etch depth of 765 nm. The sidewall	
	angle of the simulated profile superimposed on the grating image is	
	approximately 8 degrees.	57
5.10	Simulation for sloped walls with GaAs grating data	58
5.11	Simulation for vertical sidewalls as compared to the experimental spec-	
	trum for the deep GaAs grating. Clearly, the region between the peaks	
	is not fit well when the sidewalls are assumed to be vertical.	59

viii

. . .

Chapter 1

Introduction

The alpha particles emitted by ^{228}Th and its radioactive daughters are sufficiently energetic that they can travel though several microns of most thin films and emerge with a non zero velocity. The particles can then be detected, their energy measured and the energy lost in the film determined. If the constituents of the film are adequately characterized, the film thickness can be calculated from the alpha particle energy loss. Conversely, if the thickness of the film is known, the energy lost by the alpha particle gives a measure of the film's constituent properties. The basis of this characterization is the formal relation derived in the 1930s by Hans Bethe (1) and Felix Bloch (2). The Bethe-Bloch formula, discussed in the Chapter 2, relates a material's properties to the energy loss rate of a charged particle traveling through it.

Spectroscopy by alpha particle energy loss (AEL) has been applied as an in-situ thickness monitor for thin film growth (3), as a means to determine the stoichiometry of thin films (4), for the mapping of non-planar surfaces (5) and as an in-situ etch rate monitor for the fabrication of 1D gratings in thin films of gallium arsenide (6).

CHAPTER 1. INTRODUCTION

Another film thickness monitoring technique, ellipsometry, involves the reflection of a polarized beam of light off the surface of the thin film to be etched. The change in the polarization angle and intensity of the beam upon reflection can be used to determine the film thickness and refractive index[(7) p. 79]. Interference of a variable source of monochromatic light at near normal incidence can also be used to determine film thickness. As the source wavelength is scanned, the interference of the incoming and outgoing beams will repeatedly fall in and out of phase. The smallest wavelength interval, $\Delta\lambda$, separating two maxima or two minima in the reflected light intensity is related to the film thickness t_f by $t_f = \frac{\Delta\lambda}{2n}$, where n is the refractive index of the film (*Ibid*).

A limitation of both ellipsometry and thin film interference is that they cannot be used to monitor the thickness of a film grown on substrates of the same composition as the film; they require that the film and its substrate be made of optically contrasting materials. Conversely, AEL has been successfully applied to monitor the thickness of gallium arsenide (GaAs) thin films grown on GaAs substrates, both during growth and etching of the thin films[(3), (6)]. Reference (6) also describes the more complicated, but typical scenario in which the surface of an etched film has been masked, and an etch monitor is needed to determine not only the etch rate and depth, but also the profile of the etch at the mask edges.

Etching is the critical stage in many electronic and opto-electronic device fabrication processes. Tolerences of device specifications are typically < 1 percent which, in the sub micron regime, is on the order of a few nanometers. There is an ongoing need to develop new, more accurate in-situ techniques for characterizing the extent and quality of the etching. In this thesis, we evaluate alpha particle spectroscopy as a means for determining the *profile* of etched 1D gratings. The extraction of geometric grating parameters from AEL spectra is achieved with the aid of a simulation that calculates alpha particle spectra, using models of 1D gratings of arbitrary depth and duty cycle. The slope of the grating sidewalls, being a variable parameter in the simulation, can be determined from a least squares fit of the actual alpha particle transmission spectra and the simulation. Chapter 3 is devoted to a discussion of the simulation.

Application of the AEL method requires that a source of energetic alpha particles is placed beneath the grating structure to be imaged. The source is generated by the implantation of alpha emitting isotopes into the surface of a semiconductor wafer by exposure of the wafer surface to a source of ²²⁸Th for 2-10 days. Details of the implantation process appear in Section 4.4

AEL spectra of the aforementioned gallium arsenide (GaAs) grating are presented in Section 5.2. The GaAs grating was formed by plasma etching of a GaAs thin film masked with a 1D oxide grating. The GaAs grating spectra were collected as the plasma etch was monitored by AEL *in-situ*. Here, simulated data are compared to the GaAs grating data at three different etch depths. Measurements of the grating height made by the AEL method agree with SEM measurements in each case. The accuracy of the SEM and AEL measurements is comparable. Also in Section 5.2 the utility of the simulation for detecting non-vertical sidewalls is assessed. Fabrication of the GaAs grating structure is discussed in Section 4.2.

In Section 5.1, the AEL spectra of a free standing membrane structure is pre-

sented. The membrane structure, consisting of silicon bars supported by a uniform oxide thin film, was fabricated from a Silicon-on-Insulator (SOI) wafer. The anisotropic etchant used to generate the silicon bars from the (100) thin film of the SOI wafer's polished surface, proceeded preferentially in the [100] direction and formed the bars with a sidewall angle of $\sim 37^{\circ}$. Comparison of real and simulated data for the silicon structure was used to make quantitative measurement of the grating sidewall angle. The AEL measurement of the sidewall angle was 10° greater than the SEM measurement. Overestimation of the slope by the AEL method demonstrates the method's sensitivity to sample quality and uniformity.

The means by which the silicon membrane structure was fabricated is discussed in Section 4.3. The experimental setup for data collection is presented in Section 4.1.

Chapter 2

Theory : Charged Particle Energy Loss

A charged particle moving through matter loses energy by two principal mechanisms: it interacts by the Coulomb force with the material's electrons and nuclei, or, if highly energetic, radiates photons. For the alpha particles used in the AEL work reported here, radiation losses may be ignored.

When a particle is near the end of its range in a material, nuclear scattering may be significant in deflecting the particle from a straight-line path. However, the alpha particles that we use for thin film spectroscopy emerge from the film with several MeV energy, so they rarely experience nuclear scattering; rather, they are influenced primarily by electronic interactions. In Section 2.2 we outline the derivation of the Bethe Bloch formula for a charged particle's energy loss rate by electronic scattering, and in Section 2.3 we address the issue of fluctuations in the energy loss of a charged particle in matter. These fluctuations, due to the statistical nature of the Coulomb scattering process, set the intrinsic resolution of the AEL method.

2.1 Nuclear scattering

The energy of a charged particle determines whether electronic or nuclear interactions occur most frequently. Because of screening by bound electrons, the nuclear potential is not strongly felt by a passing charge unless the charge moves slowly relative to the orbital velocity of the bound electrons. Formally, the nuclear scattering cross section σ , for a particle of charge q and velocity v, is as follows [(8)p.643]:

$$\sigma = \pi (R q Q/\hbar v)^2 \tag{2.1}$$

where Q is the nuclear charge and R is the nuclear radius. The nuclear radius can be approximated by $1.4 \text{ A}^{1/3} \times 10^{-15} \text{m}$, where A is the nuclear mass number.

An alpha particle with 8 MeV kinetic energy moves at approximately 0.065 times the speed of light. In GaAs, $\sigma \approx 2.2 \ x \ 10^{-26} \text{m}^2$. For the same particle in silicon, $\sigma \approx 2.3 \ x \ 10^{-27} \text{m}^2$. The lattice parameter for GaAs or silicon is of order 10^{-10} m; the cross-sectional area of either crystal's unit cell is more than 8 orders of magnitude larger than its nuclear scattering cross section. In a material of thickness 1 micron, or 10^4 monolayers, nuclear scattering will affect approximately one particle in 10^4 .

2.2 Electron scattering

Typically, an energetic particle in matter loses a few eV/Å due to electron scattering. For particles much more massive than an electron, no significant deflection of the particle from its original trajectory occurs by electron scattering and the particle moves in an essentially straight-line path. The rate of energy loss of a charged particle in matter has been derived by Hans Bethe (1) and F. Bloch (2). In the classical limit, the Bethe-Bloch formula approaches an expression derived previously by Niels Bohr (9, 8).

We can arrive at Bohr's expression by considering firstly the energy imparted to a stationary electron by the impulsive Coulomb force of a passing charge. In Fig 2.1, an electron of charge e and mass m is situated a vertical distance b away from the path of a particle of charge Ze, mass M and velocity v that moves a horizontal distance dx in time v dt.



Figure 2.1: Depiction of the scenario for deriving Bohr's classical energy loss calculation.

