Study of roughness evolution during reactive ion etching

Santosh Kumar Pani

2006

Santosh Kumar Pani. (2006). Study of roughness evolution during reactive ion etching.
Doctoral thesis, Nanyang Technological University, Singapore.

<https://hdl.handle.net/10356/35966>

<https://doi.org/10.32657/10356/35966>

Study of Roughness Evolution during Reactive Ion Etching

Santosh Kumar Pani

School of Materials Science and Engineering

A thesis submitted to the Nanyang Technological University
in fulfillment of the requirement for the degree of
Doctor of Philosophy

2006

Abstract

Polymers are promising candidates for cost-sensitive integrated optical components such as waveguides and waveguide based devices for communication applications and optical interconnects for chip to chip, board to board and back plane applications because of their versatility. Reactive ion etching (RIE) is a commonly used patterning technique for the fabrication of polymeric waveguides and waveguide based devices. One important consequence of RIE in the fabrication process is the evolution of roughness at different interfaces. Current research focuses the fabrication of polymeric optical waveguides by photolithography and RIE with the emphasis on issues which are bottlenecks for the waveguiding/interconnects technologies. In particular, this research work concentrates on studies of various types of roughness evolved during RIE of polymeric waveguides. Important contributions to the field of waveguiding/interconnect technologies include measurement of various types of roughness evolved during RIE, new discovery of etch depth dependences and the explanation of this phenomenon based on Monte Carlo simulation of etch-ion dynamics, formulation of the hypotheses and the etching mechanism, and controlling these roughnesses by developing novel recipes.

Various types of roughness such as planar film roughness (PFR), line edge roughness (LER) and sidewall roughness (SWR) are evolved during RIE of polymer waveguides. They were defined distinctly to avoid ambiguities and were directly measured by atomic force microscopy (AFM). It was confirmed that line edge roughness and sidewall roughness are different and should be treated as two different roughness parameters for various theoretical and experimental formulations of optical loss prediction.

The etched planer films were studied to obtain the information on evolution of sidewall roughness. Planar film roughness of these etched films exposed to pure O₂ plasma at different pressures during RIE was measured by AFM. It was found that with the increase of pressure, planar film roughness decreases in a linear trend and this effect is known *Pressure Effect* of planar film roughness.

Effect of RIE parameters on SWR of fluorinated polyether (FPE) waveguides was studied by varying RF power and pressure. According to our study, it was found

that higher RF power provides more vertical profile along with better etch bias, though SWR increases with increase of power. Sidewall roughness can be controlled by varying the pressure. It was also found that vertical etch rate increases with increase of pressure. Furthermore, it was confirmed that higher pressure renders rougher sidewalls, and vice versa and this effect is known as *Pressure Effect* of sidewall roughness. Therefore, the *pressure effect* of sidewall roughness and planar film roughness is opposite to each other.

Variation of sidewall roughness along the vertical depth of the waveguides was first observed by field emission scanning electron microscopy (FESEM) and measured and confirmed directly by AFM for the first time. The effects of RF power and pressure on variation of sidewall roughness were studied. It was observed that by varying RF powers, sidewall roughness along the depth of the waveguides does not vary significantly and the nature of variation of SWR remains almost same in all power ranges. We found that pressure is the most influential parameter which causes the variation of sidewall roughness along the depth of the waveguides. At low pressure of 35 mT, the sidewall roughness initially increases, then decreases, and finally increases and it varies between 22.1 nm to 33.3 nm along the depth (thickness) of the waveguides. However, at a higher pressure of 95 mT, sidewall roughness shows a gradual decline from 57.5 nm to 13.1 nm along the same vertical depth. We confirmed the pressure dependence (*pressure effect*) of sidewall roughness for shallow structures. In other words, variation of sidewall roughness is higher at higher pressure and lower at lower pressures for the waveguides of lower etch depths. We also discovered an additional etch depth dependence for deep etched structures which counteracts the pressure dependence at certain critical depth. After critical depth, lower O₂ pressure etching produces sidewall roughness which increases with etch depth while higher O₂ pressure etching produces decline of sidewall roughness with the depth. This effect is known as *Depth Effect*.

Evolution of sidewall roughening of fluorinated polyether waveguides fabricated by RIE in pure O₂ plasma was investigated. Lower pressure sidewall roughness variation along the vertical depth was due to the combination of both shadowing and the first order reemission effect and higher pressure sidewall roughness variation was postulated to be due to a continuous shadowing effect

(CSE). It was discovered that the autocorrelation length (ACL) in each RIE condition varies in a similar manner as the sidewall roughness (i.e. the tracking of autocorrelation length with sidewall roughness) along the depth of the waveguides, suggesting that the surface roughness evolution at the sidewalls during plasma etching possesses the same dynamics as that of planar thin film etching. Furthermore, the average value of roughness exponent increases as the pressure increases.

Evolution of sidewall roughness studies was further carried out with the help of Monte Carlo simulation method. It was further found that with the decrease of shadowing sticking coefficient, sidewall roughness increases along the depth of the waveguide. Additionally, the noise also increases as the shadowing sticking coefficient decreases because of increase in number of reemitted particles. The experimental results were compared with the simulated results. It was found that experimental results and postulations agreed well with the simulated results.

Sidewall roughness can be *controlled* at an optimum RIE conditions using pure O₂ plasma during RIE. However, in this research work, SWR was *controlled* by varying the gas composition during RIE. Particularly, N₂ was added in pure O₂ plasma. It was observed that O₂/N₂ plasma follows the similar *pressure effect* as pure O₂ plasma does. However, O₂/N₂ plasma provides smoother sidewalls at all conditions by varying pressure than pure O₂ plasma. The pressure effect on sidewall roughness in O₂/N₂ plasma can also be explained by using the oblique to normal ratio of incident ions mechanism. The reduction of sidewall roughness in O₂/N₂ plasma could be due to the presence of additional nitrogen containing bonds such as N⁺, CN⁺ and CNO⁺ bonds in the surfaces of etched thin films characterized by time of flight secondary ion mass spectroscopy (ToF-SIMS). The presence of nitrogen containing bonds was further confirmed by depth profiling the etched thin films.

Acknowledgements

I would like to take this opportunity to express my heartfelt gratitude and appreciation to my thesis supervisor, Professor Chee Cheong Wong whose guidance and support has always been a source of motivation to me throughout this work. While working with him, I learned the level of independency that a motivated researcher has to pursue in his research career to explore novel ideas, concepts and to trust my innate abilities and intuition. He rendered me an encouraging, goal-oriented and prolific atmosphere in order to practice a self-motivated research by making individualistic decisions as well as made sure that my research works remain on the right track without which I would not have come up to this stage.

I am equally greatly indebted to my co-supervisors Dr. K. Sudharsanam, Dr. Mahadevan K. Iyer and Mr. C. S. Premachandran for providing the training, support and guidance throughout this research work at *Institute of Microelectronics (IME), Singapore*. I am also greatly obliged to my co-supervisor Dr. David K. Y. Low for rendering training, collaboration, cooperation and guidance to work in the recent emerging Femtosecond laser induced waveguide studies at *Singapore Institute of Manufacturing Technology (SIMTech), Singapore*. My special thanks to Prof. Subodh G. Mhaisalkar and Prof. Murukeshan Vadakke Matham for their invaluable advices, motivations and encouragements which enabled me to finish my research works despite the difficulties.

I am very thankful to IME Staff- Mr. Pamidighantam V. Ramana, Mr. Damaruganath Pinjala, Mr. N. Ranganathan, Dr. V. Kripesh, Dr. Seung Wook Yoon, Dr. Zheng Yuan Jin, Mr. S. Mohanraj, Mr. Kum Weng, Ms. Vanissa Lim, Ms. Jasmine, Mr. Bryan Lee, Ms. Joanne Tan and all other IME staff for their generous help, support and assistance in this research. I am also grateful to Dr. John Eccles, Director of Millbrook Instruments Limited, Blackburn, UK for carrying out SIMS, and also to Ms. Doreen Mei Ying Lai and Dr. Nikolai Yakovlev, at Institute of Materials Research and Engineering (IMRE) for providing me assistance in carrying out ToF-SIMS. My training on Prism Couplers for Refractive Index measurement by Dr. S. R. Natarajan is greatly appreciated. Valuable assistance from all my Microelectronics Materials Lab Technicians in Nanyang Technological University (NTU) is highly acknowledged. I am also indebted to Ms. Chan Soon Keng for correcting and providing invaluable suggestions to finally complete this thesis.

Last but not least, I would like to express my sincere and heartfelt gratitude towards my beloved parents, brothers, wife, and brother-, mother- and father-in-laws, relatives and all of my friends in NTU, NUS and IME who have showered their love, affection and support towards me during this needful time.

Contents

	Page No.
Abstract	i
Acknowledgements	iv
Contents	vi
List of Figures	xi
List of Tables	xix
Acronyms	xx
1. Introduction	1
1.1. Waveguides and their issues	4
1.2. Objectives	5
1.3. Scopes	6
1.4. Thesis organization	6
2. Literature Review	8
2.1. Materials for the waveguides	8
2.1.1. Why polymer materials are so attractive for waveguides	9
2.1.2. Polymers for waveguides	10
2.1.3. Absorption loss in polymers	19
2.2. Fabrication of waveguides	21
2.2.1. Evolution of roughnesses during dry etching	22
2.2.1.1. Evolution of planar roughness	23
2.2.1.2. Evolution of sidewall roughness of polymer waveguides	24
2.2.2. Measurement of SWR and LER	25

2.2.3. Analytical approaches of sidewall roughness studies	28
2.3. Summary	33
3. Experimental Studies	35
3.1. Materials Characterization for cladded waveguides	35
3.1.1. Refractive index measurement (Curing Optimization)	35
3.1.2. Moisture absorption	38
3.1.3. Thickness measurement of planar films (Spin coating optimization)	40
3.2. Process studies for fabricating cladded waveguides	42
3.2.1. Spin coating	42
3.2.2. Hard mask fabrication	43
3.2.3. Dry etching	44
3.3. Surface Characterizations	44
3.3.1. SEM	44
3.3.2. AFM	45
3.3.2.1.Characterization of SWR	45
3.3.2.2.Characterization of LER	46
3.3.2.3.Characterization of PFR	46
3.3.3. SIMS	47
4. Direct Measurement of Sidewall Roughness of Polymeric Optical Waveguides	48

4.1. Classification of various types of roughness evolved during RIE	48
4.2. Measurement of LER at different pressure conditions	50
4.3. Measurement of surface area roughness of sidewall	50
4.4. Measurement of planar film roughness	52
4.5. Summary and conclusions	52
5. Effect of Process Parameters on Sidewall Roughness in Polymeric Optical Waveguides	54
5.1. Process parameter studies	54
5.1.1. Effect of RF power on SWR	54
5.1.2. Effect of pressure on SWR	60
5.1.2.1. Pressure Effect Explanation	62
5.2. Summary and conclusions	65
6. Variation of Sidewall Roughness of Polymeric Waveguides during Reactive Ion Etching	67
6.1. Variation of SWR	67
6.1.1. Variation of SWR at different RF powers	67
6.1.2. Variation of SWR at different Pressures	70
6.2. Evolution of variation of SWR	77
6.2.1. Pressure effect	77
6.2.2. Depth effect	79
6.2.3. Mechanisms for pressure and depth effects	80
6.2.4. Variation of ACL at different pressures	83
6.2.5. Variation of Roughness Exponent (α) at different pressures	86

6.3. Summary and conclusions	88
7. Study of evolution of planar film roughness during reactive ion etching of polymers	90
7.1. Variation of PFR with pressure	90
7.2. Evolution of PFR with time at different pressures	92
7.3. Summary and conclusions	104
8. Monte Carlo Simulation Studies of Sidewall Roughness Evolved during Reactive Ion Etching	105
8.1. Monte Carlo Method	105
8.2. Simulation Model	106
8.3. SWR Analysis	112
8.4. Evolution of SWR with Different Etching Modes	113
8.5. Variation of other simulation parameters	125
8.6. Comparison of simulated data with experimental results	126
8.7. Summary and conclusions	128
9. Reduction of roughness in polymer waveguides using O₂/N₂ gas mixtures during reactive ion etching	130
9.1. Pressure effect on SWR	130
9.2. Comparison of pressure effect on SWR treated with pure oxygen plasma	131
9.3. Pressure effect of on variation of SWR	132
9.4. Surface characterization by SIMS	134
9.5. Optimum Conditions for cladded Waveguide fabrication	140

9.6. Summary and conclusions	141
10. Conclusions and Future Work	142
10.1. Conclusions	142
10.2. Future Work	146
References	147
List of Publications	154
Appendix A	156
Appendix B	167

List of Figures

Figure Captions	Page No.
Fig.1.1 Schematic Diagram of DWDM System	2
Fig 1.2 Cross-section of waveguide with various layers	3
Fig. 2.1 Absorption spectra of deuterated phenylsilyl chloride ($C_6D_5SiCl_3$) monomer and normal phenylsilyl chloride ($C_6H_5SiCl_3$)	15
Fig. 2.2 Optical loss dependence on the exposure time to 85% relative humidity at 85 °C for waveguides on silicon substrate	16
Fig. 2.3 Chemical structure of crosslinkable fluorinated poly (arylene ethers) with phenylethynyl group (FPAE-PEP)	18
Fig. 2.4 Refractive Index stability of FPAE at 120 °C	19
Fig. 2.5 Calculated absorption losses for various vibrational motions	21
Fig 3.1 Chemical formulae of (a) cladding (ZP2145M) and (b) core (ZP1370M)	36
Fig. 3.2 (a) Variation of Refractive index with moisture conditioning time of ZP1370M	39
Fig. 3.2 (b) Variation of Refractive index with moisture conditioning time of ZP2145M	39
Fig. 3.3. Schematic diagram of a six inch Si wafer, where the numbers represent the location of the wafers at which thickness was measured	40
Fig: 3.4 Attenuation loss of ZP1370M at different spinning speed	41
Fig.3.5 Curing profile of FPE	43
Fig. 3.6 Process flow chart for waveguide fabrication	43

Fig 3.7. Schematic diagram of sample preparation method to characterize sidewall roughness by AFM (a) before cleaving and (b) after cleaving	46
Fig. 4.1. Schematic diagram of (a) various types of roughness, LER, SWR and PFR, which are evolved during RIE (b) In LER, variation in x occurs along z; (c) in SWR, variation in x occurs within y-z plane; and (d) in PFR, variation in y occurs within x-z. The arrows indicate the height variations	49
Fig. 4.2 . 3D sidewall profile of the 15 μm width waveguide measured by AFM. The arrow indicates the scanning direction of AFM tip	51
Fig. 4.3. AFM figures with the variation of etching time at $t = 35$ min for 50 mT, 300W and 18 sccm O_2 flow rate in (i) 2D, showing hillocks and valleys and (ii) 3D, showing peaks and valleys	52
Fig 5.1. SEM micrographs at different RF powers (a) 225 W (b) 275 W and (c) 300W at 45 mT Pressure, 18 sccm O_2 flow rate and 35 min etching time	56
Fig. 5.2 Variation of vertical etch rate and sidewall roughness with RF power at 45 mT pressure, 18 sccm O_2 flow rate and 35 min etching time	57
Fig. 5.3. AFM images at different RF powers at (a) 225 W, (b) 275 W and (c) 300 W at 45 mT pressure, 18 sccm O_2 flow rate and 35 min etching time	58
Fig. 5.4. SEM micrographs at different pressures (a) 65 mT and (b) 95 mT at 300 W RF power, 18 sccm O_2 flow rate and 35 min etching time	61
Fig. 5.5 Variation of vertical etch rate and sidewall roughness with pressure at 300 W RF power, 18 sccm O_2 flow rate and 35 min etching time	62
Fig. 5.6 (a) Shadowing effect explanation at higher pressure. The number 1, 2 and 3 represents particles from different directions	63