By Gauss's Law, the traveling particle's normal field component \mathbf{E}_{\perp} integrated over a cylinder of radius b, and length dx =v dt, must satisfy:

$$\int E_{\perp} dx = Z e / (2\pi b \epsilon_o)$$

The impulse Δp imparted to the electron in this time, dt = dx/v, is given by:

$$\Delta p = e/v \int E_{\perp} dx = Z e^2 / (2\pi \epsilon_o bv)$$

The particle velocity is taken outside of the integral on the assumption that it is very

nearly constant, the energy imparted to the electron during a single collision being negligibly small. The energy imparted to the electron is:

$$\Delta E = \frac{\Delta p^2}{2m_e} = \left(\frac{e^2}{4\pi\epsilon_o}\right)^2 \frac{2Z^2}{m_e b^2 v^2}$$
(2.2)

In volume $dV = 2\pi b \, db \, dx$ that contains a number density of electrons, N_e , the scattering particle's incremental energy loss rate, $\frac{dE(b)}{dx}$ is

$$\frac{dE(b)}{dx} = \Delta E N_e dV = \left(\frac{e^2}{4\pi\epsilon_o}\right)^2 \frac{4\pi Z^2}{m_e v^2} N_e \frac{db}{b}$$
(2.3)

If Eqn. 2.3 is integrated with respect to the scattering parameter, b, between the integration limits, b_{min} and b_{max} (yet to be determined), the resulting expression is

$$\frac{dE}{dx} = \left(\frac{e^2}{4\pi\epsilon_o}\right)^2 \frac{4\pi Z^2}{m_e v^2} N_e ln \frac{b_{max}}{b_{min}}$$
(2.4)

The condition for adiabatic interaction of an atomic system with a time dependent potential determines b_{max} . If the potential varies slowly relative to the frequency of the orbiting electrons, the interaction will be elastic. For energetic exchange over the interaction time, $t \approx b/v$, we require that $b_{max} = v/\overline{\nu}$, where $\overline{\nu}$ is a mean frequency defined over the bound electron states.

The maximum energy that can be transferred to an electron occurs in a head on collision with the scattered particle. In this case, the electron's kinetic energy is $\frac{1}{2}m_e(2v)^2$. This maximal energy transfer can be substituted into the left hand side of Eqn. 2.2 to give for the impact parameter at its minimum value: $b_{min} = \frac{Ze^2}{4\pi\epsilon_o m_e v^2}$ Finally, we have Bohr's classical energy loss rate :

$$\frac{dE}{dx} = (\frac{e^2}{4\pi\epsilon_o})^2 \frac{4\pi Z^2}{m_e v^2} N_e ln(\frac{2m_e v^2}{I})$$
(2.5)

where $I = \eta \hbar \overline{\nu}$ and $\eta = \frac{Ze^2}{2\pi\epsilon_o \hbar v}$, a dimensionless parameter.

Calculation of $\overline{\nu}$ involves the summation over all electronic states of the state frequency weighted by the oscillator strength of the state. Because the oscillator strengths of electronic states are difficult to evaluate, and because the formula is only approximate, the value of I is most frequently obtained empirically. It is well approximated for most materials by $I = 16 Z^{0.9} eV$ (10)

2.3 Fluctuations

The energy loss rate in Eqn. 2.5 is an average energy loss per unit distance that results as the charged particle undergoes a large, yet finite, number of electronic collisions. The amount of energy transferred in a single collision is variable - so that the actual energy loss rate for a particle fluctuates about the average value of our calculation. We know by the central limit theorem that if the number of collisions is very large and the energy transfer of a single collision is not too great, then for a path of length l, the dispersion in the energy loss, known as energy straggling, is a Gaussian distribution about the mean energy loss over the distance l. If the particle is not ultra-relativistic, the variance, σ_S^2 , of the straggling is proportional to l, and is given, in Gaussian units, by [(8), p.631]:

$$\sigma_S{}^2 = 2\pi N_e Z^2 e^4 (\gamma^2 + 1)l \tag{2.6}$$

where $\gamma = (1 - \frac{v^2}{c^2})^{-\frac{1}{2}}$. For MeV energy alpha particles, $(\gamma \approx 1)$. The variance due to straggling is 166 $(keV)^2$ /micron in gallium arsenide and 70 $(keV)^2$ / micron in silicon. For a detector resolution of variance σ_D^2 , the overall variance associated

with the AEL resolution is:

$$\sigma^2 = \sigma_D^2 + \sigma_S^2 \tag{2.7}$$

As an example of the effects of straggling, for a detector resolution width of 35 keV FWHM (typical of our setup), the additional broadening due to straggling is 11 keV in a 1 μ m film of gallium arsenide, and 5 keV in a 1 μ m film of silicon.

Chapter 3

Simulation

3.1 Simple scenario

In an idealized application of AEL for imaging gratings, a parallel, spatially uniform beam of alpha particles passes though the grating at normal incidence. A schematic representation of the grating appears in Fig. 3.1, where all the segments are assumed to be straight lines. For a grating of given period A, there are altogether 4 independent parameters which characterize the cross section: the thickness of the base, h_1 , the height of the ridge, h_2 and the widths of the ridge at its bottom and at its top, b_1 and b_2 respectively.



Figure 3.1: Idealized profile of a 1D grating structure with period A.

To illustrate the basic concept of AEL as a means of evaluating grating profiles,

we start by assuming that the amount of energy lost by an alpha particle is proportional to the length of its trajectory in the grating material. Each spatial element of the grating gives rise to a specific alpha-energy according to its thickness, resulting in a well defined alpha-energy spectrum.

The typical idealized alpha spectrum corresponding to this scheme is shown in Fig. 3.2, where two sharp peaks are joined by a continuum of counts stretched between them. The energy shift of the upper peak relative to the original alpha-energy is proportional to h_1 . The energy difference between the two peaks is proportional to h_2 . The fraction of counts in the higher peak, in the lower peak and in the intermediate region are equal to $(A - b_1)/A$, b_2/A and $(b_1-b_2)/A$ respectively.



Figure 3.2: Idealized alpha particle energy spectrum for a parallel beam of alpha particles that pass through a 1D grating structure at normal incidence. The source energy is centered at energy E_0 .

In reality, of course, the situation is not quite so straightforward, since the observed alpha-particle energy loss is not strictly proportional to the path length, and because the alpha particles are not collimated as they enter the substrate. To extract the observed geometric parameters from AEL spectra, one therefore has to incorporate Eqn. 2.5 in a simulation that includes a distribution of alpha particle trajectories at various angles with respect to the selected normal. Sources of dispersion must also be accounted for: for each given path length there is a dispersion of energy loss values due to straggling as in Eqn. 2.6, and further dispersion due to finite energy loss in a collimator on the face of the detector; finally, for any given energy loss the actual recorded value will be modified by the intrinsic resolution of the detector (about 35 keV in our case).

3.2 Realistic simulations

Two simulations of alpha particle energy loss in 1D gratings were developed in this work. Both rely on a specified set of initial alpha particle energies and trajectories as they enter the grating region, and a specified angular acceptance function for the detector. One model assumes straight line trajectories while the other simulates the particle motion through the sample using GEANT¹, a Monte Carlo routine developed at CERN. Both simulations treat straggling and detector resolution as uncorrelated sources of dispersion, and use the straggling parameter given by Eqn. 2.6.

 $^{^{1}}$ available online : CERNLIB@CERNVM.CERN.CH

3.2.1 Monte Carlo simulation

The Monte Carlo simulation generates the spectrum of alpha particles passing through a grating structure, by firstly encoding the geometric arrangement of the grating, the detector, the collimator and the region from where the alpha particles originate. The alpha particle trajectories are then constructed. The alpha particle starts out in a random direction from the implanted region with a momentum determined by the decay energy of the parent nucleus. The particle's path is broken up into distance intervals of length determined by the particle's momentum and a specified timeinterval. At the end of each time interval, the particle's position and energy are recorded. The direction and energetic events associated with the next time interval are determined by the probabilities that govern the interactions of the particle with its host material. The energy loss associated with each step is calculated by 2.5. Straggling effects in the Geant routine can be incorporated or neglected by appropriate declaration of "flags" at runtime. Monte Carlo simulations without straggling were used to test the analytic simulation.

3.2.2 Analytic simulation

In the analytic simulation, a straightline trajectory is assumed for all alpha particles. The energy loss for each particle is assumed to be proportional to the material thickness that the particle passes through. The constant of proportionality, $\overline{dE/dx}$, is an average energy loss rate, calculated as follows:

The most energetic alpha particle to reach the detector from beneath the grat-

ing structure of Fig. 3.1 will pass through the grating trough at normal incidence. Let the energy of this particle be E_1 . The least energetic particle to reach the detector will pass through the grating step at the maximum angle allowed by a collimator that is directly in front of the detector, as in Fig. 4.1. Let this minimum energy be E_2 . Let dE/dx(E) be the energy loss rate of Eqn. 2.5. Finally,

$$\overline{dE/dx} = \frac{1}{2} \{ dE/dx(E_1) + dE/dx(E_2) \}$$

The detected energy, E, associated with an alpha particle that travels an arbitrary distance L in the grating is:

$$E = E_1 - \overline{dE/dx}(L - h_1)$$
, for h_1 as in Fig. 3.1.

In the analytic simulation it is assumed that any particle that hits the collimator material does not reach the detector. In reality, detected particles may have passed though a finite region of the collimator. To assess the significance of energy lost in the collimator, the Monte Carlo simulation was run for a planar source of alpha particles incident on a lucite wafer which was punctured with a trapezoidal lattice of cylindrical holes to a filling fraction of 40 percent. The holes had a 5:1 height: diameter ratio, appropriate to the collimator used for AEL data collection in Section 5.1. The simulated alpha particles were incident on the collimator at random angles of up to 90 degrees with energies occurring with equal probabilities at 8.6, 8.7, 8.8 and 8.9 MeV. The result of running the Monte Carlo simulation for 10 million events appears in Fig. 3.3.

The presence of the collimator has resulted in a uniform background to the left of each source peak. The height of the background in a region is proportional to the number of peaks to the right of the region. Because the background is featureless it



Figure 3.3: Energy dispersion due to the presence of a collimator. The height of the peaks is approximately 600 000 counts.

will not influence the spacing and height of the peaks. Also, the integrated background in the imaged region constitutes less than 0.5 percent of the spectrum. Reasonably, then, dispersion in the collimator can be neglected in our full simulations.

The height: diameter ratio of the holes that perforate the collimator determines the acceptance angle for particles that reach the detector. Two collimators were used for our experimental work. For imaging of the silicon grating structure, Section 5.1, the acceptance angle was $\pm 11.3^{\circ}$ off normal incidence. For the GaAs grating structure, Section 5.2, an acceptance angle of $\pm 26.5^{\circ}$ off normal incidence applied.

The analytic simulation divides the angular range of the collimator into a discrete set of angles spaced at equal solid angle increments, and for each angle in the set calculates the spectral contribution of particles that approach the detector at that angle. In this way, dispersion due to finite angular range of incidence is accounted for.

What follows in the remainder of this chapter is a description of how the spectrum for a given angle of incidence is constructed.

¢

3.3 Analytic simulation: defining particle trajectories

The length of a particle's path in the grating material depends on three variables:

- θ : trajectory angle off the surface normal
- ϕ : trajectory angle off the long direction of the grating
- X : horizontal position of the trajectory origin from an arbitrarily chosen fixed point on the grating

An alpha particle trajectory is illustrated in Figure 3.4.



Figure 3.4: Parameters descriptive of alpha particle trajectories

The simulation proceeds by calculating the energy loss for a discrete set of angular orientations, each specified by a (θ, ϕ) pair. The angular orientations are chosen so that each is representative of the same steradian interval.

Formally, let there be N values of θ : $\{\theta_j\}$, for $j \in \{1,2...N\}$, and M values of ϕ : $\{\phi_k\}$ for $k \in \{1,2...M\}$. If the number of counts in the ith spectral bin are C_i then the spectrum is the set $\{C_i\}$. For each (θ, ϕ) pair, one has a "sub-spectrum" $\{C_i(\theta_j, \phi_k,)\}$. The total spectrum is the sum of the sub-spectra over angular orientations:

$$\{Ci\} = \sum_{j} f(\theta_j) \sum_{k} C_i(\theta_j, \phi_k)$$
(3.1)

where $f(\theta)$, independent of ϕ , is a scaling factor determined by the collimator's effective aperture, Σ . The dependance of Σ on θ and the collimator geometry is illustrated in Fig. 3.5. For an aperture cross-section, $\Sigma_o = \pi r^2$, $f(\theta) = \Sigma / \Sigma_o$



Figure 3.5: A beam of alpha particles at angle θ and arbitrary angle ϕ is incident on a cylindrical aperture of the collimator. The collimator thickness is t and the aperture radius is r. The "effective" aperture, illustrated as region Σ , is a geometric function of t, r and θ .



Figure 3.6: Cross-sectional view of the grating and alpha particle trajectories that pass though the grating surface at points where the surface slope is discontinuous. The trajectories are indicated by dotted lines. Note that for $\phi \neq \frac{\pi}{2}$, the trajectories and the labeled distances, t_{A1} and t_{A2} , are not confined to the cross-sectional plane

In Fig. 3.2 the shape of $\{C_i(0, \frac{\pi}{2})\}$ is shown for the type of grating appearing in Fig. 3.1. Now we consider the construction of an arbitrary $\{C_i(\theta_j, \phi_k,)\}$.

The slope of the grating surface is discontinuous at four points. In Fig. 3.6 the points of surface discontinuity are labeled {A,B,C,D}. Also shown in Fig. 3.6 are the trajectories for a fixed angular orientation that pass through the discontinuities, and the horizontal distance separating them, illustrated as {L_{AB},L_{BC},L_{CD},L_{DA}}. It is important to note that the trajectories are not confined to the cross-sectional plane for $\phi \neq \frac{\pi}{2}$. The trajectory that passes through the discontinuity A, passes through the grating material over the distances t_{A1} and t_{A2}. Let {T_A,T_B,T_C,T_D}, be the total grating material traversed by the trajectories (i.e. T_A = t_{A1} + t_{A2}).

The material thickness that a trajectory passes through, determines the energy loss, and hence the detected energy associated with it. Let $\{E_A, E_B, E_C, E_D\}$ be the detected alpha energies that correspond to $\{T_A, T_B, T_C, T_D\}$. Given E_A and E_B as above, let ΔE_{AB} be the absolute difference between E_A and E_B , and let a similar definition define all members of set: { ΔE_{AB} , $\Delta E_{BC} \Delta E_{CD} \Delta E_{DA}$ }

In "pseudo-code" the algorithm for constructing the sub-spectrum $\{C_i(\theta_j, \phi_k,)\}$ is as follows:

Let BW = the channel width of the pulse height analyser²

Let A = grating period

For each value of $n \in \{1,2,3,4\}$,

Let ENERGY_INTERVAL = the nth element of { ΔE_{AB} , $\Delta E_{BC} \Delta E_{CD} \Delta E_{DA}$ } Let SPATIAL_INTERVAL = the nth element of {L_{AB}, L_{BC}, L_{CD}, L_{DA}};

If the energy of the ith spectral bin is in ENERGY_INTERVAL $C_i(\theta_j, \phi_k,) = \frac{\text{SPATIAL_INTERVAL}}{\text{ENERGY_INTERVAL}} \frac{\text{BW}}{\text{A}}$

Spectra generated by the incorporation of this algorithm in Eqn. 3.1 appears with spectra generated by the Monte Carlo simulation in Fig. 3.7 for three grating geometries. The good agreement of the spectra with regard to the peak separation shows that the use of the average energy loss and straightline trajectories in the analytic simulation is justified. Agreement with respect to the peak height and width demonstrates that the geometric functions representing the grating and collimator in the analytic simulation capture the relevant features of these structures. The shape of the peaks is determined by the effective aperture of the collimator (see Fig. 3.5). Dispersion due to detector resolution and straggling was not incorporated in these simulations.

²equivalently, BW = the width of each energy bin in the AEL spectrum



Figure 3.7: Normalized analytic and Monte Carlo spectra for three surface geometries

When spectra are convolved with the straggling and the detector response functions, much of the detail in them is lost. In Fig. 3.8, the Monte Carlo data of the previous figure's middle plot is shown again, along with an analytic simulation that assumes normal incidence for all alpha particles on the same grating structure. Though the unbroadened spectra of Fig. 3.8 are significantly different, distinction between them is virtually lost when they are convolved with a 20keV FWHM Gaussian.



Figure 3.8: Monte Carlo data and analytic data, with and without 20 keV detector resolution broadening. In the Monte Carlo simulation the acceptance angle cutoff of the collimator was 26.5 °. For the analytic data, normal incidence was assumed. Δ is the difference between the Monte Carlo and the simulated data.

Relatively rapid generation of the simulated data greatly facilitates the least squares fitting procedure used to extract grating parameters from the AEL spectra. Since broadening due to straggling and detector response is at least 40 keV in all of our experimental scenarios, we can can optimize the time efficiency of our simulations by assuming that the alpha particle trajectories are perfectly vertical, without introducing significant errors in the least squares fitting procedure. Normal incidence of the alpha particles was assumed for the best fit simulations shown in Chapter 5.

Chapter 4

Experimental Procedure and Setup

Two grating structures were fabricated for this work: one was a dry etched GaAs thin film on a GaAs substrate (Section 4.2), the other was a wet etched silicon thin film on a free standing membrane of silicon dioxide (Section 4.3). An alpha source was generated for the imaging of each grating by the alpha particle implantation process described in Section 4.4. Spectral data for each structure was collected in an evacuated etch chamber to which a solid state detector was retro-fit as an in situ etch monitor.