Fig. 5.6 (b) Etching at the peaks due to shadowing effect. The sticking coefficient for shadowing effect, $S_S = 1$, whereas the sticking coefficient for reemission effect, $S_R = 0$. Etching occurs in downward directions indicated by an arrow. 63

Fig. 5.7(a) Reemission of the particles at low pressure due to normal incidence and (b) Etching at the valleys due to reemission which is responsible for the higher roughness. The number 1 and 2 represents particles from different directions 64

Fig. 5.8. AFM images at different pressures at (a) 65 mT with oxygen and (b) 95 mT with O_2 at 300 W RF power, 18 sccm O_2 flow rate and 35 min etching time 65

Fig. 6.1. 3D AFM images at (a) 225 W, (b) 275 W and (c) 300 W 69

Fig. 6.2. Variation of SWR along the vertical depth at different rf powers 70

Fig. 6.3 (a) (i) SEM micrograph of FPE waveguide and (ii) its sidewall, fabricated at 300 W rf power, 35 mT pressure and oxygen gas flow rate of 18 sccm. Different types of textures were obtained along the vertical depths of the sidewall 72

Fig 6.3 (b) (i) SEM micrograph of FPE waveguide (ii) textures at sidewall, and (iii) magnified trenches at the sidewall of waveguide fabricated at 300 W rf power, 95 mT pressure and oxygen gas flow rate of 18 sccm. Different types of textures were obtained along the vertical depths of the sidewall and evolved trenches are indicated by arrows 73

Fig 6.3 (c) (i) SEM micrograph of FPE waveguide (ii) textures at sidewall,

and (iii) magnified trenches at the sidewall of waveguide fabricated at 300 W rf power, 95 mT pressure and oxygen gas flow rate of 18 sccm. Different types of textures were obtained along the vertical depths of the sidewall and evolved trenches are indicated by arrows.	75
Fig 6.4. 3D AFM images of sidewall roughness of FPE waveguides at (i) 35 mT and (ii) 95 mT pressures	76
Fig. 6.5 Variation of SWR with vertical depths at various pressures	77
Fig. 6.6 (a) Schematic diagram of shadowing effect explanations for 35 and 65 mT pressure curves. Inset plot is an example for the shadowing effect regions for 35 mT pressure	82
Fig. 6.6 (b) Schematic diagram of first order reemission effect explanations for 35 mT pressure curve. Inset plot is an example for the first order reemission effect regions indicated by a vertical dashed line and an arrow for 35 mT pressure	83
Fig. 6.6 (c). Schematic diagram of CSE explanations for 95 mT pressure curve at different etch times, where, $t_1 < t_2 < t_3 < t_4$. Inset is an example for the CSE regions for 95 mT pressure	83
Fig. 6.7 (a-c) Variation of ACL with vertical depths at 35-95 mT. Variation of SWR at respective pressures shown with dotted curves is repeated for the comparison with ACL	85
Fig. 6.8. The log-log plot of HHCF and distance (r) for different vertical depths of for 35 mT pressure	87
Fig. 6.9. The log-log plot of HHCF and distance (r) for different vertical depths	

of for 65 mT pressure	87
Fig. 6.10. The log-log plot of HHCF and distance (r) for different vertical depths of for 95 mT pressure	88
Fig. 7.1 Variation of PFR with pressure	91
Fig. 7.2 Etching at the peaks due to shadowing effect. The sticking coefficient for shadowing effect, $S_S = 1$, whereas the sticking coefficient for reemission effect, $S_R = 0$. Etching occurs in downward directions indicated by an arrow	92
Fig. 7.3. AFM figures for 50 mT, 300W and 18 sccm O_2 flow rate and etching time, $t = 5$ min in (a) 2D and (b) showing the relatively flat surface having randomly distributed hillocks and valleys	93
Fig. 7.4. AFM figures for 65 mT, 300W and 18 sccm O_2 flow rate and etching time, $t = 5$ min in (a) 2D and (b) showing the relatively flat surface having randomly distributed hillocks and valleys	93
Fig. 7.5. AFM figures for 95 mT, 300W and 18 sccm O_2 flow rate and etching time, $t = 5$ min in (a) 2D and (b) showing the relatively flat surface having randomly distributed hillocks and valleys	94
Fig. 7.6. AFM figures with the variation of etching time from $t = 10$ min to $t = 35$ min for 50 mT, 300W and 18 sccm O_2 flow rate in (i) 2D, showing hillocks and valleys and (ii) 3D, showing peaks and valleys	98
Fig. 7.7. Variation of PFR with etching time at the pressure of 50 mT, 300 W rf power and oxygen flow rate of 18 sccm	99
Fig. 7.8. Variation of PFR with etching time at the pressure of 65 mT, 300 W	

rf power and oxygen flow rate of 18 sccm	99
Fig. 7.9. Variation of PFR with etching time at the pressure of 95 mT, 300 W	
rf power and oxygen flow rate of 18 sccm	100
Fig. 7.10. AFM figures with the variation of etching time from $t = 10$ min to $t = 35$ min for 95 mT, 300W and 18 sccm O_2 flow rate in (i) 2D, showing hillocks and valleys and (ii) 3D, showing peaks and valleys	103
Fig. 8.1 Schematic of simulation model	108
Fig. 8.2 Monte Carlo simulation illustration: incident particle bounce off the surface and etches the surface elsewhere	111
Fig. 8.3 Flow chart of Monte Carlo Simulation	113
Fig. 8.4. Simulation 1: SWR along vertical etch depth for $s_0 = 1$	116
Fig. 8.5. Simulation 1: x - y cross section at different vertical etch depth ($z = 50, 100, \text{ and } 150$) for $s_0 = 1$	116
Fig. 8.6. Simulation 2: SWR along vertical etch depth for $s_0 = 0.5$	117
Fig. 8.7. Simulation 2: xy cross section at different vertical etch depth ($z = 50, 100, \text{ and } 150$) for $s_0 = 0.5$	118
Fig. 8.8. Simulation 3: SWR along vertical etch depth for $s_0 = 0.2$	118
Fig. 8.9. Simulation 3: x - y cross section at different vertical etch depth ($z = 50, 100, \text{ and } 150$) for $s_0 = 0.2$	119
Fig. 8.10. Simulation 4: SWR along vertical etch depth for $s_0 = 0.05$	120
Fig. 8.11. Simulation 4: x - y cross section at different vertical etch depth ($z = 50, 100, \text{ and } 150$) for $s_0 = 0.05$	120
Fig. 8.12. Comparison of SWR along vertical etch depth of Simulations 1- 4	121

Fig. 8.13. Comparison of x - y cross section of Simulations 1- 4 at position $z = 100$	122
Fig. 8.14. Comparison of y - z cross section of Simulations 1-4 at position $x = 100$	123
Fig. 8.15. SWR dependence on etch depth for pure O_2 at low pressure (35 mT)	126
Fig. 8.16. Variation of fraction of ions with shadowing sticking coefficient, s_0 , where N_S , N_R and N_O are the number particle taking part in shadowing, reemission and neither shadowing nor reemission effects	128
Fig. 9.1. Pressure effect on SWR in O_2/N_2 plasma	131
Fig. 9.2. Comparison of SWR with variation of pressures in pure O_2 and mixture of O_2/N_2 plasma	132
Fig. 9.3. Dependence of SWR on etch depth for O_2/N_2 plasma at 50 mT to 95 mT. All samples are etched at 300 W, O_2 and N_2 flow rate of 18 sccm and 46 sccm respectively	133
Fig. 9.4. Negative ToF-SIMS surface spectra for pure O_2 samples at 95 mT, 300 W and 18 sccm O_2 flow rate	135
Fig. 9.5. Negative ToF-SIMS surface spectra for pure O_2/N_2 treated samples at 50 mT, 300 W, 18 sccm and 46 sccm O_2 flow rate	135
Fig. 9.6. Negative ToF-SIMS surface spectra for pure O_2/N_2 treated samples at 65 mT, 300 W, 18 sccm and 46 sccm O_2 flow rate	136
Fig. 9.7. Negative ToF-SIMS surface spectra for pure O_2/N_2 treated samples at 95 mT, 300 W, 18 sccm and 46 sccm O_2 flow rate	136
Fig. 9.8. Negative ToF-SIMS depth profile for pure O_2 samples at 95 mT, 300 W and 18 sccm O_2 flow rate	137
Fig. 9.9. Negative ToF-SIMS depth profile for pure O_2/N_2 samples at 50 mT,	

300 W, 18 sccm and 46 sccm O ₂ and N ₂ flow rate	138
Fig. 9.10. Negative ToF-SIMS depth profile for pure O ₂ /N ₂ samples at 65 mT,	
300 W, 18 sccm and 46 sccm O ₂ and N ₂ flow rate	139
Fig. 9.11. Negative ToF-SIMS depth profile for pure O ₂ /N ₂ samples at 95 mT,	
300 W, 18 sccm and 46 sccm O ₂ and N ₂ flow rate	140

List of Tables

Table Descriptions	Page No.
Table 2.1. Different materials used in making waveguides and their properties	10
Table 2. Various types of polymers used in optical waveguides	11
Table 3.1. Specifications of polymer materials	36
Table 3.2. Observed refractive index (R. I.) of ZP2145M and ZP1370M materials	38
Table 3.3 Variation of thickness at different speed measured by Prism coupling	
Method	42
Table 4.1. Summary of LER and SWR at various pressures at 300W R.F. power, 18 sccm O ₂ flow rate	50
Table 5.1. Variation of RF Power	59
Table 5.2. Variation of Pressure	61
Table 6.1. Comparison of roughness parameters	86
Table 9.1. Summary of RIE parameters in mixture O ₂ /N ₂ plasma	131

Acronyms

OCN	Optical communication networks
DWDM	Dense wavelength division multiplexing
OIC	Optical integrated circuits
TDM	Time division multiplexing
AWG	Arrayed waveguide grating
VOA	Variable optical attenuator
TIR	Total internal reflection
RIE	Reactive ion etching
SWR	Sidewall roughness
ACF	Autocovariance function
ACL	Autocorrelation length
PFR	Planar film roughness
LER	Line edge roughness
AFM	Atomic force microscopy
EDFA	Erbium doped fiber amplifier
TOC	Thermo-optic coefficient
PMMA	Polymethylmethacrylate
PCHS	Poly(cyclohexylsilyne)
PPSQ	Poly(phenylsilsequioxane)
TGA	Thermo-gravimetric analysis
BCB	Benzocyclobutene

RH	Relative humidity
NFP	Near field pattern
PC	Polycarbonates
ICP-RIE	Inductively coupled plasma- reactive ion etching
TEM	Transmission electron microscopy
CD	Critical dimensions
SEM	Scanning electron microscopy
SIMS	Secondary ion mass spectroscopy
PWG	Planar optical waveguides
PSD	Power spectral density
FPE	Fluorinated polyether
Si	Silicon
FESEM	Field emission scanning electron microscopy
Ga	Gallium
Ar	Argon
rms	Root mean square
sd	Standard deviation
rf	Radio frequency
HHCF	Height-height correlation function
CSE	Continuous shadowing effect

Chapter 1

Introduction

Bandwidth-hungry applications, explosive growth of internet, and data traffic are increasing the proliferation of optical communication networks (OCNs). Planar waveguides and waveguide-based devices for OCNs are attracting a great deal of attention since the evolution of dense wavelength division multiplexing (DWDM) system, which can carry multiples of signals in a single optical fiber. DWDM system is an important innovation in OCNs, which can increase the capacity of existing networks by transmitting many signals simultaneously in a single optical fiber. The performance of DWDM system has been boosted and driven by the advancement of the fiber optic components, optical integrated circuits (OICs), and advanced packaging technology. While all-optical technologies are pervasive in transmission lines, the nodes of the networks and cross-connections still depend upon the relatively slow electronic technologies. Due to limitations in the electronic circuitry, nodes in the networks limit the throughput. One solution to this problem is to migrate from electronic and, or, electro-optic nodes to all-optical nodes; hence, there is the need for multiplexing, demultiplexing, and cross-connection by optical technologies.

DWDM system is based on all-optical multiplexing and demultiplexing technologies without the need for electrical to optical and optical to electrical conversions as required in the time division multiplexing (TDM) systems.

Typical DWDM networks consist of transmitter (Tx), communication media, AWG (multiplexer / demultiplexer), amplifiers and receiver, (Rx). The schematic diagram

of DWDM optical network is shown in Fig.1.1 and the operational principle of DWDM system is as follows.

Signal Generation: Different wavelengths are transmitted from various transmitter modules, which provide stable light in narrow bandwidth, and carry the digital data.

Combining the Signals: In DWDM system, multiplexer (AWG) combines all such optical signals. Inherent loss associated with multiplexing or demultiplexing can be boosted up by the optical amplifiers.

Fig.1.1. Schematic Diagram of DWDM System

Transmission of Signals: The multiplexed signals are transmitted through the optical fiber. In DWDM system, each signal has its own dedicated bandwidth and all signals arrive at the same time, rather than being broken up and carried in different time slots, unlike in time division multiplexing (TDM).

Separation of the received Signals: At the receiving end, these signals are separated by the demultiplexer (AWG) and finally, each of the receivers (photodiodes) receives a different separated signal.

Many of the optical networking components including optical fiber in DWDM system operate by the principle of light confinement i.e. total internal reflection (TIR) or waveguiding in dielectric media like glass, polymer, sol-gel etc. There are several waveguide based components like AWG (multiplexer/demultiplexer), variable optical attenuator (VOA) and optical power splitters/combiners that are employed in optical networks.

Waveguide is a planar optical device, comprising lower cladding, core and upper cladding as shown in Fig. 1.2. Essential requirement of waveguiding is that the refractive index of core is higher than that of cladding in order to obey TIR principle. The details of the waveguides along with their issues are described in following paragraphs.