4.1 Setup for AEL data collection

A schematic representation of the data collection setup is shown in Fig. 4.1.

The detector was an EG&G ORTEC Ion Implanted Silicon charged particle detector with a specified resolution of 20 keV. A DC bias of 70 V was applied across the detector with an ORTEC 459 DC power supply. The detector response passed into an ORTEC 142-A Preamplifier, and then to a TENNELEC TC 241 Amplifier from where it was input to an APTEC MCArd Multi Channel Analyzer. A collimator consisting of a thin disk of polymerized material("Vespel", by Dupont), perforated with a trapezoidal lattice of 0.5 mm diameter cylindrical holes to a filling fraction of 40 % was installed directly over the active region of the detector . The collimator thickness was 2.5mm for the data collection of Section 5.1 and 1mm for the data collection of Section 5.2. The detector is shown in Fig 4.1 in its extended position over the sample. When a sample was introduced to, or removed from the chamber, or when a sample was being etched, the detector was withdrawn from the chamber and sealed under vacuum behind a gate valve.



Figure 4.1: Schematic drawing of the experimental setup used for AEL imaging of the GaAs and membrane grating structures.

4.2 Gallium Arsenide grating structure

Fabrication of the gallium arsenide gratings and collection of the associated AEL data that is presented with simulations below, was the work of Yuval Levy (6). Grating fabrication began with the implantation of alpha sources in a polished 2" GaAs quarter wafer by the procedure described in Section 4.4. Immediately after implantation, a GaAs thin film, 850 nm thick was grown on the substrate by molecular beam epitaxy in the MBE lab of T. Tiedje at UBC. After thin film growth the wafer was shipped to NRC, Ottawa where it was patterned holographically with a 380 nm pitch oxide grating (6). The oxide strips were 160 nm wide and 70 nm thick. The structure of the wafer is illustrated in Fig. 4.2



Figure 4.2: Structure of the gallium arsenide sample.

The wafer was shipped back to UBC and cleaved into 11 roughly equal portions. Care was taken to ensure that the implanted region remained intact. The 11 wafer pieces were mounted with silicone grease in a circular arrangement about the center of a 3 inch silicon wafer. The silicon wafer was laid in a sample holder for the plasma etcher and was installed in the etch chamber. The plasma etcher was a load locked Plasma Quest series II ECR plasma etcher. An RF field at 13.56 MHz was supplied to the sample holder. A 2.455 GHz microwave field ionized the gases. Ions accelerated by the RF field were collimated by a 1200 Gauss magnetic field.

Relevant process parameters for the etching of the GaAs grating appear in Table 4.1

BCl ₃	2 sccm
Cl_2	1 sccm
Ar	$10~{ m sccm}$
RF bias	100 Vdc
Microwave power	100 W
Backside He Pressure	10 Torr
Process pressure	10mTorr

Table 4.1: Process parameters for the etching of the GaAs grating masked in oxide (sccm \equiv standard cubic centimeters per minute).

Etching of the GaAs samples was conducted in a series of discrete steps, each about 50s in duration. At the end of each etch, the detector was extended into the chamber and spectral data from the implanted portion of the wafer was collected. Also at the end of each etch, one of the 10 non-implanted portions of the wafer was removed from the etch chamber. Ultimately, the grating depth in each portion of the wafer was measured with an Hitachi S-4100 SEM. Selections of the AEL data and SEM images of the etched grating are presented in Section 5.2.
4.3 Textured membrane fabrication

The membrane grating structure was fabricated from a Silicon on Insulator (SOI) wafer. The SOI wafer consisted of a one micron thick oxide layer sandwiched between a silicon thin film and 550 microns of bulk silicon. Ultimately, the bulk silicon was fashioned into a support structure for the thin films that were left free standing in $(1 \times 3) \text{ mm}^2$ regions where the bulk silicon was removed. The oxide thin film formed the uniform base of the membrane structure. Above the oxide was a 5 micron pitch grating, fabricated from the silicon thin film. The sequence of steps involved in fabricating the textured membrane appear in Fig. 4.3. The wafer was cleaned and thermally oxidized in the clean room facilities of Simon Fraser University under the supervision of Bill Woods. Subsequent lithography and etching was done in the AMPEL clean room at UBC.

4.3.1 SOI wafer

The free standing silicon membrane existed initially as the polished surface of an SOI (Silicon on Insulator) bonded wafer (11). SOI technology achieves the electrical isolation of a silicon thin film from a supporting substrate, by introducing an oxide layer between the substrate and the thin film. Steps involved in the formation of SOI bonded wafers are illustrated in Fig. 4.4.

Original SOI structure

1

2

One micron Si thin film \longrightarrow One micron Si0₂ thin film \longrightarrow 550 microns bulk Si

Six Stages of Textured Membrane Fabrication



Growth of a 420 nm thick thermal oxide on both surfaces of the SOI wafer



Fabrication of a 5 micron pitch grating, 60% duty cycle, 100 nm deep in the top surface oxide

Definition of 1 X 3 mm slots in the bottom surface oxide



5

6

Anisotropic etch of the bulk silicon

Isotropic etch of the oxide to reduce the height of the oxide grating

Transference of the grating pattern to the silicon thin film by anisotropic etching

Figure 4.3: Membrane fabrication



Figure 4.4: Fabrication of SOI bonded wafers

A bonded SOI wafer is made by bringing an oxidized silicon wafer into contact with a polished silicon substrate. Pre-contact treatment of the wafers includes a cleaning process designed to make the wafer surfaces hydrophilic. The $(OH)^-$ groups that become adsorbed on the wafer surfaces after cleaning facilitate the bonding process, which happens spontaneously upon contact of the wafers at room temperature.

The spontaneous bond is reinforced by annealing at $\sim 1100^{\circ}$ C. As the temperature of the bond region is rising, bond reinforcement begins at around 300° C by the replacement of hydroxyl group bonding with groups of Si-O-Si:

 $Si - OH + OH - Si - > Si - O - Si + H_2O$

At 1100° C the oxide between the silicon layers flows viscously and effectively welds the surfaces together (12).

The SOI wafer used to fabricate the membrane structure was manufactured in Japan by Shin Etsu and was supplied by SEH America. The wafer diameter was 125mm. It had a $1 \pm 0.5 \ \mu$ m bond layer of N-type silicon, resistivity 1-3 Ω -cm, bulk level crystal quality. Thickness of the oxide thin film was $1 \pm 0.05 \ \mu$ m. The substrate was 550 microns of N- type silicon, resistivity 6-14 Ω -cm, bulk level crystal quality.

4.3.2 Wafer preparation

Processing of the SOI wafer began with an industry standard RCA clean. This cleaning recipe was developed by Werner Kern in 1965 while he was working at RCA (Radio Corporation of America). Details of the clean recipe appear in Table 4.2.

Motivation	Temperature	Time	Solution
Removal of organic contaminants	80° C	10 min	$5:1:1$ $H_2O:H_2O_2:NH_4OH$
Stripping of hydrous oxide film	80° C	10 min	$20:1$ $H_2O:HF$
Desorption of atomic ionic contaminants	90° C	10 min	$6:1:1$ $H_2O:HF$

Table 4.2: RCA clean recipe. Each 10 minute bath was immediately followed by a 5 minute rinse of the wafer in distilled water

4.3.3 Thermal oxidation

After cleaning, a film of silicon dioxide, SiO_2 was thermally grown on both surfaces of the SOI wafer. The oxide must eventually serve as a wet etch mask for the underlying silicon. Attempts to mask the silicon with oxide films grown by e-beam deposition failed: etch pits covered nearly 10 percent of the masked silicon before etching of the exposed silicon was complete. The etch pit density is indicative of the oxide film's porosity (13). Thermal oxides being denser than evaporated oxides are less porous, and so are better suited as wet etch masks for this experiment. Characteristics of thermal and e-beam evaporated thin films are reported in Table 4.3 along with those of quartz for comparison.

	Quartz (14)	Dry oxide (15)	Wet oxide (15)	Evaporated oxide (16)
Density g/cm^3	2.68	2.25	2.2	2.01
Youngs Modulus 10 ¹² dynes/cm ²	1.03	$0.67 \ (325 nm)$	0.57 (425 nm)	

Table 4.3: Density and Youngs modulus for quartz and themal oxides. Where Young's modulus values are given for the films the thickness of the thermal oxide film is specified.

The basic building block of SiO_2 is 4 oxygen molecules at the corners of a triangular polyhedron. A silicon atom is at the polyhedron's center. In the crystal structure of quartz, each oxygen atom is part of two polyhedra and so forms a bridging bond between silicon atoms. In the SiO_2 that grows thermally on the surface of silicon, the arrangement of the oxygen polyhedra is largely disordered - many oxygen molecules form nonbridging bonds in which they are bound to only one silicon atom.

Stage No.	Condition of O_2	${ m O}_2 { m flow rate} {(\ell/min)}$	${f N_2}$ flow rate (ℓ/min)	Stage duration (min)
1	dry	1.80	0	20
2	wet	0.45	1.80	45
3	dry	1.80	0	20

Table 4.4: Gas flow rates and stage duration for the three stage oxidation process used to form a 420 nm thin film on the surface of the SOI wafer at 1100° C

Comparing thermal oxides grown in the presence of steam to those grown under dry conditions, the oxides grown in dry conditions are comprised of a greater proportion of abridging bonds and so are denser and less porous.

Oxidation of the SOI wafer began immediately after the RCA clean. A 420 nm Si0₂ layer was grown on the SOI wafer by thermal oxidation at 1100° C. During oxidation the SOI wafer stood upright in a 21 ℓ quartz tube. The oxidation was conducted in three stages. During the first and third stages, dry oxygen flowed. The oxygen that flowed during the second stage passed through boiling water just before it entered the oxidation tube. Molecular nitrogen was also flowing in the second stage. Flow rates are specified for each stage in Table [4.4]

It was in the second stage that most of the oxidation occurred. When the wet oxygen flows, the oxidation rate is nearly 100 times the rate of oxidation in the presence of dry O_2 . The water molecule, being smaller, diffuses more rapidly into the oxide than does O_2 (7).

Though dry oxidation proceeds relatively slowly, it is preferred for the initial stage of the SiO_2 growth because dry oxides, being denser than wet ones, are more

effective in their intended role as barriers to silicon etchants. Ending the series with a dry oxidation causes less stress at the Si/SiO_2 interface than would the growth of a wet oxide directly on the silicon surface (17).

As the oxidation progresses, the silicon surface is consumed at a rate of 0.45nm per nm of oxide grown (18). Because of thin film interference, the thickness of an oxide can be determined by its colour, provided that its approximate thickness is known. Over a thickness interval of about 100 nm the thickness-colour relation is unique. Given the time for which the wafer was oxidized, the oxide thickness was approximately 400nm. With reference to oxide thickness colour charts (7) the pale pink colour of the oxide indicated a thickness of 420 ± 20 nm. By inference, the one micron membrane's thickness after oxidation was 810 ± 10 nm.

4.3.4 Patterning the oxide

A sample that measured approximately 1 cm^2 was cleaved from the oxidized SOI wafer. Subsequent processing began with the creation of a 5 micron pitch grating of photoresist on the sample's "top side" (see Fig. 4.3). The photoresist grating was made using photo-lithographic methods that are detailed in Appendix A.

The photoresist ran in 3 micron wide strips along the full length of the $1 cm^2$ region that was patterned in the center of the SOI sample. The sample then was immersed in a BOE, *buffered oxide etchant* until the unmasked SiO₂ regions had been etched to a depth of 100nm. The oxide etchant used was a distilled water solution comprised of 0.79% HF, 3.1% NH₄F. The etch rate of SiO₂ in the dilute BOE was approximately 12 nm/minute.

The oxide is etched by the reaction:

$$Si0 + 6HF - > H_2 + SiF_6 + 2H_2O$$

The role of the NH_4 buffer is to maintain the HF concentration so that the etch proceeds at constant rate. NH_4 dissociates to form ammonia gas and HF by the following reaction:

$$NH_4F \rightleftharpoons NH_3 + HF$$

Next, a uniform resist was spun onto the oxide grating and baked for 10 minutes on a hot plate at 100°C. The resist was needed to protect the grating while the backside oxide was patterned. By the lithographic procedure of Appendix A, a resist was spun onto the backside oxide and then patterned with (2 X 5) mm² slots. The sample was immersed in BOE so that the slotted pattern in the resist was transferred to the backside oxide.

4.3.5 Anisotropic etch of the bulk silicon

In the regions where the backside oxide was stripped, the bulk silicon was removed by an anisotropic silicon-etchant, ethylenediamine pyrocatechol (EDP). Desirable properties of EDP as a silicon etchant include the negligible rate at which it etches SiO_2 (200\AA /hr), and the marked directionality with which it etches silicon. Specifically, a relative etch rate of 17:10:1 (19) has been reported for the (100):(110):(111) surfaces of silicon, respectively. Silicon bars, fabricated from a 1 micron thick silicon thin film masked in SiO_2 and etched in EDP appear in Fig. 4.5. The (111) planes form the bar sidewalls. At the ends of the bars, the (110) and (011) planes are exposed.



Figure 4.5: Microscope image of the silicon bars at an edge of the patterned region. Also, schematic representation of the bars from three points of view.

The EDP for this experiment was a commercially prepared aqueous solution made up of 58% ethylenediamine and 11% pyrocatechol. Ethylenediamine ionizes in water to form hydroxide:

$$NH_2(CH_2)_2NH_2 + H_2O \rightarrow NH_2(CH_2)_2NH_3 + (OH)^{-1}$$

In aqueous ethylenediamine, silicon forms hydrous silica and hydrogen:

$$Si + 2(OH)^{-} + 4H_2O \rightarrow Si(OH)_6^{-} + 2H_2$$

The etch rate of silicon in a 2:1 ethylenediamine:water solution at $110^{\circ}C$ is $\sim 10\mu m/hr$ (13). To speed up the etch rate, pyrocatechol is added to ethylenediamine and water to form commercial EDP. Pyrocatechol serves as a complexing agent, removing the silica film that can form on the silicon surface during etching (19). The commercial EDP used here had a specified etch rate of $25\mu m/hr$ at $90^{\circ}C$. The observed etch rate was more than $50\mu m/hr$ because the EDP was exposed to air during the etch. Atmospheric oxygen, incorporated in the solution facilitates the etch process (19).

The etch rate can be further increased by increasing water concentration. For example, Reisman et al. (19) investigated the effect of adding water to EDP made up of 750 m ℓ of ethylenediamene with 120 g pyrocatechol and 4.5 g pyrazine, the latter being significant in deterring the formation of etch residues. When 100 m ℓ water was added to the solution, the etch rate at 75°C was less than 15 $\mu m/hr$. Increasing the volume of water added to 240 m ℓ resulted in an etch rate of more than 20 $\mu m/hr$.

In this experiment, 50 $m\ell$ of water was added to 75 $m\ell$ of EDP in a 400 $m\ell$ pyrex beaker at the start of the etch process. As illustrated in Fig. 4.6, an inverted pyrex funnel, lined at its base with aluminum foil was snuggly fit into the beaker mouth and served as a simple condenser. The funnel was easily removed to allow the sample to be introduced to, or removed from the EDP solution. Where the beaker rim dipped down to form a pouring spout, a pyrex thermometer was slipped into the beaker.



Figure 4.6: Arrangement of the inverted funnel and thermometer with the beaker that held the sample and the EDP solution

The beaker was set onto a hot plate in a vented clean bench. The hot plate set point was 180°C. After approximately 30 minutes, the EDP temperature stabilized at $92\pm2^{\circ}$. The sample was held vertically in a teflon holder that did not obstruct the flow of the EDP over the sample surface (see Fig. 4.7). A teflon string was attached to the holder and was the means by which the holder was lowered into the EDP solution.



Figure 4.7: Schematic drawing of the SOI sample in the teflon holder. The teflon holder does not extend across the base of the sample so that the upward flow of the hot etchant over the etch region is not obstructed.

Over the course of the etch, water was intermittently added to maintain the solution volume at 125 $m\ell$. In previous trials, no water was used in the original 125 $m\ell$ of solution, and only EDP was added to keep the solution volume constant, with the result that after more than 2 hours of etching, the etch appeared to stop. It was apparent that the loss of water by evaporation was dramatically reducing the etch rate.

The temperature of the EDP was kept between 90 - 95 °C for the first 5 hours of the etch. Then, the temperature was allowed to drop continuously to 75 °C where it was maintained until the removal of the bulk was complete. In previous trials the temperature was maintained at over 90 °C throughout the etch and the membrane broke as the final layers of the bulk were removed. Vigorous circulation of the EDP in the etch regions was thought to be the cause of the membrane failure. In the case that the etch temperature was reduced to 75 °C for the remainder of the etch, the membrane did not break in the EDP.

4.3.6 Isotropic etch to deepen SiO₂ grating

At stage 5 of Figure 4.3, the 100 nm deep SiO_2 grating was etched in dilute BOE until the silicon was exposed. While in the BOE the membrane was prone to breaking, likely because it was considerably deformed by compressive stress. An example of the effect of compressive stress on an free standing SiO_2 membrane is shown in Fig 4.8. The silicon substrate on which the SiO_2 film was grown by thermal oxidation has been masked and wet etched by the processes described in Section 4.3.5. Now free standing, the SiO_2 film responds to compressive stresses by buckling.