Fig 1.2. Cross-section of waveguide with various layers

1.1 Waveguides and their issues

Most of the planar optical waveguides in general and polymer waveguides in particular are conventionally fabricated by photolithography, and reactive ion etching (RIE) and the details of the fabrication steps are provided in chapter 3. Polymers are attracting a good deal of attention because of the properties such as ease of processing, and low cost fabrication process and matching of thermal stability, and photo-stability with silica. Since RIE is the common patterning technique for polymers [1], one important consequence of RIE in the fabrication process is the evolution of roughness [1] at various surfaces constituting a waveguide and details of which are discussed in Chapter 4 onwards. The roughness at these surfaces in general degrades the optical performance; however, sidewall roughness (SWR) in particular causes the scattering loss in the waveguides and could be the major source of optical loss in the waveguides [2] and deep-etched waveguides [3] because the increased interaction of the optical waveguide modes with the sidewalls could lead to significant scattering and optical loss [2]. Recently, it has been also reported that the sources of scattering loss could be the end face of the waveguides and rough planar surfaces of the waveguides along with the sidewall, but their contributions to the scattering loss are not significant [4]. Since the deep-etched waveguides allow strong lateral optical confinement [3], hence, by using ray optic approach [5], the number of reflections from the boundaries of the core will exacerbate the optical performance due to scattering from the sidewalls for non-ideal waveguides. The scattering loss will be further intensified for higher refractive index contrast Δn ($\Delta n = n_{\text{core}} - n_{\text{clad}}$) systems as scattering loss is proportional to $(\Delta n)^3$ [6]. Since in the deep-etched waveguides the curvature induced bending loss and leakage loss to the substrate are

negligible or can be reduced, the SWR for such structures completely determines the optical performances [3].

Both theoretical and experimental studies on scattering loss due to SWR have been conducted previously [2, 7-10]. Theoretical studies require experimental statistical information such as autocovariance function (ACF), standard deviation of roughness or root mean square (rms) roughness, and autocorrelation length (ACL) of SWR to model accurately optical losses due to sidewall scattering. Even though various characterization techniques have been attempted to measure the SWR [11], quantitative studies on SWR in order to obtain above parameters are still quite limited.

Therefore, the aim of this thesis is to study the nature of these roughnesses including SWR along with their evolutions during RIE, new means for their control, and the direct measurement of various types of roughnesses which have significant implications for better performance of optical devices.

1.2 Objectives

The objectives of the research work are as follows:

1. To directly measure various types of roughnesses such as planar film roughness (PFR), line edge roughness (LER) and SWR by developing the novel methodologies
2. To study the effect of process parameters on profile as well as SWR and PFR
3. To study the evolution of variation of SWR of the waveguides

4. To investigate new means of CONTROL of roughness

1.3 Scopes

The scope of the research work for each objective is as follows.

1. To directly measure various types of roughnesses
 - a. Establish experimental methodology to characterize PFR, LER and SWR by AFM
 - b. Calculate various statistical roughness parameters
2. To study the effect of process parameters on profile as well as SWR and PFR
 - a. Variation of RF power
 - b. Variation of pressure
3. To study the evolution of variation of SWR
 - a. Variation of pressure
 - b. Development of mechanisms and hypothesis
 - c. Monte Carlo Simulation studies
4. To investigate new means of CONTROL of roughness
 - a. Investigation of various process parameters
 - b. Optimization of process parameters
 - c. Development of novel recipes

1.4 Thesis organization

The thesis is organized in the following chapters. Chapter 2 describes the literature review on different types of materials used for the fabrication of cladded waveguides, experimental techniques to measure sidewall roughness, and study of different types

of roughness with their evolutions. Chapter 3 discusses experimental studies for cladded waveguides. Chapters 4 – 7 describe the experimental results and discussions. Studies of evolution of SWR by Monte Carlo simulation methods are described in Chapter 8. Control of roughness is described in Chapter 9 and finally, conclusions and future work are provided in Chapter 10.

Chapter 2

Literature Review

The concept of waveguides for optical communication has been developed for decades. Optical fiber, an example of optical waveguides, carries optical signals and is used for short and long distance optical communications. Today's most established waveguiding technology is glass fiber technology because of the advantages of Erbium doped fiber amplifier (EDFA) operating around 1550 nm wavelength in optical transmission windows. Both long and short distance optical communications have been boosted by the development of several passive devices such as multiplexers / demultiplexer, attenuators, filters, switches, couplers, and splitters. The basic building block in all these passive devices is the waveguide irrespective of its types (e.g. buried or ridged). This chapter reviews the current status of the types of materials available to fabricate waveguide (the basic building block for any complex structured devices), fabrication steps, one of them being reactive ion etching (RIE) and its consequences such as roughnesses at different interfaces, measurement of roughness, and various mechanisms developed so far to understand the evolution of these roughnesses. Finally, a summary is provided.

2.1 Materials for the waveguides

Materials for the waveguiding core have been realized by inorganic and organic materials. Inorganic materials such as doped silica (SiO_2) i.e. GeO_2 doped silica deposited by flame hydrolysis process at 1250 °C; amorphous silicon nitride and oxynitride; amorphous silicon; polycrystalline and crystalline silicon [12]; III-V

semiconductors such as InP and GaAs [13, 14]; metal oxides and oxide ceramics, have been used as the core materials for the waveguide fabricated for optical communication purposes. However, various types of organic (polymer) materials are also used as the waveguiding core materials because of their attractive properties.

2.1.1 Why polymer materials are so attractive for waveguides

The attractive features of polymers often quoted in the literature are as follows:

- (i) *Ease of processing*: These materials can be formed into waveguides by the techniques like spin coating, embossing, wet and dry etching, injection molding [15-16].
- (ii) *Thermo-optic coefficient (TOC)*: Polymers possess large TOC ($-1 \sim -4 \times 10^{-4}/^{\circ}\text{C}$) which is more than ten times greater than that of their inorganic counterparts. Therefore, the transmission peaks of waveguide based device such as polymeric AWG can be tuned over large wavelength [15-19].
- (iii) *Optical loss*: In comparison to silica based system, low optical loss is also obtained from polymer waveguides [16-18].
- (iv) *Thermal and environmental stability*: They possess good thermal and environmental stability [18-19].
- (v) *Refractive index (RI)*: By blending and copolymerizing, tunable RI contrast between core and cladding of polymers can be obtained [20].
- (vi) *Birefringence*: Polymer-based waveguides renders low birefringence, which is comparable to silica based system [15-17, 21-22].

(vii) *Adhesion*: Polymers provide good adhesion to the substrates such as silicon [15-17].

In addition to the above properties, good processability of polymer allows fabrication of waveguides on different substrates like InP, Cu-polyimide and plastic substrates besides silicon substrate [23-24], and polymeric waveguides can also be realized on free standing films [23]. The properties of various types of materials used for waveguide fabrication are summarized in Table 2.1 below.

Table 2.1. Different materials used in making waveguides and their properties [17]

Materials	Propagation loss dB/cm	Refractive Index (n)	Refractive Index contrast (Δn) in waveguide	Birefringence
Silica (SiO ₂)	0.1	1.5	0-1.5% (Channel)	10^{-4} - 10^{-2}
Silicon (Si)	0.1	3.5	70% (SOI Rib)	10^{-4} - 10^{-2}
Silicon Oxynitride (SiO _x N _y)	0.1	SiO ₂ : 1.5 Si ₃ N ₄ : 2.0	0-30% [30% Si ₃ N ₄ core] (Channel, SiO ₂ Cladding)	10^{-3} - 5×10^{-6}
Sol-Gels	0.1	1.2-1.5	0-1.5% (Channel)	10^{-4} - 10^{-2}
Polymers	0.1	1.3-1.7	0-35% (Channel)	10^{-6} - 10^{-2}

2.1.2 Polymers for waveguides

Several polymers have been reported in the literature for fabricating waveguides. Some component manufacturers such as NTT have started supplying polymeric devices. A summarized version of various available polymers and their properties are tabulated in Table 2.2 and are described in the next section.

Table 2.2. Various types of polymers used in optical waveguides [16]

Manufacturer	Polymer Type (Trade-name)	Patterning Tech.	Optical Loss in dB/cm (wavelength , nm)	Other Properties
Allied- Signal	Acrylate	Wet etching, RIE, Photo exposure, Laser Ablation	0.2 (840) 0.2 (1300) 0.5 (1550)	Birefringence (2×10^{-4}) (1550) Cross linked, T_g : 25° C Environmentally Stable
Amoco	Fluorinated Polyimide (FPI), (Ultradel ™)	Wet etching, Photo exposure	0.04 (1300) 1.0 (1550)	Birefringence (2.5×10^{-2}) Cross linked, Thermally Stable
Dow Chemical	BCB (Cylotene™)	BCB (Cylotene™)	0.8 (1300) 1.5 (1550)	$T_g > 350^\circ \text{C}$
	PFCB (XU35121)	Wet etching, Photo exposure	0.25 (1300) 0.25 (1550)	$T_g : 450^\circ \text{C}$
Dupont	Acrylate (Polyguide™)	Photolocking	0.18 (800) 0.2 (1300) 0.6 (1550)	Laminated Sheets Excimer laser machinable
General Electric	Polyetherimide	RIE, Laser ablation	0.24 (830)	Thermally Stable
Hoechst Celanese	PMMA Copolymer (P2ANS)	Photobleaching	1.0 (1300)	NLO Polymer
JDS Uniphase Photonics	(Beam Box™)	RIE	0.6 (1550)	Thermally stable

NTT	Halogenated acrylate	RIE	0.02 (830) 0.07 (1310) 1.7 (1550)	Birefringence (1×10^{-6}) (1310) T_g : 110° C
	Deuterated Polysiloxane	RIE	0.17 (1310) 0.43 (1550)	0.17 (1310) 0.43 (1550)
	Fluorinated Polyimide	RIE	TE: 0.3, TM: 0.7 (1310)	Birefringence: 0.008-0.009 & Environmentally Stable
ZEN Photonics	Fluorinated Polyethers	RIE (Plasma Etching)	Optical Loss: 0.2 (1550)	Thermally and Mechanically stable Low Moisture absorption

(a) Acrylates

Acrylate monomers are esters having vinyl groups, which contain two carbon atoms doubly bonded to each other, and directly attached to carbonyl carbon atom. Acrylate polymers have been used to fabricate compact and low-cost waveguides and waveguide based devices for low loss optoelectronic applications [25-27] and they are optically stable at 125 °C. By blending with co-monomers, refractive indices have been controlled. The smallest dimensions of core-layer of ridge waveguide achieved by this material is $3 \times 3 \mu\text{m}^2$. Refractive index contrast for this material between core and cladding is 0.8-2% [26]. The optical losses at 840, 1300 and 1550 nm are 0.02, 0.2 and 2 dB/cm respectively [27]. Fluorinated acrylates, which further reduce the absorption loss due to the suppression of intrinsic vibrational absorption owing to replacement of C-H bonds by C-F bonds, are also available.

Polymethylmethacrylate (PMMA) is also an acrylate polymer having an extra methyl group attached to α carbon atom. It is a well known photoresist which is easily patterned by photo exposure, and chemical or reactive ion etching (RIE). With proper cladding material, PMMA can be used as core material, which is patterned by the above techniques. Sidewall roughness of the PMMA channel waveguides was minimized by using UV laser writing process at 257 nm with the fluence of 300 mJ/cm² and scan rate of 16 mm/min [28-29]. The channel waveguide having the crosssection of $3 \times 7 \mu\text{m}^2$ exhibited a loss of 0.08 dB/cm at the wavelength of 633 nm [29].

(b) Polysiloxanes

In contrast to other polymer backbones, the backbone in polysiloxanes is silicon based. Poly(cyclohexylsilylene) (PCHS) is a Si-backbone polymer, which has the refractive index of 1.51 at 632.8 nm [30] and it is a UV curable polymer. Using the exposure energy of 7.2 mJ/cm² at 310 nm, waveguides can be fabricated following a negative masking process. Propagation loss of 0.7 dB/cm has been reported for the waveguide crosssection of $0.5 \times 4 \mu\text{m}^2$. Thin film waveguides from poly(phenylsilsequioxane) (PPSQ) have also been fabricated and characterized. PPSQ is thermally curable at 125 °C and possesses the refractive index of 1.55 at 632.8 nm and the propagation loss of 1.7 dB/cm for 1.3 μm thick film. This film also exhibits good thermal and chemical stability for the temperatures up to 400 °C [19].

Considering the important aspects of optical waveguiding another type of material called deuterated polysiloxane has been synthesized and it possesses two distinct properties. First, C-C bonds were replaced by polysiloxane backbone (-Si-O) and secondly, adoption of deuterated substituent in the phenyl group (-C₆D₅) provided lower absorption at both 1.3 and 1.55 μm wavelengths [19], which means by substituting deuterium for hydrogen, the vibrational absorption peaks were shifted towards the longer wavelength as shown in Fig 2.1. Hence, material absorption loss was suppressed at both the communication wavelengths, 1.3 and 1.55 μm . Due to large molecular refraction of phenyl group, refractive index of this polymer was controlled by controlling the contents of phenyl group. Refractive index increment of 0.0046 was obtained with 8 mol % increase in phenyl group content and hence, a refractive index contrast (Δn) between core and cladding polymers can be created [31]. This polymeric material was also thermally stable, as verified by thermogravimetric analysis (TGA) [19]. The degradation temperature of polysiloxane was about 400 °C, which is superior to poly(methylmethacrylate) (PMMA); degradation of the latter was 150 °C. Fabrication of passive optical waveguides has also been carried out. The propagation loss at both the wavelengths 1.31 and 1.55 μm measured by cut-back method were 0.17 and 0.41 dB/cm respectively, while the propagation loss at 1.55 μm was considerable less than that of deuterated PMMA at 1.55 μm .

Fig. 2.1. Absorption spectra of deuterated phenylsilyl chloride ($C_6D_5SiCl_3$) monomer and normal phenylsilyl chloride ($C_6H_5SiCl_3$) [19]

Benzocyclobutene (BCB) is another kind of siloxane material from which single mode optical waveguides have been fabricated for optical interconnects applications for very short distance data communications. It also possesses important properties like low moisture uptake (<2.3 wt. % after 24 hours in boiling water), good adhesion to various substrates and itself, and thermal stability greater than 350 °C [32]. Both photo-definable and non-photo-definable versions of BCB are available. The photo-definable version of BCB polymeric waveguide can be fabricated without the need of plasma or reactive ion etching (RIE), which possesses the refractive index of 1.562 at the wavelength of 589 nm, and used as a core material for waveguides. The non-photo-definable BCB polymer having the refractive index of 1.559 at 589 nm was used as cladding materials. The fabrication of waveguides includes many steps and one of them was the curing of polymer after spin coating into Si-wafer. The aim of curing was to produce a film to be solvent resistant and to provide adhesion to the next layer of BCB [32]. Curing was carried out in N_2 atmosphere (<100 ppm O_2) in order to avoid oxidation of polymer. Oxidized films proclaimed the rise of refractive index approximately by 0.025 [32-

34] and hence, the physical properties were degraded and the propagation loss at 1300 nm measured by cutback method was 0.81 dB/cm.

(c) Polyimides

Polyimides are also attractive materials for the fabrication of optical waveguides which contain imide group in the polymer chains. They exist in two different forms. The first one is a linear structure where the atoms in the imide group are part of a linear chain, and the second one is a heterocyclic structure where the

Fig. 2.2. Optical loss dependence on the exposure time to 85% relative humidity at 85 °C for waveguides on silicon substrate [35]

imide group is a part of a cyclic unit in the polymer chain [36]. They also possess high thermal stability up to 300 °C [35, 37-39]. The optical loss was higher in case of polyimide and was limited to the wavelength of 0.83 μm and the loss at this wavelength was 0.3 dB/cm. However, by replacing hydrogen with fluorine, the fluorinated polyimide gained optical transparency at the telecommunication wavelengths of 1.3 and 1.55 μm. In addition to the above property, water absorption of fluorinated polyimides was between 0.2 % and 0.7 % which was less than ~2 % that of nonfluorinated polyimides [35]. In order to stabilize the optical loss of

waveguides in the humid conditions, low water absorption condition criterion was maintained. Optical losses at the wavelengths 1.3 and 1.55 μm increased as the intensity of O-H absorption peak increased at 1.44 μm . The moisture resistance of polyimide was evaluated by measuring the optical loss at room temperature after exposure of several hours in 85 °C and 85 % relative humidity (RH) as shown in Fig. 2.2 (page 16). The variation of optical loss was 0.05 dB/cm for both TE and TM modes after exposure to 85 % RH at 85 °C for 200 hours. The polarization dependent loss (PDL) which is the difference in losses between the TE and TM modes was constant at 0.4 dB/cm (calculated from above figure). These waveguides continued to operate in single mode (which can be understood from the Gaussian distribution confirmed from near field pattern (NFP)) after exposure to humid conditions. Therefore, this study suggests that fluorinated polyimide possesses high moisture resistance confirming low water absorption.

(d) Polycarbonates

Polycarbonates (PC) contain carbonate groups in the backbone chain. Planar waveguides have been fabricated using dip coating in a bisphenol-A polycarbonate solution in dioxane and subsequent curing at the temperature of 120 °C [40]. PC films of 2 – 3.5 μm thickness showed the propagation loss of < 2 dB/cm and possessed a refractive index of 1.59 at 633 nm. PCs, synthesized from bisphenol-Z (PCZ), waveguides showed a propagation loss of 0.78 dB/cm for the core of $5.3 \times 8 \mu\text{m}^2$ crosssection and a refractive index of 1.55 at 1310 nm.

(e) Ethers

For the fabrication of waveguides, crosslinked polymer has advantages like higher thermal stability, chemical resistance, gap-filling ability, and improved adhesion properties to the substrate [41]. Acetylene-terminated prepolymers are good candidates of crosslinked polymers for optical waveguides because of the absence of aliphatic C-H bond which leads to significant absorption loss, absence of catalyst, and evolution of volatile substances during curing [41]. Fluorinated poly(arylene ethers) with phenylethynyl group (FPAE-PEP) at the chain end is one such candidate of crosslinked polymers. The chemical structure of this polymer is shown in Fig. 2.3 [41].

Fig. 2.3. Chemical structure of crosslinkable fluorinated poly (arylene ethers) with phenylethynyl group (FPAE-PEP) [41]

By substituting hydrogen with fluorine, FPAE-PEP exhibits good optical transparency at 1.55 μm with optical loss of 0.2 dB/cm. It has high thermal stability of up to 510 $^{\circ}\text{C}$ and chemical resistance for organic solvents. In FPAE structure, 60 % of C-H bonds have been replaced by C-F bonds. It also exhibits low absorption loss [42-43]. Refractive index of the polymer was controlled by altering the feeding ratio of the co-monomers or blending ratio between the copolymers [44]. In this way, the core and cladding refractive index can be selected appropriately in a range. Thermal stability, which is an important issue, is closely related to the decomposition

of the polymers, whose decomposition or degradation temperature is defined as the temperature at which the weight loss is 5 %. TGA thermograms show that these polymers exhibit 5 % weight loss at ~ 500 °C. Thermal stability is also verified by noticing the variation of refractive index at elevated temperatures as shown in Fig. 2.4. Both 1×16 and 16×16 polymeric arrayed waveguide grating multiplexers, the waveguide based devices which are used in DWDM system and functioned as multi/demultiplexer, were fabricated using FPAE with 100 GHz (0.8 nm) channel spacing [43-44].

Fig. 2.4. Refractive Index stability of FPAE at 120 °C [44]

2.1.3 Absorption loss in polymers

Generally, polymers, in contrast to inorganic materials, exhibit high optical loss (before replacing hydrogen from polymer chain) in the second (1310 nm) and third telecommunication transmission windows (1550 nm) due to the substantial C-H bond vibrational absorption. Optical absorption in polymeric materials is determined both by the energies of molecular or polymeric electronic excited states, and by the locations of fundamental and overtone vibrational excited states of covalently

bonded nuclei like carbon and hydrogen [16]. The location of these vibrational states is also largely determined by the 'spring constant' of the bond k , and reduced mass, μ of two nuclei. The reduced mass μ is expressed as

$$\frac{1}{\mu} = \frac{1}{m_1} + \frac{1}{m_2}, \quad (2.1)$$

where m_1 and m_2 are the masses of two nuclei. The vibrational transition of these two nuclei can be modeled as a harmonic oscillator and the frequency ν of this oscillator is expressed as

$$\nu = \frac{1}{2\pi} \sqrt{\frac{k}{\mu}} \quad (2.2)$$

Hence the vibrational energy,

$$E(\nu) = \left(\nu + \frac{1}{2}\right)h\nu \quad (2.3)$$

is inversely proportional to the reduced mass which indicates that bonds with low reduced masses such as C-H, vibrate with high frequencies extending well into the infrared communication windows (in wavelength unit, it ranges from 1 to 2 μm). Hence, this vibrational absorption energy can greatly be minimized by replacing C-H bonds by C-F bonds [18] or by C-Cl bonds [16] or by introducing deuterium in the place of -H [18-19, 45]. Thus, fundamental and overtone vibrational absorption are virtually eliminated from 1 to 2 μm . Absorption strengths of different molecular bonds are shown in Fig 2.5 in terms of calculated absorption loss vs. wavelength for the transition from ground vibrational state to excited vibrational state. Replacement of C-H bond with C-F, C-Cl or C-D bonds reduces intrinsic absorption. However, synthesis of polymers with C-D bonds is difficult and expensive. Using this

principle, the absorption losses in polymers are greatly reduced. Many fluorinated polymers are commercially available in the market with very low losses, which are comparable to silica and the properties of them are provided in Table 2.2.

Fig. 2.5. Calculated absorption losses for various vibrational motions [16]

2.2 Fabrication of waveguides

Realization of inorganic or organic microphotonic devices based on optical waveguides has been enabled because of the recent progress in dry etching technologies [1-3, 10, 46]. Most of the polymeric optical devices like waveguides are fabricated by a conventional process, which includes the following steps like spin coating, photolithography process and reactive ion etching (RIE). The details of the fabrication process are described in Chapter 3. RIE is a commonly used patterning technique for the fabrication of polymeric waveguides and waveguide based devices [1]. One important consequence of RIE in the fabrication process is the evolution of roughness [1] at different surfaces. During this fabrication process roughness is

introduced in the device and the details of which are described in the following sections.

2.2.1 Evolution of roughnesses during dry etching

Dry etching techniques to fabricate optical waveguides are RIE, reactive ion beam etching (RIBE), and inductively coupled plasma- reactive ion etching (ICP-RIE) [3-47]. Roughening of plasma etch front is the consequence of dry etching. It is reported that under non-equilibrium conditions etch fronts can exhibit different degree of roughness depending on the processing conditions such as temperature, pressure, etch rate and local environment [48]. Roughness evolution during growth has been widely studied. However, studies on roughness evolution during plasma etching of polymers are still quite limited. Various types of roughness at different interfaces of the waveguide are evolved during RIE, a commonly patterning technique to etch polymer. The roughness at the interfaces includes planar film roughness (PFR), sidewall roughness (SWR), and line edge roughness (LER). A description of these types of roughnesses evolved during RIE is described in Chapter 5.

The rough surfaces are described by three basic roughness parameters namely σ , ξ and the roughness exponent, α . The rms roughness σ represents the surface height fluctuations in vertical direction; however, the ACL, ξ , and roughness exponent, α represent the fluctuations in lateral directions. The ACL, ξ is the distance within which the surface heights of any two points are correlated. The value of α is within 0, and 1 and α describes how wiggly the surface is. In other words, the higher

α is, the smoother the surface becomes, and the lower α represents the more jagged surface.

2.2.1.1 Evolution of planar film roughness

This sub-section describes the plasma etch front roughening of planar films for both inorganic and organic materials. The roughness evolution of Si (100) was studied [49]. Si surfaces were etched using CF_4 and O_2 mixture of gases at different etch times. The surface morphology during the beginning of etching consists of randomly distributed holes and hillocks. However, at the higher etch times, the holes coarsen together to form bigger holes and the surface morphologies possess self-similar structure in time-scale. It was reported that the rms roughness (σ) and the autocorrelation length (ACL) (ξ) increases with etch time (t).

Roughness evolution of planar polymer film has been studied for shallow etch depth [46]. It has been reported that the rms roughness increases linearly with the increase of etch depth. The ACL, ξ does not show any noticeable change in their experimental results. *The coarsening effect suggested by Zhao et al.[49] and described in the above paragraph, was not observed for the polymer materials as the etch depth increases. In other words, as the etching continues, the reacting flux do not create any self-similar structure with the etch time.* This is an important dissimilarity observed for polymer materials. In this research we also extend this piece of work and the experiments and results are discussed in Chapter 7. Therefore, two possible reasons are suggested for this discrepancy. First, the experimental result measured by Agarwal et al. [1] may not have entered the dynamic scaling regimes

for the *shallow etched structures* (the structure having thickness less than 1500 nm). Second, *the lateral scale is controlled by the factors other than arrival statistics and etchant redistribution*. It is also reported that at low pressure the flux is normal to the surface morphology and is oblique to morphology at high pressure. It has been experimentally observed that the high pressure etched thin films provide smoother surface compared to low pressure etched film.

2.2.1.2 Evolution of sidewall roughness of polymer waveguides

As has been described earlier, the consequence of RIE in micro- and optoelectronic devices is the evolution of roughness at different interfaces [1]. Evolution of sidewall roughness (SWR) of patterned structure such as waveguide is rarely studied. Pressure is the most dominant factor affecting the SWR. As opposed to the arrival dynamics of etchants to the planar films, it has been suggested that at low pressure, the reactant flux are more oblique to the sidewall and at high pressure, the reactant flux are more normal to the sidewall. Hence, due to shadowing effect the SWR at low pressure is lower and due to reemission, the SWR is higher at the high pressure for shallow etched structures. It has also been reported that with the increase of pressure, SWR increases, which was *indirectly proved by measuring the propagation loss*. Therefore, no studies on the evolution of roughness for deep etched structures have been carried out so far.

2.2.2 Measurement of SWR and LER

It has been discussed in the previous paragraphs that SWR is the major source of optical loss and particularly, it is the major cause of scattering loss. Both

theoretical and experimental studies on scattering loss due to SWR have been conducted previously [2, 7-10]. Theoretical studies require experimental statistical information such as autocovariance function (ACF), standard deviation of roughness or rms roughness and autocorrelation length (ACL) of SWR to model accurately the optical losses due to sidewall scattering. Thus, in order to achieve a low loss optical waveguide an appropriate characterization tool to measure SWR and etching technology to produce vertical and smooth sidewall are required [11]. This subsection will describe various techniques approached so far to obtain quantitative information of SWR.

Transmission electron microscopy (TEM) has also been employed to measure the interface line-edge roughness (LER) between the core and the cladding layers of Si/SiO₂ optical waveguides [2]. The auto-correlation length (ACL) as well as rms roughness of LER was obtained. It is important to note that the authors assumed that the SWR was identical to the LER of the top surface; however, this may not be true for the deep-etched waveguides. In other words, SWR, which is assumed to be constant through out the sidewall for theoretical and experimental works, is not constant for the deep etched structures.

Smaller features in semiconductor devices make it imperative to control the sidewall, line and line-edge roughness, which contributes to the variation in critical dimensions (CDs). For smaller dimension improvement, CD is a major challenge. Line-edge roughness is one of the important factors that affect the CD control. Atomic force microscopy (AFM) alone or AFM and scanning electron microscopes

(SEMs) can be used to study line-edge roughness due to wet etch process and edge roughness on mask features [50]. Another important factor, which affects the performance of optical waveguides, is the sidewall roughness [50].

To measure the line-edge roughness, line-edge was scanned with a conical tip and with a flared tip to obtain a low frequency line edge roughness [51]. A flared tip was used for scanning in such a way that the device to be scanned was rotated at an angle to the flared tip and scanning was carried out from one of the corners of the tip. Roughness of a line-edge was measured perpendicular to the scanning direction at various z-positions along the sidewall.

Various techniques have been utilized to measure the SWR quantitatively. However, it was difficult to measure the roughness at the sidewalls of vertical three-dimensional structures having small dimensions, such as waveguides [11], due to two reasons: the difficulty to accurately determine the measuring position, and the shape of the waveguides that prohibits the AFM tip to directly access the sidewalls of the waveguides [52]. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) are commonly used techniques to measure planar surface morphology or texture of the surface [11]. SEM-based measurement technique using electron probe roughness analyzer has been utilized to obtain the roughness of an etched sidewall profile. This technique was advantageous as it is non-contact measurement technique and sample damage can be prevented; however, its resolution was limited to 1 nm [53]. AFM is well suited for the measurement of surface roughness in the nanometer scale and is better than SEM, but it is difficult to use to measure the vertical surface

of three-dimensional (3-D) structures due to the reasons above. Various approaches, including modification in the AFM system, have been attempted to overcome these difficulties. A specially prepared high aspect ratio boot-shaped AFM tip which was mounted in an AFM system with two-dimensional (2-D) servo control system has been demonstrated for this purpose [54]. In another measurement technique, AFM system was used to characterize the sidewall profile of a photoresist line, but the scanning area was limited by the length of the AFM tip. The positional stage controlling the lateral motion of the AFM tips limited the resolution of the scanned image. Hosomi *et al.*[52] obtained the sidewall profile of ridge waveguide structures using AFM and SEM by mounting the sample at a tilted angle [52]. However, it is very difficult to obtain the roughness information quantitatively for deep-etched optical waveguides with highly vertical sidewalls. Recently, direct measurement of SWR was carried out for InGaAsP/InP heterostructure systems in which a staircase pattern was used to assist characterization [11]. Therefore, in order to directly measure the SWR, either a modification of the AFM-system has to be followed or a specially designed structure has to be used. Hence, no studies on evolution of variation of roughness for shallow or deep etched structures have been carried out so far.

2.2.3 Analytical approaches of sidewall roughness studies

Attempts have been made to understand the effect of imperfection caused during the fabrication process on the transmission of optical signals. During the

propagation of these signals within the waveguides, it was assumed that the lowest order guided mode carried most of the power. The power loss of this lowest order guided mode was due to conversion of guided mode to spurious guided modes, as well as to the conversion of guided mode to radiation mode, the unguided mode [7]. The spurious guided mode occurs because of the scattering either in a forward or backward direction inside the core layer of waveguide and the radiation mode goes out of the waveguide. The theoretical development for understanding the conversion of modes was carried out by considering a sinusoidal variation of the thickness of the waveguide and the variation of width of the waveguide [7]. The imperfection at the sidewall was viewed from the top surface whereas the variation of the width is assumed to be the same as that of the sidewall. Because of these sinusoidal variations discussed above, the power loss due to coupling between guided modes to unguided mode appeared to be the true loss, since the coupling between guided and spurious guided modes leads to the conservation of energy, which means that the loss of energy in one of the guided modes is the gain in energy in the other guided mode [7]. By statistical treatment of the above approach it was found that the total power dissipated to the lowest order guided modes was the sum of the losses to the radiation modes and the spurious guided modes. With the increasing number of guided modes supported by the waveguide, the losses to both the guided modes and unguided modes increased. The power scattered into the radiation mode escaped so rapidly that no appreciable amount of power re-conversion took place from radiation mode to the guided modes. The conversion of power and coupling of different types of modes in the vicinity and inside of core layer (near field) and outside of waveguide (far field) led to mathematical complexity.