Figure 4.8: A free standing SiO_2 thin film, 420 nm thick

Using a model for thin film stress devised by Stoney (1909), Jaccodine and Schlegel (14) show that the stress σ_s of an isotropic film is related to the strain ϵ by Young's modulus, Y, and Poisson's ratio, γ : ¹

$$\sigma_s = Y \epsilon / (1 - \gamma) \approx \frac{5}{4} Y \epsilon$$

An approximation to the strain at a single Si/SiO₂ interface at temperature T is given by $\epsilon = \Delta_{TEC}\Delta_T$, where Δ_{TEC} is the difference in the thermal expansion coefficients of silicon and SiO₂ and Δ_T is the difference between T and the temperature at which the oxide was grown (20). Taking $\Delta_T = 1080^{\circ}C$, Y as in Table 4.3 and $\Delta_{TEC} = 32.5X10^{-7}T^{-1}$ ², the stress at the Si/SiO_2 interface is $2.9X10^9 dynes/cm^2$. This estimate agrees well with measured stress values of $2.8X10^9 dynes/cm^2$ reported by Schegel and Jaccodine for an 800 nm SiO₂ film grown on silicon at 1100 degrees.

Boron doped silicon is an alternative to oxide as an etch stop for EDP, if the doping concentrations is > 5×10^{19} [(21), p.425]. Silicon nitride, which exists under tensile strain on silicon can also serve as an etch stop. When the bulk silicon is removed, theses films do not wrinkle and bow as do membranes of SiO₂. [*ibid*, p.438].

4.3.7 Transfering the grating pattern to the silicon thin film

Finally the silicon thin film, now a free standing membrane patterned with a SiO_2 grating, was dipped again in EDP at 80°C. After one minute, time enough for the EDP to etch through the thin film in unmasked regions, the sample was removed to a 1 litre volume of distilled water and soaked there, without agitation, for 5 minutes.

¹Poisson's ratio is defined as minus the transverse strain divided by the axial strain in the direction of a stretching force

 $^{{}^{2}}TEC_{Si} = 38X10^{-7}T^{-1}$ [(12),8], $TEC_{SiO2} = 5.5X10^{-7}T^{-1}$ [(21),421]

The sample was then removed to a second water rinse, and again soaked for 5 minutes. In previous trials, a single water rinse was not sufficient to remove all traces of EDP from the sample surface. After the second water rinse was complete the sample was dipped in isopropyl alcohol for the purpose of removing water from the membrane surface; otherwise, the membrane could be broken by surface tension of water droplets as they evaporated off of the membrane surface. Once removed from the isopropyl, the membrane was left in air to dry. A microscope image of an SOI membrane grating structure is shown in Fig. 4.9.



Figure 4.9: A free standing SOI membrane grating structure

4.4 Implantation

The method employed here for profiling 1D gratings, involved the placement of a planar, monoenergetic source of alpha particles beneath the grating to be profiled. We discuss in this chapter how the planar alpha source was made; the process involved the recoil implantation of alpha-emitters in the polished surface of a crystalline wafer consisting of gallium arsenide or silicon. An implanted wafer of silicon, plated with a thin film of silicon dioxide after implantation, was used as an alpha source for profiling the silicon membrane structure. The thin film into which the studied gallium arsenide gratings were etched was grown by MBE directly on an implanted substrate of gallium arsenide.

4.4.1 Implantation chamber

To generate a planar source of alpha-emitting nuclei a polished wafer is brought into near contact with a radioactive source of ²²⁸Th for several days. When a ²²⁸Th nucleus decays by alpha particle emission, its ²²⁴Ra daughter, by conservation of momentum, recoils away from the ejected alpha particle. Many of the recoiling daughters travel in the direction of the wafer surface and so become implanted to a depth of a few nanometers. The implantation chamber is evacuated during implantation to a few millitorr in order that the ²²⁴Ra nuclei are not absorbed by atmospheric particles when traversing the ≈ 1 mm gap between the Thorium source and the wafer surface.

A photograph of the implantion chamber is shown in Fig. 4.10 and a schematic diagram of it is shown in Fig. 4.11. The chamber can accommodate two implantation sources, one in each of the retractable cylindrical containers that appears in the center of the chamber. A Thorium source has been installed in one of the containers. It is normally withdrawn from the chamber and housed behind a shuttered enclosure, except during implantation, when it is extended as shown. The circular aperture in the upper left corner of the image lies directly in front of the wafer to be implanted. The aperture diameter is 7 mm. The aperture mount is attached at to the chamber



Figure 4.10: View of the implantation chamber as seen from above with both source holders extended. The aperture plate in the upper left corner of the image has a hole diameter of 7 mm.

lid. When the sources are retracted, the aperture may be lowered into the chamber. When the lid is on tightly, the aperture is exactly aligned for optimal exposure to the extended source.

4.4.2 Implantated activity

The concentration of ²²⁴Ra nuclei as a function of the implantation time, t, can be calculated as follows: Let $N_p(t)$, $N_d(t)$ denote the concentration of ²²⁸Th , and ²²⁴Ra respectively, and let λ_p and λ_d be their respective decay constants. Let t=0 be the time when implantation begins and let $N_p(0) = N_0$, $N_d(0) = 0$.

Given the exponential decay of $^{228}\mathrm{Th}$ and $^{224}\mathrm{Ra}$,

$$dN_d/dt = \lambda_p N_0 e^{-\lambda_p t} - \lambda_d N_d$$



Figure 4.11: Schematic side view of the implantation chamber with the Thorium source extended and a wafer in place for implantation

The complete solution for the time dependent concentration of 224 Ra is:

$$N_d = N_0 \frac{\lambda_p}{\lambda_d - \lambda_p} (e^{-\lambda_p t} - e^{-\lambda_d t})$$

The half life of ²²⁸Th is 1.9 years and that of ²²⁴Ra is 3.7 days; therefore $\lambda_d - \lambda_p \approx \lambda_d$. For implantation times, t, such that, $\lambda_p t \ll 1$, $\lambda_d t \gg 1$, we have $N_p \approx N_0$ and for the maximum ²²⁴Ra activity,

$$\lambda_d N_d \approx N_p \lambda_p$$

The activity of the Thorium source is 2.5 μ C, or 90 kBq. The maximum ²²⁴Ra will actually be *one half* the Thorium activity since no more than half of the nuclei ejected from the planar Thorium source will travel in the direction of the implanted wafer.

For this experiment the gallium arsenide and silicon wafers were implanted for approximately one week, by which time the Ra activity was 75 % of its maximum value.

4.4.3 The alpha particle spectrum

Each implanted ²²⁴Ra nucleus eventually decays by one of several possible decay sequences to ²⁰⁸Pb (22). Six alpha lines appear in the spectrum of an implanted wafer. The lowest energy alpha lines are due to the alpha decay of ²²⁴Ra into ²²⁰Rn which creates an alpha particle with an energy of 5685 or 5449 MeV; the 5685 MeV alpha particle occurs in 95 % of all ²²⁴Ra decays. The alpha energy loss rate in matter, dE/dx, increases as energy decreases, making the low energy alphas of ²²⁴Ra decay relatively sensitive to incremental changes in thin film thickness and especially useful for determining the height of shallow gratings.

 $^{220}\mathrm{Rn}$ and its daughter, $^{216}\mathrm{Po}$ both decay by alpha particle emission. $^{220}\mathrm{Rn}$ emits an alpha particle with 6250 keV. The alpha particle of $^{216}\mathrm{Po}$ decay has a kinetic energy of 6779 keV. The beta decay of $^{216}\mathrm{Po}$'s daughter produces $^{212}\mathrm{Bi}$ which decays in one of three ways. There is a 36 % chance that a $^{212}\mathrm{Bi}$ nucleus will alpha decay, producing a 6090 keV or 6050 keV alpha particle, with respective probabilities of 27 % and 70 % . Otherwise, $^{212}\mathrm{Bi}$ beta decays to $^{212}\mathrm{Po}$ which by alpha emission produces an alpha particle with 8785 keV kinetic energy. This relatively high energy alpha particle is useful for the spectroscopy of very deep gratings since its spectrum, being well removed from the lines of other alpha decays, is not "blurred" by the spreading of neighboring spectra. In Fig. 4.12 the spectral lines of $^{224}\mathrm{Ra}$ and its daughters are shown. This data was collected when the AEL method was applied as a thin film growth monitor. Immediately after $^{224}\mathrm{Ra}$ nuclei were implanted in a silicon wafer, a thin film of SiO₂ was evaporated by electron beam deposition onto the wafer. The



Figure 4.12: Alpha particle spectra of an implanted silicon wafer before and after the growth of 490 nm of Al. The greater height of the "pre-deposition" spectra is due to the longer collection time associated with it.

oxide film served as a protective barrier for the implanted region. The spectral data in Fig. 4.12 was collected before and after thin film of aluminum was sputtered on top of the oxide layer.