In order to avoid the mathematical complexity, a simpler approach was followed in which the far field effect has been described for random wall imperfections. In the theoretical development to express the loss caused by scattering from the sidewall, waveguide was treated as a radiating antenna and random sidewall imperfections were treated as an equivalent current source, the source of scattering (radiation) [9, 55]. A two-dimensional roughness analysis was carried out by considering the imperfections at the interfaces between core and cladding. The vertical deviations of the interfaces from their mean positions were considered. The interface described here was the top surface of the core layer, which had the average width $2d$. It was assumed that the vertical deviation at the interfaces was the same through-out the sidewalls. A two-dimensional expression for the radiated field, which carries power away from the waveguide into the space, was carried out, and from which power loss was calculated. The expressions for attenuation constant were evaluated by considering both forms of autocorrelation function (ACF), namely exponential and Gaussian [8-9]. ACF describes the distribution of height variation along the surface and it can be defined as the extent to which the knowledge of height at one point on the surface on an average determines the height at some other point, 'u' away. The two forms of ACF, the exponential and the Gaussian [56] are described respectively as, $C(u) = \sigma^2 \exp(-\frac{|u|}{\xi})$ and

$$C(u) = \sigma^2 \exp(-\frac{u^2}{\xi^2}) \quad (2.6)$$

where σ is the root mean square (rms) deviation of roughness, and ξ is the ACL.

ACL is defined as the shortest distance at which the value of ACF drops to a fraction,

usually 10 % or $1/e$ of its zero shift value [57-59]. The zero shift value of ACF, $C(u)$ is σ^2 .

Theoretical treatments of waveguide scattering problem due to surface roughness are limited to two-dimensional geometry as described above. Attempts have been made for three-dimensional analyses of scattering of optical signals due to surface roughness. In an attempt to describe the three-dimensional rough surface, one of the surfaces was considered as smooth while another surface was considered as rough [60]. The analysis of roughness was carried out by applying boundary conditions. It has been mentioned that the power scattered per unit solid angle into the two different planes was different [60].

It has been considered that the fundamental mode always carries most of the optical power than any other possible modes that propagate inside the waveguides. The higher modes that existed in the waveguide also dissipated power due to scattering and one such mode was called substrate radiation mode. This mode was confined in the upper cladding of refractive index n_1 (cover layer) and varied sinusoidally in the lower cladding (buffer layer) of refractive index n_3 , where $n_3 > n_1$ for asymmetric waveguides [5]. It is noteworthy to mention that the refractive index of core layer was n_2 and the order of refractive index was $n_2 > n_3 > n_1$ for the electromagnetic waves to be guided in the core. In an attempt to describe the scattering phenomena in three-dimension, scattering of guided mode from a straight rough rib waveguide into the substrate radiation modes has been considered. The analysis was limited to a very small rib height [61]. The theoretical analysis also

considers both the near field and far field distribution of the electric field as described previously. The roughness of the sidewall was represented as regular sinusoidal corrugation which is practically impossible to obtain by fabrication process. It is an exceptional case to be considered [7]. Also, the conversion of guided mode to substrate radiation mode invites complications in the theoretical formulation.

To describe scattering loss in three-dimension due to sidewall roughness, another analytical approach was followed in which the edge roughness was assumed to be the same as that of the sidewall roughness. This was the Paney-Lacey [8] assumption in two dimensions. In order to consider it for the three-dimensional case, the effective refractive index was calculated and used in the expression for attenuation coefficient to predict scattering loss. Two assumptions were considered in the approach: (a) the top and bottom surface of the waveguide was atomically smooth and (b) the roughness in the sidewall was assumed to be constant vertically [2]. The height under consideration was 200 nm and width was varied from 0.5 μm to 8 μm , which is the dimension of single mode waveguide.

Planar optical waveguides (PWG) which serve as the basis of the various optical integrated circuits (OIC) need a low level of optical losses of intensity of guided modes due to scattering at rough, irregular boundaries and heterogeneities of waveguide layers [61]. Two main approaches were followed to explain the phenomenon of light scattering in waveguides. The first one considered the general theory of irregular planar optical waveguides and peculiarity of process of intermode

waveguide scattering (mode conversion- Marcuse's approach [7]). The second one includes the phenomenon of waveguide scattering for the investigation of irregular properties and determination of their characteristics and parameters [62]. Radiation of scattered light in a PWG was of two types. The first one was the out-of-plane scattering where substrate modes and substrate-cover modes were included and the second one was the scattering in the plane of waveguide, which was also called in-plane scattering. Both of these cases required a correct solution of direct and inverse scattering problems. Far zone (far field) scattering problem was considered for the solution of direct problem in terms of scattered intensity. The waveguide of consideration was a symmetrical PWG with upper and lower cladding of refractive index 1.43 ($n_{upper\ cladding} = n_{lower\ cladding}$) of quartz material and a liquid core layer of refractive index 1.59. Excitation of fundamental TE-mode was considered in symmetric PWG. Secondly, in the solution of inverse problem statistical irregularities, which consisted of parameters like ACF and power spectral density (PSD), were reconstructed for far zone analysis. Gaussian PSD of the statistical roughness of quartz surface (upper and lower cladding) was considered. Gaussian ACF, which is the Fourier transform of PSD, was chosen.

But in real cases, the top and bottom surfaces are not atomically smooth and the sidewall roughness, which is created during the fabrication process, is not constant in vertical direction. It depends upon the process parameters like pressure [16], RF power and gas flow, angle of inclination of reacting particles [16] and photoresist induced micromasking [63-64].

Resist-induced micromasking resulted during RIE process in which resist was used as a mask. The mechanism of micromasking was that during O_2 plasma etching, and some of the byproducts were deposited after reaction with reacting particles and the polymer. These byproducts once deposited remained in the sidewall [65] and acted as a micromask. This phenomenon is called micromasking. The parameters that could be helpful for cleaning the micromasking were the higher radio frequency power and the time for 5-10 % over-etching.

2.3 Summary

This section summarizes the previous works. In one of the experimental studies, it was assumed that SWR is the replica of the mask edge roughness [10]. Another experimental study equated SWR with the LER between the core-cladding layers (top surface) [2]. Theoretical models [7-10] developed for scattering losses so far have only treated the SWR as being constant throughout the sidewall, with a value equivalent to LER. But LER of the top surface is not the same as that of SWR for deep etched structures [11], even though both LER and SWR consists of same variation of x (Fig. 4.1, Chapter 4). Deep etched waveguides allow strong lateral optical confinement [3]. Hence, according to the ray optic approach [5], the number of reflections from the boundaries of the core will be higher for higher refractive index contrast, Δn ($\Delta n = n_{\text{core}} - n_{\text{clad}}$) systems. This situation will be further intensified for higher Δn systems as scattering loss from the sidewalls of non-ideal waveguides is proportional to $(\Delta n)^3$ [6]. Furthermore, in the deep-etched waveguides, the curvature induced bending loss and leakage losses to the substrate are negligible or can be reduced [3]. In such systems, the SWR may completely determine the

optical performances [3]. Hence, understanding of evolution of SWR will have severe implications on the optical performance of such waveguides. Recently, it has been reported that the angle of incidence of the reacting particles in the plasma for a planar film is different from that of the sidewalls [1]. Therefore, it is reasonable to expect that the SWR is neither the replica of mask edge roughness nor the constant along the depth of a waveguide, and its variation should depend on etching parameters. Furthermore, the discrepancies of *self similar structures* with etching time for planar film evolutions are also found for shallow etched structures between inorganic and organic materials. So far, no studies on PFR for deep etched structures and the variation of SWR have been carried for cladded waveguides and no particular (unique) techniques to directly measure the SWR and LER have been reported.

Chapter 3

Experimental Studies

Fluorinated polyether (FPE) was used as a polymer material for the fabrication and characterization of the cladded waveguides in this research work. The ZP2145M and ZP1370M, procured from Zen Photonics Co. Ltd, were used as the cladding and core and have the refractive indices of 1.4840 and 1.5479 respectively. Adhesion promoter, ZAP1010 also procured from the above company was used for achieving sufficient adhesion to substrates. The details of the experimental procedures for studying materials properties, cladded waveguide fabrications, and surface characterizations are described below.

3.1 Materials characterization for cladded waveguide

In this section, curing optimization as measure of refractive index of polymer materials has been described followed by moisture absorption, and thickness measurement of planar films. Using these optimized parameters, cladded waveguides were fabricated for process studies. Surface characterizations of etched films by using scanning electron microscopy (SEM), AFM and secondary ion mass spectroscopy (SIMS) are also been described.

3.1.1 Refractive index measurement (Curing Optimization)

In this research work, a thin layer (about 1000 Å) of SiO₂ with refractive index (R. I.) of 1.46 was deposited by wet oxidation method on a six inch silicon (Si) wafer, which acted as the cladding layer for R. I. measurement of polymer thin films.

The chemical formulae of cladding (ZP2145M) and core (ZP1370M) are shown in

Fig. 3.1

Fig 3.1 Chemical formulae of (a) cladding (ZP2145M) and (b) core (ZP1370M)

The specifications of cladding (ZP2145M) and core (ZP1370M) used in this thesis are provided in the Table 3.1 below.

Table 3.1. Specifications of polymer materials

Product Items		ZP1370M	ZP2141M
Liquid	Viscosity (cps ± 30 at 25 $^{\circ}\text{C}$)	321	234
	Filtration	0.1 – 0.2 μm absolute PTFE filter	
	Curing Temp (T_c)	250 $\pm$ 10 $^{\circ}\text{C}$ available	
Film	Propagation Loss	0.3 - 0.4 dB/cm at 1.55 μm wavelength	
	PDL	0.02 dB/cm at 1.55 μm wavelength	
	Refractive Index at 1.55 μm	1.5480	1.4845
	Birefringes ($n_{TE} - n_{TM}$)	0.004 $\pm$ 0.001	
	Glass Transition Temp	235 $^{\circ}\text{C}$	132 $^{\circ}\text{C}$
	Degradation Temp	445 $^{\circ}\text{C}$	409 $^{\circ}\text{C}$
	CTE (ppm/ $^{\circ}\text{C}$)	74.2	116
	Film Thickness	< 10 μm per coating	

Initially, both cladding and core were spin-coated separately on different Si wafers after which the spin coated polymers were cured in the convection oven in N₂ ambient in order to avoid oxidation. The curing profile of these polymers included 3 minutes dwell at room temperature and ramping up to 250 °C at the rate of 5 °C/min. At 250 °C, both of these polymers were soaked for 4 hours and then ramped down to room temperature in 2 hours. While preparing samples to measure R. I. by prism coupling method, it was found that the spin-coated cured polymers peeled off. Therefore, in order to provide sufficient adhesion to SiO₂ layer, about 5 ml of adhesion promoter (ZAP1010) was spin-coated on top of SiO₂ and baked on the hot plate for 3 minutes at 110 °C. About 7 ml of ZP2145M and ZP1370M was spin-coated separately on different SiO₂ deposited wafers (with adhesion promoters) at definite speeds and were cured in the convection oven in N₂-ambient as described above.

The cured samples were used for R. I. measurement by prism coupling method using Metricon 2010. The details of the theory and working principles of prism coupling method have been described elsewhere [65-69]. For the measurement of refractive indices of ZP2145M and ZP1370M, the sample size about 5 cm × 5 cm was used and all measurements were carried out at the wavelength of 1550 nm.

Refractive indices of cured thin films of ZP2145M and ZP1370M were measured by prism coupling method both in TE and TM modes at 1550 nm wavelength. It was found that the refractive indices of ZP2145M in TE and TM modes were 1.4826 and 1.4771 respectively. Similarly, refractive indices of

ZP1370M in TE and TM modes were found to be 1.5475 and 1.5441 respectively. The observed refractive indices of both of these materials were compared with the supplied data and as shown in Table 3.2. It was observed that measured refractive indices were matching well with the supplied data sheet (ZEN Photonics Co., Ltd.).

Table 3.2. Observed refractive index (R. I.) of ZP2145M and ZP1370M materials

Materials	Observed n_{TE}	Observed n_{TM}	Supplied n_{TE}	Supplied n_{TM}	Observed Δn	Supplied Δn
ZP2145M	1.4826	1.4771	1.4840	NA	0.0055	0.004 $\pm$ 0.001
ZP1370M	1.5475	1.5441	1.5479	NA	0.0034	0.004 $\pm$ 0.001

It has been reported that during the curing process cross-linking of different polymer structures takes place [70] and the material acquires its optical property (e.g. R. I.) upon proper curing. The R. I. and birefringence of polymers is highly dependent upon the curing process [70]. Since the observed R. I. and birefringence match well with the vendor’s specification, it implies that the curing process is complete and optimized.

3.1.2 Moisture absorption

For the measurement of moisture uptake by the FPE material R. I. of this material was measured by the prism coupling method as a function of time. The R. I. is the critical physical parameter, which changes after absorption of moisture. It was found that the change of R. I. of the material in TE mode of core and cladding were

0.12 % and 0.17 % and that in TM mode were 0.14 % and 0.23 % respectively over 1815 hours of conditioning at 85 °C and 85 % RH which as shown in Fig. 3.2. Therefore, it could be concluded that the change of refractive index of FPE is insignificant after conditioning.

Fig. 3.2 (a). Variation of Refractive index with moisture conditioning time of ZP1370M

Fig. 3.2 (b). Variation of Refractive index with moisture conditioning time of ZP2145M

3.1.3 Thickness measurement of planar films (Spin coating optimization)

In order to obtain a desired thickness with minimum non-uniformity, thin films were prepared following the procedures as mentioned in section 3.1.1 at different speeds ranging from 700 to 1250 rpm. The thickness of the films at a particular speed at different locations in the six-inch wafer as shown in Fig. 3.3 was measured by stylus profiler (Dektak) and prism coupler. Nearly identical locations were chosen for the thickness measurement by a non-destructive prism coupling method. The procedure of measurement of thickness by prism coupler was the same as that of refractive index measurement. The wavelength used for all of the measurements was 1550 nm. In order to obtain R I and thickness parameters from prism coupling method, two guided modes were required. Finally, the attenuation loss measurement for thin film by prism coupling method was further carried out at 1550 nm to confirm the quality of uniformity of the thin film.

Fig. 3.3. Schematic diagram of a six inch Si wafer, where the numbers represent the location of the wafers at which thickness was measured.

The thickness of the thin films of ZP1370M was measured by prism coupling method by choosing different locations in the six-inch SiO₂ wafer as shown in Fig. 3.3. The spinning speed was chosen from 700 revolutions per minute (rpm) to 1250 rpm in order to obtain a desired thickness for fabricating waveguides. For a particular speed, the uniformity of the thickness was calculated from the thickness value at different locations. The thickness values at different speeds were tabulated as shown in Table 3.3 and the variation was found to be 2-3 %. For different speeds, the attenuation loss of thin films was calculated by the prism coupling method. It was found that at 1250 rpm, the attenuation loss was 1.27 dB/cm, which was the minimum loss achievable for the desired thickness of about 5 μ m as shown in Fig.3.4. At all other spinning speeds the attenuation loss is higher than that of 1250 rpm and this is due to the non-uniformity of film thickness and because of that scattering of light occurs rendering higher loss. For various studies, a particular spinning parameter was chosen based on the requirements.

Fig: 3.4. Attenuation loss of ZP1370M at different spinning speeds.

Table 3.3. Variation of thickness at different speeds measured by Prism coupling method

Speed (rpm)	No. of readings	Min. Thickness (μm)	Max. Thickness (μm)	Mean Thickness (μm)	Max. % variation
700	5	6.86	7.23	7.08	3.1
900	5	5.84	6.19	6.01	3.0
1000	5	5.79	5.98	5.93	2.36
1100	5	5.23	5.43	5.36	2.43
1250	5	4.97	5.11	5.07	1.97

3.2 Process studies for fabricating cladded waveguides

The process of fabricating polymeric cladded waveguides comprises various steps such as spin coating, curing, hard masking and RIE. Details of the process studies are described below.

3.2.1. Spin coating

Following the spin coating of an adhesion promoter, lower cladding of R. I. of 1.4826 was spin-coated on six-inch silicon wafers and was cured in the convection oven in N_2 atmosphere following the curing profile as shown in Fig. 3.5. Similarly, the core of R. I. of 1.5475 was spin coated on top of the cladding layer and cured following the same curing profile as lower cladding in N_2 atmosphere as shown in Fig. 3.5. At the optimum spinning speed, thickness of both lower cladding and core was chosen based on the requirements within $\pm 0.2 \mu\text{m}$ accuracy.

Fig.3.5. Curing profile of FPE

Fig. 3.6. Process flow chart for waveguide fabrication

3.2.2. Hard mask fabrication

The steps involved in hard mask fabrication are shown in steps (ii) to (v) in Fig. 3.6. Thin layer of about 1000 Å of Ti-metal was sputtered on top of the core layer. About seven ml of AZ P4620, a positive tone photoresist, was spun on the top of Ti-metal and was baked on the hotplate at 100 °C for 60 seconds. Four-inch

chrome bright field mask was used for patterning the photoresist in a proximity mask aligner. The width of the cladded waveguide to be fabricated was selected at 15 μm for ease of sample preparation for AFM measurements. After resist development, the samples were baked (post development baking) for 60 sec at 100 $^{\circ}\text{C}$. In the next step, Ti-etchant consisting of HF: HNO_3 : H_2O in the ratio of 1:1:50 was used for wet etching.

3.2.3. Dry etching

In this step, RIE was carried out by RF parallel plate etcher to transfer the pattern from hard mask (Ti) to the core polymer as shown in the Fig. 3.6 (vi) to (vii). RIE was carried out with pure oxygen and oxygen / nitrogen gas mixtures for various purposes, which are discussed in subsequent chapters in this thesis. The different process variables used in RIE are tabulated in chapters 5-6 and 8.

3.3 Surface Characterizations

Subsequently, after dry etching, surfaces of the cladded waveguides were characterized by SEM, AFM and SIMS and described in the following sub-sections.

3.3.1 SEM

Field emission SEM (FESEM) analysis for different samples processed under various RIE conditions was carried out for the following objectives.

- (a) To obtain the profile as well as the dimension of the core of cladded waveguides as well and to calculate vertical etch rate
- (b) To observe the sidewall roughness (SWR) of the waveguide and

- (c) To observe the *variation* of SWR of the waveguides

The details of the SEM studies are discussed in Chapters 4-7 and 9.

3.3.2 AFM

AFM was used to obtain quantitative information of SWR, LER, and PFR of waveguides at different interfaces and the characterization techniques are described in the following sections.

3.3.2.1 Characterization of SWR

AFM can be employed to directly measure the SWR of cladded waveguides. However, characterization of SWR by AFM is not straight forward since the AFM tip cannot reach the sidewalls because of the shape of the waveguides, and the detail reasons and various techniques approached to measure SWR have been discussed in section 2.2.2 of chapter 2. In this research work, a technique was developed in which an AFM was employed to directly measure the SWR of FPE waveguides neither modifying the AFM system nor the design of the waveguide patterns. For this characterization, waveguides of 15 μm width was selected in order to facilitate the cleavage necessary for the measurements. As shown in Fig. 3.7 (a), waveguides were cleaved using a diamond-tipped pen parallel to the edge of the waveguides (z -direction). The sample was then tilted vertically as shown in Fig. 3.7 (b) so that the AFM tip could get full access to the sidewall. An AFM in the tapping mode was used to directly measure the SWR of FPE waveguides. AFM scanning was carried out from top to bottom along the depth of waveguides (in y - z plane) with a scan length of 4.2 μm at the sidewalls.

Fig 3.7. Schematic diagram of sample preparation method to characterize sidewall roughness by AFM (a) before cleaving and (b) after cleaving.

3.3.2.2 Characterization of LER

LER is another roughness parameter which has often been described in conjunction with SWR in section 2.2.2 of chapter 2. In one of the experimental studies, LER is assumed to be the same as the SWR [2], while in theoretical studies [7-10], the SWR has typically been assumed to be constant throughout the sidewall, with a value equivalent to LER. In this research work, LER was measured by AFM at the top of the sidewall at the vertical depth of $(0 / 0 + 33 \text{ nm})$ in the z -direction for the scan length of $4.2 \text{ }\mu\text{m}$. An AFM in the tapping mode was used to directly measure the LER of FPE waveguides.

3.3.2.3 Characterization of PFR

Behavior of plasma-matter interactions is often studied by observing the evolution of PFR. AFM in tapping mode was also employed to measure the PFR of

FPE waveguides. After RIE at various conditions, PFR was measured. The sample size for PFR measurement was $10 \times 10 \mu\text{m}^2$ for this research works.

3.3.3 SIMS

In this research work, time of flight SIMS (ToF SIMS) was employed for surface characterization in order to obtain various surface elements on the etched thin films that were treated at various RIE conditions. The etched thin films were treated with pure O_2 , pure N_2 and mixture of O_2/N_2 plasma. For the surface characterization of these films, gallium (Ga) gun with standard parameters such as 25 keV energy, 2.00 pA of current were considered. The analysis area for surface characterization was $50 \mu\text{m} \times 50 \mu\text{m}$ and the negative mass spectra were collected. Depth profiling of these etched thin films were also carried out with Ga gun with 25 keV energy, 2.00 pA of current and argon (Ar) gun with 3 keV energy, 60.00 nA of current. The sputtered area was $50 \mu\text{m} \times 50 \mu\text{m}$ and the analysis area was $30 \mu\text{m} \times 30 \mu\text{m}$. Depth profiling data were collected with negative polarity and the sputtering of the etched films was carried out for 3600 sec.

Chapter 4

Direct Measurement of Sidewall Roughness of Polymeric Optical Waveguides

In this chapter, atomic force microscopy (AFM) was employed for the direct measurement of various types of roughness in general and SWR in particular, which are evolved at different interfaces of the polymer waveguides, neither modifying the AFM system nor the waveguide designs. The types of roughness are line edge roughness (LER), SWR and planar film roughness (PFR) and the details are described in following sections.

4.1 Classification of various types of roughness evolved during RIE

Both theoretical and experimental studies on scattering loss due to SWR have been discussed in chapter 2. In these studies, it has been repeatedly misconstrued that SWR is constant along the depth of the waveguide and LER is equivalent to SWR. The details about these ambiguities have already been discussed in Chapter 2. However, no distinction about PFR, LER and SWR was found in the literature.

Various types of roughness evolved during RIE at the interfaces of waveguides are shown in Fig. 4.1 (a). LER is the line edge roughness of the core layer (shown without upper cladding) in which variation in x occurs along z (Fig. 4.1 (b)). SWR is the sidewall roughness where variation in x occurs within the y - z plane (Fig. 4.1 (c)). Planar film roughness (PFR) is the roughness of the planar film where variation in y occurs within the x - z plane (Fig. 4.1 (d)). All these three types of

roughness are present in different planes of the waveguide and the measurements of them are discussed in the following sections.

Fig. 4.1. Schematic diagram of (a) various types of roughness, LER, SWR and PFR, which are evolved during RIE (b) In LER, variation in x occurs along z ; (c) in SWR, variation in x occurs within y - z plane; and (d) in PFR, variation in y occurs within x - z . The arrows indicate the height variations.

4.2 Measurement of LER at different pressure conditions

A roughness parameter called LER, as described earlier, has often been described in conjunction with SWR. In one of the experimental studies, it was assumed that LER is the same as the SWR [4]; and in theoretical studies [5, 7-9], the SWR has typically been assumed to be constant throughout the sidewall, with a value equivalent to LER. In order to remove this ambiguity, LER was measured at the top of the sidewall at the vertical depth of (0 / 0 + 33 nm) in the z-direction for the scan length of 4.2 μm and the standard deviation (sd) value of LER was 35 nm. On the other hand, the sd roughness value of the scanned area of 4.2 μm $\times$ 4.2 μm of sidewall of the waveguide was 30 nm. Both LER and SWR at various pressure conditions were measured and summarized in Table 4.1. It was found that LER and SWR are different for each condition. Therefore, this confirms our earlier contention that LER is not equivalent to SWR and should be treated as separate roughness parameters.

Table 4.1. Summary of LER and SWR at various pressures at 300W R.F. power, 18 sccm O₂ flow rate.

Pressure (mT)	LER (nm)	SWR (nm)
35	35.0	29.9
65	19.4	36.6
95	76.4	45.3

4.3 Measurement of surface area roughness of sidewall

The SWR of the waveguide with the etch depth of 4.2 μm was measured by AFM in the tapping mode from top to the bottom of the sidewall with the scan length of 4.2 μm and the details about the sample preparations for SWR measurement has

been described in section 3.3.2 in chapter 3. Fig. 4.2 shows SWR of waveguide in which the scanning area was $4.2\ \mu\text{m} \times 4.2\ \mu\text{m}$ (in y - z plane). The sd of roughness for the scanning area of $4.2\ \mu\text{m} \times 4.2\ \mu\text{m}$ was 30 nm. It is commonly believed that the rms or sd roughness, which is the height variation in the vertical direction only, of the sidewall provides the quantitative information. However, this roughness value neither conveys any information on the variation of SWR as a function of vertical etch depth of waveguides nor provides the roughness information which varies along the horizontal direction. It is also noteworthy to mention that the rms or sd roughness is the first order statistical roughness parameter; however, ACF, ACL, HHCF, and roughness exponents are the second order statistical roughness parameters. The second order statistical roughness parameters convey roughness information along the horizontal or lateral direction. Therefore, the second order statistical roughness parameters must be considered along with the first order statistical roughness parameter to differentiate various types of rough surfaces. The details about the evolution of SWR and the variation of SWR with etch depth and pressures will be described in Chapters 5-6.

Fig. 4.2 . 3D sidewall profile of the $15\ \mu\text{m}$ width waveguide measured by AFM. The arrow indicates the scanning direction of AFM tip.

4.4 Measurement of planar film roughness

Evolution of SWR can be correlated with the help of planar film roughness (PFR). The details about the evolution of roughness at the sidewalls as well as in the planar film are described in Chapters 5-6 and 7. In order to measure PFR, FPE thin films were treated with various RIE conditions and the roughness at the planar films was measured by AFM in tapping mode for the scanning area of $10\ \mu\text{m} \times 10\ \mu\text{m}$. Fig. 4.3 shows the 2D and 3D AFM images for the scanning area of $10\ \mu\text{m} \times 10\ \mu\text{m}$ which was treated at the pressure of 50 mT, RF power of 300W and O_2 flow rate of 18 sccm at the end of 35 min of etching. The PFR at this RIE treated conditions is 105.1 nm. The details about the evolution of PFR with etch time are described in Chapter 7.

Fig. 4.3. AFM figures with the variation of etching time at $t = 35$ min for 50 mT, 300W and 18 sccm O_2 flow rate in (i) 2D, showing hillocks and valleys and (ii) 3D, showing peaks and valleys.

4.5 Summary and conclusions

Various types of roughnesses evolved during RIE at different surfaces were classified and defined distinctly in order to remove the ambiguities which have been

described so far in the literature. LER of different samples treated at various pressure conditions were measured successfully and were compared with the corresponding SWR. It was confirmed that LER and SWR are different and should be treated as two different roughness parameters. SWR of the sidewalls were successfully measured from the top to the bottom of the sidewalls by following a special technique and the standard deviation (sd) of roughness at the sidewall was measured. However, this sd of SWR does not convey any information about the variation of SWR along the vertical depth of the waveguides. PFR of the RIE treated samples were also measured successfully. It is noteworthy to mention that LER, SWR and PFR are all three different parameters in different planes of the surfaces, and the effects and evolution of these roughness parameters will be described in subsequent chapters.

Chapter 5

Effect of Process Parameters on Sidewall Roughness in Polymeric Optical Waveguides

This chapter describes the importance of understanding the effects of RIE parameters on sidewall roughness as well as profile of the waveguides. The following sections in this chapter describe the fabrication of polymeric (fluorinated polyether, FPE) optical waveguides by metallic hard mask and proximity exposure followed by RIE process. The effect of RF power and pressure on SWR are discussed in sections 5.1.1 and 5.1.2 and pressure effect is explained in section 5.1.2.1.

5.1 Process parameter studies

In this section, effect of process parameters such as RF power and pressure on SWR is described. Important mechanisms for the effects are also provided.

5.1.1 Effect of RF power on SWR

The variation of RF power was carried out from 225 W to 300 W keeping other parameters such as pressure, oxygen flow rate and etching time constant at 45mT, 18 sccm and 35 min respectively. As shown in Fig. 5.1 (a), at low power (225 W) (split 1 in Table 5.1), the profile of the waveguide was non-vertical while increasing the power (splits 2 to 3), the profile became more vertical as shown in Figs. 5.1 (b - c). The hillock in the Fig. 5.1 (a) is the vestige of photoresist which clearly indicates that this RF power is insufficient to remove the photoresist as well

as to produce vertical sidewalls. As shown in Fig. 5.2, the etch rate increases with RF power from 225 W to 275 W and decreases with power from 275 W to 300 W. The photoresist which remained during split 1 was completely etched away in split 2. It is interesting to note that at maximum etch rate, the root mean square (rms) sidewall roughness is at a minimum. At a higher power (300W) the sidewall roughness again increases. Fig. 5.3 shows the sidewall roughness of the waveguides scanned by AFM for the above RIE conditions.

Plasma consists of atoms, neutral molecules, ions and electrons. In oxygen-RIE for polymers, oxygen atoms initiate the etching by extracting hydrogen atoms from a polymer chain leaving the radical species. These radicals can react with oxygen molecules from the plasma to form functional groups such as carbonyl and alcohol groups. These are the precursors to volatile species which are pumped away after desorption.