The centroid of each spectral line in the "post-sputtering" spectrum lies to the left of its position before sputtering took place. Notice that the energetic shift is greatest for the ²²⁴Ra spectral lines.

Chapter 5

Data

5.1 Silicon membrane structure

Following the membrane fabrication processes of Chapter 4.3 and the creation of the alpha source, as in Section. 4.4, a silicon membrane structure was placed in the experimental setup of Fig. 4.1. The structure used for spectroscopic analysis appears in Fig. 5.1. The grating is evident in the striated appearance of the membrane's surface. Bowing of the membrane is apparent. The gently bending, shiny regions are raised regions of the membrane. The sharply folded dark regions are depressions or "shallows". Fractures can also be seen in the image, often in the folds of the shallows. As will be discussed, evidence of the fractures also appears in the spectral data.

The bowing is due to stresses inherent in the oxide layer of the original SOI structure, as discussed in Chapter 4.3. The amplitude of the vertical undulations was found to be approximately 12 microns by measurement with an optical microscope; the microscope's stage translation was calibrated so that the stage translation required to bring the uppermost and lowermost regions of the grating into focus could be

CHAPTER 5. DATA

converted to a vertical distance measurement.



Figure 5.1: Grating viewed with an optical microscope at 5X magnification

A view of the membrane's underside in Fig. 5.2 shows that some of the silicon bulk remains as a supporting structure that may keep the fractured membrane from breaking completely.



Figure 5.2: Underside of grating viewed at 5 X magnification

Bordering the regions where the thick residual silicon appears black are lighter regions where the silicon remains as a transparent, relatively uniform thin film. The thick silicon regions contribute to spectral events over a broad energy interval that extends well below the spectral region of the membrane grating, and therefore do not interfere with the analysis.

The experimentally observed alpha spectrum is shown in Fig. 5.4 along with the simulated spectrum corresponding to the best fit. The extracted "best" geometric dimensions are shown to scale in Fig. 5.3, superimposed on an actual cross-sectional view of the grating. By comparison of the distance scale on the micrograph with the known pitch of the grating pattern, the horizontal dimensions in Fig. 5.3 appear to be 6 percent less than their true value. At the end of Section 4.3.3, the stated thickness of the silicon membrane is 810 ± 20 nm. In Fig. 5.3, the silicon bars appear to be 790 ± 50 nm thick.



Figure 5.3: Cross-sectional view of the membrane grating overlaid with a sketch of the simulation profile that best fit the spectral data.

In Fig. 5.3 it can be seen that the oxide mask overhangs the grating sidewalls. The overhangs will cause spectral events between the peak energies to depart from a perfectly uniform distribution; though in this case, dispersion because of the overhangs is negligible - their presence might be simulated as grating sidewalls that sloped more or less linearly, depending on the extent and thickness of the oxide overhangs.

The grating parameters extracted by the simulation are $h_1=830\pm 20$ nm, $h_2=895\pm 30$ nm, $b_1=3200\pm 50$ nm, $b_2=2290\pm 50$ nm. The fit between the simulation and the actual data is rather striking with particular attention to be paid to the slope of the sidewall. To demonstrate the essential role played by this slope in obtaining the fit, we show, in Fig. 5.5, the best fit that would have been obtained if the sidewalls were forced to be perpendicular to the membrane. The inadequacy of such an ansatz is clearly apparent.



Figure 5.4: AEL spectrum (discrete points) for the membrane structure of Fig. 5.3 with best fit simulation (continuous line).



Figure 5.5: As in Fig. 5.4 except that the best fit simulation was found under the constraint that the grating sidewalls were perfectly vertical.

However, the average slope of the sidewalls, as determined by the simulation, was 47° off the normal with an accuracy of \pm 4°. By SEM measurement the sidewall

CHAPTER 5. DATA

slope was approximately $37\pm2^{\circ}$, very nearly the angle of the (111) plane with respect to the [100] direction in which the etch proceeded. The excess in sidewall slope measurement by the AEL method is likely due to sources of dispersion not accounted for in the simulation, such as etch pits or residual EDP in the trough regions of the grating.

The fitting of the simulated spectrum to the observed one was carried out over a "region of interest" extending roughly from 8550 keV to 8750 keV. The experimental spectrum, however, contains counts outside this region as well : about 9 % of the total in a somewhat broadened peak at the original alpha energy; about 5 % of the total distributed over lower energies and corresponding to larger thickness. The high energy peak is attributable to the cracks in the structure, visible in Fig. 5.1. The low energy tail is attributable to the thin residual silicon material evident in Fig. 5.2.

5.2 GaAs ridges

Spectra of the GaAs grating were collected in the setup of Fig. 4.1, during the etch process described in Section 4.1. The spectra for three etch depths are presented here. The etch depth is taken to be the depth of the grating including the thickness of the SiO_2 mask, with a correction to account for the reduced energy loss rate for alpha particles in SiO_2 . Measurements of the etch depth made with an Hitachi S-4100 SEM are compared to etch depth values determined by a least squares fitting of simulated spectra to the experimental spectra.

5.2.1 Shallow grating depth

The spectral data in Fig. 5.6 was taken from a GaAs sample into which the grating had been etched to a depth, $t_{GaAs} = 80 \pm 5$ nm, as measured with the SEM. The silicon dioxide mask thickness, t_{Si02} , was 55 ± 5 nm, a measurement also made with the SEM. After passing though 80 nm of GaAs the alpha particles of the ²²⁴Ra decay have a kinetic energy of 5450 keV. At this energy, the energy loss rate in SiO₂ is 51% of its value in GaAs. An *effective* grating height, t_{eff} , served as the grating height input to the simulation. It was calculated as $t_{eff} = t_{GaAs} + 0.51 * t_{Si02}$.

The spectrum of Fig. 5.6 is not dramatically distinct from the uniform Al film spectrum of Fig. 4.12. The linewidth and centroid of the peak are, however, sensitive to the depth of the grating. The peak of the simulated spectrum passes though the AEL spectral peak as the effective grating thickness input to the simulation is varied from 100 to 130 nm, the best fit being achieved at 115 ± 15 nm, as shown in

Fig. 5.6. Based on the SEM measurements of the GaAs ridge height and the oxide cap thickness, the effective grating thickness was 108 ± 8 nm. To make the uncertainty of the AEL measurement comparable to that of the SEM measurement, longer data collection times would have to be employed. Given that the half life of the implanted nuclei is 3.7 days and the present data set was collected over 5 minutes, longer data collection times are quite feasible.



Figure 5.6: Simulation for effective etched depths of 100, 115 and 130 nm shown with GaAs grating spectral data. Δ is difference between the actual and simulated data. The Gaussian linewidth convolved with the simulated data is 44 keV FWHM

5.2.2 Intermediate grating depth

An SEM image of the grating when the etch depth was approximately 415nm appears in Fig. 5.7. Superimposed on the micrograph is the grating profile for the simulation that best fit the AEL spectra and that appears with the AEL data in Fig. 5.8.



Figure 5.7: Image of GaAs grating etched to 415 nm depth with the profile of the best fit simulation superimposed

Because of vertical distortion due to viewing angle of the micrograph, only horizontal dimensions of the overlaid profile and the micrograph should be compared.

Clearly, the simulation has overestimated the extent to which the sidewalls are not perfectly vertical, likely because sources of dispersion such as etch products in the grating troughs or etch pits beneath the oxide caps have raised the level of the intra-peak region in the AEL spectra. By SEM measurement, the etch depth in GaAs was 415 ± 10 nm. The thickness of the SiO₂ mask was 40 ± 5 nm.



Figure 5.8: Best fit simulation with GaAs grating spectral data for an etch depth of 415nm.

Overall agreement of the simulated data and the real spectral data is good, both in the region of the peaks and between them. For the simulation shown, $t_{eff} =$ 425 ± 15 nm, in good agreement with $t_{eff} = 434 \pm 15$ nm obtained from the SEM measurements of the grating and oxide thicknesses.

5.2.3 Deep gratings

An SEM image of the grating at approximately 750nm etch depth appears in Fig. 5.9. Superimposed on the image is an outline of the profile assumed for the simulation that appears in Fig. 5.10 along with the spectral data. Only the horizontal dimensions in the figure should be taken as "true".



Figure 5.9: Image of GaAs grating at an etch depth of 765 nm. The sidewall angle of the simulated profile superimposed on the grating image is approximately 8 degrees.