Fig 5.1. SEM micrographs at different RF powers (a) 225 W (b) 275 W and (c) 300W at 45 mT Pressure, 18 sccm O₂ flow rate and 35 min etching time

Fig. 5.2. Variation of vertical etch rate and sidewall roughness with RF power at 45 mT pressure, 18 sccm O₂ flow rate and 35 min etching time

Fig. 5.3. AFM images at different RF powers at (a) 225 W, (b) 275 W and (c) 300 W at 45 mT pressure, 18 sccm O₂ flow rate and 35 min etching time

Table 5.1. Variation of RF Power

Split No	Pressure (mT)	Power (W)	O ₂ flow rate (sccm)	Time (min)	Vertical Etch rate (nm/min)
1	45	225	18	35	140
2	45	275	18	35	189
3	45	300	18	35	143

There exists a peak in the variation of RF power vs. etch rate as shown in Fig. 5.2. When RF power is below this peak, the etch rate is limited by the supply of atomic oxygen which initiates the etching of polymers. With the increase in RF power, more atomic oxygen will be generated because of the increased dissociation of the oxygen molecules, thus increasing the etch rate. When RF power is above this peak, the etch rate and the generation of the atomic oxygen is limited by the pressure. Further increase in RF power does not increase the generation of atomic oxygen and therefore, the etch rate decreases. Hence, in order to increase the etch rate, pressure needs to be increased as increase of pressure increases the etch rate, as shown in Fig. 5.5. There also exists a minimum in the variation of RF power vs. sidewall roughness as shown in Fig. 5.2. When RF power is below this minimum, roughness is limited by the supply of atomic oxygen. When RF power is increased, further generation of atomic oxygen etches the polymeric film faster than the sidewall i.e. etching in the planar film is faster than that of the etching of the sidewall. This corresponds to the minimum roughness condition shown in Fig. 5.2 since the angle of incidence of the reacting species for the planar films is different from that for the sidewalls [3]. When

RF power is above this minimum the etching in the film is slower than that at the sidewall. This can be attributed to the physical sputtering of the ionic species, which is stronger at the sidewall, resulting in a higher sidewall roughness because of the same reason of angle of incidence [3].

5.1.2 Effect of pressure on SWR

RIE pressure was varied from 45 mT to 95 mT, keeping all other parameters such as RF power at 300W, oxygen flow rate at 18 sccm, and etching time 35 minutes fixed as shown in Table 5.2. Scanning electron micrographs of waveguides fabricated at pressures ranging from 45 mT to 95 mT are shown in Fig. 5.4. As shown in Fig. 5.5, both vertical etch rate and sidewall roughness increase with the increase in pressure. Vertical etch rate increases from 143 nm/min to 195 nm/min when pressure increases from 45 mT to 95 mT and the corresponding rms sidewall roughness increases from 34.1 nm to 49.1 nm. It is noteworthy to point out that these roughness values were the values for the entire sidewalls of area $4.2 \mu\text{m} \times 4.2 \mu\text{m}$. This effect is known as *Pressure Effect*. When pressure increases, the mean free path between the reacting species decreases and the collision among the species generates some amount of atomic oxygen and ions and therefore, the etch rate increases in the vertical direction. At the same time, another effect of pressure is that with the increase of pressure the reacting species such as atoms, molecules, electrons and ions become non-directional and significant etching occurs at the sidewalls of the waveguides, which cause the increase of sidewall roughness, since the angle of incidence of the reacting species for the planar films is different from that for the sidewalls [3]. The details of the mechanism are described in section 5.1.2.1.

Fig. 5.4. SEM micrographs at different pressures (a) 65 mT and (b) 95 mT at 300 W RF power, 18 sccm O₂ flow rate and 35 min etching time.

Table 5.2. Variation of Pressure

Split No	Pressure (mT)	Power (W)	O ₂ flow rate (sccm)	Time (min)	Vertical Etch rate (nm/min)
1	45	300	18	35	143
2	65	300	18	35	182
3	95	300	18	35	195

Fig. 5.5. Variation of vertical etch rate and sidewall roughness with pressure at 300 W RF power, 18 sccm O_2 flow rate and 35 min etching time

5.1.2.1 Pressure Effect Explanation

At low pressure (45 mT), the ratio of oblique to normal ions to the sidewall [1] is higher due to less collisions and longer mean free path. Due to the oblique incidence of the etchant ions at the sidewalls, the peaks of the morphology block most of the particles rather than the valleys as shown in Fig. 5.6 (a). This effect is known as the shadowing effect. A shadowing model developed by Drotar *et al.* [71] suggests that the sticking coefficient, S_s during shadowing effect is greater than that of the reemitted sticking coefficient, S_R . In other words, the reemission effect during the shadowing effect is negligible or weak. Hence, at low pressure, the incoming particles at the point of incidence are etched because of higher sticking coefficient than because of reemission, and this scenario is shown in the schematic diagram, Fig.

5.6 (b), indicated by an arrow. Therefore, low RIE pressure results in lower roughness.

Fig. 5.6 (a). Shadowing effect explanation at higher pressure. The number 1, 2 and 3 represents particles from different directions.

Fig. 5.6 (b). Etching at the peaks due to shadowing effect. The sticking coefficient for shadowing effect, $S_S = 1$, whereas the sticking coefficient for reemission effect, $S_R = 0$. Etching occurs in downward directions indicated by an arrow.

At higher pressure, the ratio of normal to oblique ions is higher at the sidewall [1] owing to vigorous collisions and shorter mean free path. Therefore, the particles after hitting the morphology of the surface are reflected towards the valleys [1] as shown in Fig. 5.7 (a). Since the sticking coefficients of the incident particles are smaller than those of the reemitted particles [48], the particles reemitted to the

valleys of the morphology are stuck and etching takes place at the reemitted positions at the valleys. Hence, due to etching at the valleys the roughness increases as the

Fig. 5.7(a). Reemission of the particles at low pressure due to normal incidence and (b) Etching at the valleys due to reemission which is responsible for the higher roughness. The number 1 and 2 represents particles from different directions.

valleys etch in downward directions as indicated by the arrows as shown in Fig. 5.7 (b).

Fig. 5.8 (a-b) shows the 3D AFM images of the sidewalls of FPE waveguides etched under the variation of corresponding pressure. It has been reported that the sidewall roughness is the major source of optical loss [2], and scattering loss due to roughness is proportional to the square of the root mean square deviation of the film thickness [2]. As the rms values of sidewall roughness increases with the increase of pressure, the scattering loss increases, thus contributing significantly to the optical loss. It has been reported by Kang et al [72] that high pressure RIE etching can provide a smoother surface, which is true for planar films and the details are provided in Chapter 7. But our results indicate that this is not true for sidewalls as the sidewall roughness depends upon the angle of incidence of the reacting species [1]. Our results suggest that low pressure plasma conditions are necessary to decrease

sidewall roughness, which is in accord with the results presented by Agarwal et al [1].

Fig. 5.8. AFM images at different pressures at (a) 65 mT with oxygen and (b) 95 mT with O₂ at 300 W RF power, 18 sccm O₂ flow rate and 35 min etching time.

5.2 Summary and conclusions

FPE waveguides at different RIE conditions were successfully fabricated and the effects of RF power and pressure in the oxygen plasma were investigated. This study shows that higher RF power provides more vertical profile and vertical etch

rate decreases by reaching to a maximum value as the RF power increases. Moreover, with the increase of pressure the SWR increases and this effect is known as *Pressure Effect*, and with the increase of pressure the vertical etch rate also increases. Furthermore, a smoother sidewall can be obtained at low pressure which is because of the higher oblique to normal ions ratio at the sidewalls. Because of the shadowing effect of oblique ions and weak reemission, smoothness at the sidewall resulted at low pressure. However, at higher pressure, the first order reemission was dominant and because of that the valleys of the morphology were etched providing the higher roughness at the sidewalls.

Chapter 6

Variation and Evolution of Sidewall Roughness of Polymeric Waveguides during Reactive Ion Etching

Direct measurement of SWR, LER, and PFR has been described and discussed in Chapter 4 and pressure effects of PFR, and effect of RIE parameters such as RF power, and pressure on SWR are described in Chapter 5. However, in this chapter, the emphasis is provided on *variation* of SWR along the depth of the waveguides. Particularly, this chapter describes the experimental results of *variation* of SWR of fluorinated polyether (FPE) waveguides and its dependence on etch depth at different RIE conditions using pure O₂ plasma. The evolution of *variation* of SWR will be described and discussed in section 6.2.

6.1 Variation of SWR

Variation of SWR along the depth of the waveguides was studied by varying the RF powers as well as pressure and the details of which are described in section 6.1.1 and 6.1.2.

6.1.1 Variation of SWR at different RF powers

FPE waveguides were fabricated at various RIE conditions by varying RF power from 225 W to 300 W keeping the pressure at 45 mT and the pure O₂ gas flow rate at 18 sccm fixed. All the waveguides were etched to a vertical depth of about 4.2 μm . The SWR of the waveguides fabricated at the above RIE conditions was measured by AFM in tapping mode from top to the bottom of the sidewall at 400 nm (± 25 nm) intervals with the scan length of 4.2 μm . Fig. 6.1 shows 3D AFM images

with the variation of power from 225 W to 300 W and the arrow indicates the scanning direction (along y-direction). The variation of SWR along the depth of waveguides at different RF powers is plotted in Fig 6.2. It was observed that the variation of SWR along the vertical depth of the waveguides does not vary significantly for all RF powers since the angle of incidence of ions is similar in nature (e.g. vertical) towards the samples to be etched, and the mean free path of the particles does not change. Hence, the nature of variation of SWR remains almost the same at all power ranges. However, in our previous studies (Chapter 5) it was found that the profile of the waveguides remained vertical and the vertical etch rate increased as the power increased. Therefore, higher power was considered for further studies on the variation of SWR along the vertical depth, as described in the following sections.

Fig. 6.1. 3D AFM images at (a) 225 W, (b) 275 W and (c) 300 W.

Fig. 6.2. Variation of SWR along the vertical depth at different rf powers.

6.1.2 Variation of SWR at different pressures

It was discussed in previous sections that the sidewall profiles became more vertical with increasing RF power, providing better control of etch bias. However, in this experiment, the RF power was kept fixed at 300 W and the pure O₂ gas flow rate was fixed at 18 sccm while the pressure was varied from 35 mT to 95 mT. All the waveguides were etched to a depth of about 4.2 μm . The SEM micrographs for waveguides etched at various pressure conditions as stated above are shown in Fig. 6.3. Different types of textures, as shown in Fig. 6.3 (a(ii)-c(ii)), were observed at the sidewalls during RIE at various vertical depths, indicating the variation of SWR. It was also observed that as pressure increased, trenches at the sidewall began to develop and became prominent in case of 95 mT pressure, as shown in Fig. 6.3 (c) (iii). In order to obtain the quantitative information on variation of SWR, AFM was used. Fig. 6.4 shows the 3D AFM images of these sidewalls from 35 mT to 95 mT. The observed evolution of trenches at the sidewalls is clearly consistent with the SEM observations. The details about the evolution of variation of SWR are discussed in section 6.2.

The SWR of the waveguides (in the y - z plane) treated at various pressure conditions was measured by AFM from top to bottom of the sidewall at the vertical depth intervals (in y – direction) of 400 nm (± 25 nm) with the scan length of 4.2 μ m (along z - direction). Fig. 6.5 shows the variation of SWR with vertical depths of waveguides at different etching pressures. At lower pressure (35 mT), two different regions delineated by the point A in Fig. 6.5 are observed. In the first region, SWR increases and then saturates. In the second region, beyond the point A, SWR increases. Variation of SWR is between 18.6 nm and 33.4 nm from 400 to 4000 nm thickness (depth) of the waveguides. At a higher pressure (65 mT), the initial roughness at the sidewall also increases, reaching higher values compared to the data of lower pressure (35 mT), and finally decreases gradually beyond point B, without undergoing any changes in intermediate depths. In contrast to both these cases, at a higher pressure of 95 mT, SWR shows a gradual decline from 56.9 nm to 12.9 nm along the same vertical depths, without following the etched behavior obtained at lower pressures. These variations of SWR along the vertical depths that were claimed earlier support the SEM micrographs in Fig. 6.3, having different textures at the sidewalls at each pressure condition. This confirms our earlier contention that *SWR is not constant* along the depth of the sidewall, but it is with a *variation* which in fact is dependent on etch pressure.

(i)

(ii)

Fig. 6.3 (a) (i). SEM micrograph of FPE waveguide and (ii) its sidewall, fabricated at 300 W rf power, 35 mT pressure and oxygen gas flow rate of 18 sccm. Different types of textures were obtained along the vertical depths of the sidewall.

Fig. 6.3 (b)(i). SEM micrograph of FPE waveguide and (ii) its sidewall, fabricated at 300 W rf power, 65 mT pressure and oxygen gas flow rate of 18 sccm. Different types of textures were obtained along the vertical depths of the sidewall and evolved trenches are indicated by arrows.

(i)

(ii)

(iii)

Fig. 6.3 (c) (i). SEM micrograph of FPE waveguide (ii) textures at sidewall, and (iii) magnified trenches at the sidewall of waveguide fabricated at 300 W rf power, 95 mT pressure and oxygen gas flow rate of 18 sccm. Different types of textures were obtained along the vertical depths of the sidewall and evolved trenches are indicated by arrows.

Fig. 6.4. 3D AFM images of sidewall roughness of FPE waveguides at (i) 35 mT, (ii) 65 mT and (iii) 95 mT pressures.

Fig. 6.5. Variation of SWR with vertical depths at various pressures.

6.2 Evolution of variation of SWR

In this section, we investigate further analysis of the variation of SWR followed by mechanisms to explain the evolution of SWR. Both pressure and depth dependences of SWR are discussed in this section. Second order statistical roughness parameters such as autocorrelation length (ACL) were calculated from autocorrelation function (ACF) in order to establish the relationship between SWR and etch depth, and height-height correlation function (HHCF) was used to calculate the roughness exponents, α for different RIE parameters.

6.2.1 Pressure effect

In our study, we assume SWR to be proportional to the PFR of the etch front, and the SWR recorded as a function of etch depth is treated as a tracking indicator of the etched surface roughness as a function of etch time. Therefore, it allows us to

compare our observations with other studies of roughness evolution on planar etch surfaces. Recently, it has been reported that for planar polymer film etching, planar surface roughness increases linearly with the material etched, beyond a threshold etch depth of about 116 nm at etch pressure similar to ours [46]. Our SWR results support this assertion in general, until an etch depth of about 1200 nm for both 35 mT and 65 mT pressure conditions (Fig. 6.5), after which the roughness dependence on depth deviates drastically from a linear behavior.