Figure 5.10: Simulation for sloped walls with GaAs grating data.

The sidewall angle for the simulation of Fig. 5.10 was eight degrees. For the simulation of Fig. 5.11 the sidewalls were assumed to be vertical. By comparing the two figures we can see that the effect of non-vertical sidewalls is to redistribute counts from the peak regions into the area between the peaks. However, the trapezoidal grating shape of the simulation is only a first order approximation of the grating profile.

With proper incorporation of straggling in the simulation, the width of the low energy peak is 5 keV broader than the 39 keV FWHM of the high energy peak.

By SEM measurement, the etched depth in GaAs was 765 ± 15 nm, the thickness of the SiO₂ grating was 20 ± 5 nm; therefore, the effective grating height was 775 ± 20 nm. Independent of sidewall angle, the simulation predicts a grating height of 770 ± 15 nm.



Figure 5.11: Simulation for vertical sidewalls as compared to the experimental spectrum for the deep GaAs grating. Clearly, the region between the peaks is not fit well when the sidewalls are assumed to be vertical.

Chapter 6

Summary and Conclusions

In this thesis, various aspects of alpha-particle energy loss spectroscopy applied to grating structures were investigated. For gallium arsenide gratings, it was demonstrated that the AEL method can resolve grating depths to within 15 nm for etch depths of 110, 415 and 775 nm. This resolution is comparable to that achievable with scanning electron microscopy. Furthermore, the resolution of the AEL measurement is not worsened if the aspect ratio (ratio of the grating height to pitch) is very low - this is an advantage of the AEL method over the SEM method of imaging, since small vertical changes over large horizontal distances cannot be viewed easily with the SEM. At 750 nm depth, the non vertical nature of the sidewalls was detectable in the AEL data. By comparison of the AEL spectra with simulated data for a trapezoidal grating shape, it was demonstrated that the grating sidewalls were not perfectly vertical. However, the trapezoidal shape was not a good approximation of the GaAs ridge profile.

A free standing 1D grating structure comprised of silicon bars on a silicon dioxide membrane was fabricated from a Silicon-On-Insulator (SOI) wafer, and served

as an experimental grating structure. It was used to demonstrate the AEL method of profiling gratings with linearly sloped sidewalls. The fabricated structures were prone to breaking, probably because they were not flat. The oxide membrane, a thermal oxide grown on silicon, rippled when the bulk silicon beneath it was etched away. Alternatively, the membrane structure could be fabricated from a silicon wafer that was boron doped ($n > 10^{20}$ cm⁻³ (21)) to a uniform depth along its polished surface. The wafer could be oxidized and then patterned with an oxide grating on its topside and with slots in its backside oxide - as was done here. The bulk silicon beneath the boron doped layer could be removed in EDP. Then the p doped silicon could be plasma etched (21) through the oxide grating on its topside.

The AEL method was also applied to a grating structure of silicon bars on a free standing SiO₂ membrane. The membrane structure was fabricated from a Silicon-On-Insulator wafer, using thermal oxide as a wet etch mask and diluted EDP as the etchant. Good agreement was obtained between the AEL spectral data and simulated data based on trapzoidal shaped silicon bars. The sidewall slope was determined to be 47° off the normal with an accuracy of ± 4 °. By SEM measurement and by theoretical prediction of the etched sidewall angle, the sidewall slope was approximately 37°. The AEL method determined the sidewall angle to be larger than its actual value. The excess in the AEL measurement of the sidewall slope is likely due to sources of dispersion that were not accounted for in the simulation. The AEL method measures average properties of structures, and is sensitive to the overall quality and uniformity of an etched grating. This sensitivity can interfere with quantitative measurements but is useful for qualitative assessment of the etched grating profile.

Bibliography

- [1] H.A. Bethe. Annalen der Physik, 5,325 (1930).
- [2] F. Bloch. Annalen der Physik, 16, 285 (1933).
- [3] I Kelson, Y Levy, and E Redmard. J. Physics. D : Appl. Phys., 28, 100 (1995).
- [4] I Kelson, Y Levy, D Racah, E Redmard, M Beaudoin, T Pinnington, T Tiedje,
 and U Giesen. J. Physics. D : Appl. Phys., 30, 161 (1997).
- [5] I Kelson, C V Kaiser, Y Levy, G Biasiol, and E Kapon. Nuclear Instruments and Methods in Physics Research B, 170, 483 (2000).
- Y Levy, A Ballestad, M Davies, Y Feng, I Kelson, W J Mandeville, V Pacradouny,
 A Schmalz, T Tiedje, and J F Young. J. Crystal Growth, 26, 201 (1999).
- [7] Stephen A. Campbell. The Science and engineering of microelectronic fabrication. Oxford University Press, New York, (1999).
- [8] John David Jackson. Classical Electrodynamics. John Wiley & Sons, New York, 1999.

- [9] William R. Leo. Techniques for Nuclear and Particle Physics Experiments. Springer-Verlag Berlin Heidelberg, Germany, 1987.
- [10] et al. C. Bricman. Reviews of Modern Physics, 285 (1980).
- [11] W.Maszara, G.Goeta, A.Caviglia, and J.B.McKitterick. J. Appl. Phys, 64, 4943 (1988).
- [12] Jean Pierre Colinge. Silicon on insulator technology. Kluwer Academic Publishers, Boston, 1991.
- [13] R.M. Finne and D.L Klein. J. Electrochem. Soc., 114, 965-970 (1967).
- [14] R.J. Jaccodine and W.A. Schegel. J. of Appl. Phys., 37, 2429 (1966).
- [15] K. E. Petersen and Guarnieri. J. Appl. Phys, 50, 6761 (1979).
- [16] Norman F. Raley and Theodore Van Duzer. J. Appl. Physics, 58, 285 (1985).
- [17] J.T. Fitch and G. Lucovsky. The effect of growth ambients on the local atomic structure of thermally grown si02 thin films. In S.T. Pantelides and G Lucovsky, editors, *Si02 and Its Interfaces*, page 155, Pittsburg, Pennsylvania, 1987. Materials Research Society.
- [18] H.B. Pogge. *Electronic Materials Chemistry*. Marcel Dekker, New York, 1997.
- [19] A. Reisman, M. Berkenblit, S.S. Chan, F.B. Kaufman, and D.C. Green. J. Appl. Phys, 50, 6761 (1979).
BIBLIOGRAPHY

- [20] Subash Mahajan and K.S. Sree Harsha. Principles of Growth and Processing of Semiconductors. McGraw-Hill, Toronto, 1999.
- [21] Kurt E. Petersen. Proceedings of the IEEE, 70, 421 (1982).
- [22] C.M. Lederer and V.S Shirley. Table of Isotopes, 7th edition. Wiley, New York, 1977.

Appendix A

Recipe for patterning a 1.0 μ m resist

Spinning on the resist

Lay the sample on the spinner chuck and engage the vacuum that holds the sample to the chuck 1 .

Dispense 3-4 drops of Micro Si "MicroPrime" (hexamethyl disilazane) to the sample surface with a disposable pippet.

Spin the sample at 7000 r.p.m. for 40s.

Dispense 3-4 drops of AZ4110 resist to the sample surface with a disposable pippet.

Spin the sample at 7000 r.p.m. for 40s.

Remove the sample from the chuck to the hot-plate and bake for 5 min at 100°

C. After baking, allow the sample to cool for 10 min.

Exposure and development for bead edge removal

Place the sample in the mask aligner.

Center a rectangular piece of silicon, polished side down, on the resist exposing only corner regions of the resist where colour changes are apparent.

In "Lamp test mode" expose the resist to 200 mJ: 50s @ 40 mW/cm².

Develop the resist in 4:1 distilled water: AZ400K developer for 30 s

¹If the vacuum seal does not hold, the problem is likely the presence of residual resist on the chuck. Resist can be removed with acetone on a kim wipe.

Rinse the resist for 60 s in distilled water

Gently dry the sample with N_2 .

Soft bake the sample for 5 min at 100 $^\circ$ C, then allow sample to cool for 10 minutes

Exposure procedure for patterning the topside resist

Place sample on the mask aligner chuck, topside up, and engage the vacuum seal.

Install and align the grating ruled quartz mask in the mask aligner.

Engage the mask aligner in its programmed control mode and expose the sample to 200 mJ: 50s @ 40 mW/cm².

Exposure procedure for patterning the backside resist

Center the slotted silicon mask on the backside resist.

In "Lamp test mode" expose the resist to 200 mJ: $50s @ 40 \text{ mW/cm}^2$.

Developing the patterned resist

Develop the resist in 4:1 distilled water: AZ 400 K developer for 30 s. Rinse the resist for 60s in distilled water. Gently dry the sample with N_2 .