To explain the above behavior, we follow the theoretical framework by Drotar *et al.* [71] which describe the evolution of surface roughness for both growth and etching systems using a shadowing model. Through Monte Carlo simulation studies, it was reported that the roughness of an etched surface should increase and then saturate with time, if the roughening is shadowing limited [71]. Our lower pressure (35 mT) data obeys the predictions of the shadowing model very well up to the vertical depth of 2400 nm, thus demonstrating that roughness at shallow structures is dominated by shadowing effects. However, SWR increases at higher etch depths (beyond 2400 nm). It has also been reported by Drotar *et al.* [48] that for an etching mechanism involving the first order reemission of etchants, roughness increases with time. Therefore, we may attribute the observed increase in roughness beyond 2400 nm at 35 mT to be due to first order etchant reemission effects. Hence, the variation of SWR at lower pressure can be considered to be a combination of both etchant shadowing and first order reemission effects. At 65 mT, the decline of SWR occurs at about the vertical depth of 2600 nm, (point B in Fig.6.5) and before this point, the behavior of SWR follows the same trend as of 35 mT, suggesting the

shadowing behavior of etchants. At higher pressure (95 mT) for shallow-etched structures the roughness is even higher than 65 mT, which further supports Agarwal *et al.* that with the increase of pressure, the SWR increases [1]. In other words, lower pressure produces lower SWR and vice versa. This effect is known as *pressure effect*. Referring to the Fig. 6.5, for the pressure ranges of 35 to 95 mT, the *pressure effect* region is up to ~ 3000 nm along the vertical depth of the waveguide.

6.2.2 Depth effect

For the deep-etched structures, at 95 mT, the decline of SWR was observed as similar to the case of 65 mT, beyond the point B (Fig. 6.5). In other words, for 95 mT pressure, the variation of SWR is different from the shadowing effect and the first order reemission effect observed for low pressure (35 mT) conditions, which were discussed in section 6.2.1. In comparing the 65 mT SWR variations, the portion of the curve before point B goes up, and the portion of the curve beyond point B goes down as shown in Fig. 6.5 as indicated by arrows for the 95 mT condition. Therefore, our data suggest that the angle of incidence of ions with respect to the sidewall as described by Agarwal *et al.* [1] may undergo a change in behavior as the etch front extends beyond a certain critical depth. For our etch conditions and material, this critical depth, corresponding to the depth where SWR begins to decline, is roughly 2600 nm and 1200 nm for 65 mT and 95 mT respectively. Furthermore, in comparing 35 and 95 mT curves as shown in Fig. 6.5, our results have uncovered an additional dependence on the etch depth. As etch depth increases, the roughening trends due to pressure seem to reverse – the lower initial roughness at low pressure increases with depth, while the higher initial roughness at high pressure decreases

with depth - such that the trends cross at the critical depth. In our experiments, this happened at the vertical depth of about 3000 nm, as shown in Fig. 6.5. Before this crossover, oblique to normal ions ratio is dependent upon pressure (the *pressure effect*) which is in agreement with Agarwal et al [46]; however, beyond the crossover, the effect of depth is large enough to change the pressure effect. Hence, beyond the crossover, the roughening etching occurs for lower pressure and non-roughening etching occurs for higher pressure. In other words, in terms of sidewall smoothness, for shallow etching, lower pressure is better, while for deep etching, higher pressure may be necessary and hence, this effect is known as *depth effect*. Therefore, we believe that the decline or increase of the roughness beyond the critical depth must be contributed by different mechanisms.

6.2.3 Mechanisms for pressure and depth effects

At low pressure (35 mT), the particles are oblique to the sidewall [1]. When particles are incident at an oblique angle, they are shadowed by the peaks. As shown in Fig. 5.6 (a) above, peaks block most of the particles; thus, valleys are protected. It has been reported that during shadowing, first order reemission effect is negligible (as sticking coefficient, $s_S = 1$, $s_R = 0$, where s_S and s_R are the sticking coefficients for shadowing and the first order reemission effects respectively) [71]. Therefore, peaks are etched preferentially with a corresponding decrease in roughness until the baseline (defined here as the mean height of the roughness distribution) is reached, as shown in Fig. 6.6 (a), after which the roughness becomes fairly constant because of the relative scarcity of peaks for further smoothening and this condition should be applicable for both the 35 and 65 mT scenarios. The initial increase in roughness as

shown in Fig. 6.5 for 35 mT is due to the presence of noise [71]. It has been reported for the growth of thin film that when the particles randomly arrive on the surface small undulations are created because of noise [73]. In RIE similar noise-induced undulations would be created as particles arrive randomly towards the samples. It has also been reported that the plasma and reactive ion etch-fronts roughening may be due to (a) non-directional flux with specular reemission, (b) non-directional flux with thermal reemission, or (c) directional flux with thermal reemission and because of that the rms roughness increases with etch time [48]. Therefore, further increase of roughness in Fig. 6.6 (b) beyond the dashed vertical line in the inset during low pressure (35 mT) condition must be attributed to etching at the valleys owing to any of the above reemission mechanisms. As shown in Fig. 6.6 (b), the most likely cause is the reemission at the point C due to any of the above reemission mechanisms. In this case, particles incident on a surface are reemitted to the valleys since there are no more peaks ($s_S = 0$, $s_R = 1$) [48]. The particles which are reemitted to the valleys must etch the film as the sticking coefficient of incident particle is smaller than that of the reemitted particles [71], as shown in Fig. 6.6 (b). As a result of this, valleys are now etched preferentially and thus, roughness increases. Therefore, the baseline can be seen as a transition between shadowing and reemission effects.

The high pressure roughness phenomenon can be explained by an extension of the above mechanism. As before, the initial increase of roughness (Fig. 6.5) during high pressure (95 mT), is due to the presence of noise [48]. After the initial roughness is established, peaks are preferably etched and valleys are protected due to shadowing effect. As etching continues, peaks reach baseline. But here, the particles

are expected to be less energetic due to more frequent collisions under higher pressure. Hence, sticking coefficients are higher at the point of incidence resulting in etching rather than reemission. As shown in Fig. 6.6 (c) (i-iv), etching of peaks occurs not only during shadowing but continues beyond baseline because of weak reemission. The baseline shifts downwards and roughness continues to decrease. We have tentatively named this phenomenon as the *continuous shadowing effect* (CSE).

Fig. 6.6 (a). Schematic diagram of shadowing effect explanations for 35 and 65 mT pressure curves. Inset plot is an example for the shadowing effect regions for 35 mT pressure.

Fig. 6.6 (b). Schematic diagram of first order reemission effect explanations for 35 mT pressure curve. Inset plot is an example for the first order reemission effect regions indicated by a vertical dashed line and an arrow for 35 mT

Fig. 6.6 (c). Schematic diagram of CSE explanations for 95 mT pressure curve at different etch times, where, $t_1 < t_2 < t_3 < t_4$. Inset is an example for the CSE regions for 95 mT pressure.

6.2.4 Variation of ACL at different pressures

Besides rms roughness, the autocorrelation length (ACL) is another statistical parameter, which is equally important for the computation of scattering loss for many theoretical frameworks [7-10, 74]. The ACL was calculated from normalized

autocovariance function (ACF) and it is defined as the distance over which the ACF falls to e^{-1} of its maximum value [56, 71]. ACL was extracted from each ACF curve for various depths from top to bottom of the sidewalls at 400 nm (± 25 nm) intervals. ACLs at each pressure condition are plotted with vertical depths as shown in Fig. 6.7 (a-c) and are compared with corresponding rms SWR (shown in dotted lines in Fig. 6.7). It was observed that at each pressure condition (from 35 to 95 mT) ACL varies in a similar trend as the SWR does, which is shown in Fig. 6.7 (a-c). It has been reported that for the rough deposits of metals, there is a linear relationship between the surface roughness and ACL [75]. Our calculation (Fig. 6.7) also shows that ACL varies in the similar manner as SWR along the vertical depth, implying that surface roughness evolution during plasma etching may possess the same dynamics as that of thin film growth. Furthermore, the tracking of the variation of ACL and rms roughness along the vertical depth of the waveguide is also similar to the behavior of ACL and rms roughness with etch time [49] for the planar film, which further confirms that the evolution of variation of SWR along the depth of the waveguides possesses the same etching dynamics as that of the etching of planar films.

Fig. 6.7. Variation of ACL with vertical depths at (a) 35 mT, (b) 65 mT, and (c) 95 mT pressures. Variation of SWR at respective pressures shown with dotted curves is repeated for the comparison with ACL.

6.2.5 Variation of Roughness Exponent (α) at different pressures

The roughness exponent, α is another statistical parameter which is used to describe the rough surface. The roughness exponent, α is extracted from the slopes of the linear portion of the log-log plot of HHCF, $\log (H(r))$ and $\log (r)$, where r is the scanning length over which the experimental data are collected in our experiments. The numerical formula for HHCF to obtain α has been described elsewhere [76]. For each vertical depth, the HHCF is plotted for a particular RIE parameter (Figs. 6.8 – 6.10). It is found that the average values of α over the entire scanned sidewall area are 0.70, 0.90 and 0.91 for 35, 65 and 95 mT respectively. Lower value of roughness exponent at lower pressure is attributed to the mechanism that has been described for the increase of low pressure roughness. At low pressure, the reemitted particles cause more jagged surface due to etching at the valleys and hence, α decreases. However, at higher pressure due to continuous shadowing effect, less jagged surfaces are created and hence, α increases. As shown in Table 6.1, the average roughness exponent, α , which represents the lateral variation of the surface, increases as the pressure increases, and this increase of α supports the fact that the surfaces tend to be locally smoother [76].

Table 6.1. Comparison of roughness parameters

Pressure (mT)	Average SWR (nm)	Avearge Roughness Exponent, α
35	29.7856	0.70
65	34.658	0.90
95	40.845	0.91

Fig. 6.8. The log-log plot of HHCF and distance (r) for different vertical depths of for 35 mT pressure.

Fig. 6.9. The log-log plot of HHCF and distance (r) for different vertical depths of for 65 mT pressure.

Fig. 6.10. The log-log plot of HHCF and distance (r) for different vertical depths of for 95 mT pressure.

6.3 Summary and conclusions

FPE waveguides at various RIE conditions in O_2 plasma were fabricated by varying RF power and pressure. It was observed that the variation of SWR along the vertical depth of the waveguides did not vary significantly at all RF power ranges. The nature of variation of SWR remained almost the same in all power ranges. However, by varying pressure, variation of SWR along the vertical depth of the waveguides was observed by SEM, measured and confirmed directly by AFM. The experimental findings indicate that SWR varies with etch depth, with opposite trends for low and high pressure etches at the critical depths. This implies that while O_2 low pressure RIE may provide smoother sidewalls for shallow polymeric structures, however, O_2 higher pressure RIE may be better suited for the deep etched structures. Furthermore, a complete description of etch roughening mechanisms based on the relative ratios of oblique and normal incidence of ions must consider the influence of etch depth in addition to that of pressure. Hence, pressure is found to be the most dominant factor that causes the variation of SWR along the depth of the waveguides.

The evolution of variation of SWR of FPE waveguides using pure oxygen plasma was also investigated. The variation of SWR along the vertical depth at lower pressure was due to the combination of both shadowing effect at which reemission effects were negligible showing the minimum or saturation of roughness values and the first order reemission effect. The increase of roughness for deep-etched structures at lower pressure must be due to reemission of etchants to the valleys of the morphology where the shadowing effect is completely negligible. However, variation of SWR at higher pressure was due to continuous shadowing effect both for peaks and valleys where the reemission is weak due to less energetic etchants. From ACL calculation, it was found that ACL along the vertical depth of the waveguides varied in a similar manner as SWR, suggesting that surface roughness evolution at the sidewalls during plasma etching possesses the same dynamics as that of etching of planar film. The average value of the roughness exponent α increased as the pressure increased, suggesting that higher pressure etches tend to produce surfaces which are locally smoother.

Chapter 7

Study of evolution of planar film roughness during reactive ion etching of polymers

In this chapter evolution of roughening phenomena of the planar films was studied in order to acquire insight into the roughness evolution of the planar film, which will be further useful to understanding the evolution of SWR. Pressure effects of PFR and evolution of PFR with time at different pressures are described in section 7.1 and 7.2 respectively.

7.1 Variation of PFR with pressure

In our earlier experiments, it was discovered that pressure is the most influential parameter. From our previous studies we found that this high RF power provides vertical sidewalls with better etch bias. Hence, in this experiment, FPE waveguides were patterned by RIE at different pressures keeping RF power and oxygen flow rate fixed at 300 W and 18 sccm respectively. As oxygen is the main etchants for polymers, the above flow rate was kept fixed. The maximum etching time for all of the experiments was 35 min.

In order to study the evolution of PFR, AFM was employed for the samples treated with various RIE pressures ranging from 50 mT to 95 mT. It was found that as the pressure increases the standard deviation (sd) of PFR decreases linearly as shown in Fig.7.1. It is noteworthy to mention that the SWR variation with pressure is opposite to PFR variation i.e. with the increase of pressure SWR increases and PFR

decreases. The opposite variation of PFR with pressure can be explained by a mechanism proposed in the following section.

Fig. 7.1. Variation of PFR with pressure.

At low pressure (50 mT), the reactant particles are normally incident to the planar film [1] due to fewer collisions and longer mean free path. Therefore, the particles after hitting the morphology of the surface are reflected towards the valleys [1] as shown in Fig. 5.7 (a). Since the sticking coefficients of the incident particles are smaller than those of the reemitted particles [71], the particles reemitted to the valleys of the morphology are stuck and thus, etching takes place at the reemitted positions at the valleys. Hence, due to etching at the valleys the roughness increases as the valleys etch downwards, which is indicated by an arrow as shown in Fig. 5.7 (b).

At higher pressure, the particles are obliquely incident to the planar surface [1] due to vigorous collisions and shorter mean free path. Due to the oblique incidence of the etchant particles, the peaks of the morphology block most of the particles compared to the valleys as shown in Fig. 5.6 (a). This effect is known as

shadowing effect. A shadowing model developed by Drotar *et al.* [48] suggests that the shadowing sticking coefficient, S_S is greater than that of the reemitted sticking coefficient, S_R . In other words, the reemission effect during shadowing effect is negligible or weak. At higher pressure, the particles are less energetic and hence, the incoming particles at the point of incidence are etched due to higher sticking coefficient rather than reemission, which are indicated by arrows as shown in the schematic diagram, Fig. 7.2.

Fig. 7.2. Etching at the peaks due to shadowing effect. The sticking coefficient for shadowing effect, $S_S = 1$, whereas the sticking coefficient for reemission effect, $S_R = 0$. Etching occurs in downward directions indicated by an arrow.

7.2 Evolution of PFR with time at different pressures

Further study of roughness evolution of FPE planar films was carried out with the variation of etching time at different pressures ranging from 50 to 95 mT. Various samples with the identical conditions were exposed to the pure oxygen plasma and samples were etched at 5 min intervals. PFR was measured by AFM after each etching experiment. For all pressures (50 to 95 mT), during initial time period of etching ($t = 5$ min), the film surface is relatively flat consisting of randomly distributed “hillocks and valleys”, which are shown in Figs. 7.3 (a) and (b) to Figs. 7.

5 (a) and (b). Therefore, for all pressures the initial roughness data are excluded for the computation of PFR vs. etching time.

Fig. 7.3. AFM figures for 50 mT, 300W and 18 sccm O₂ flow rate and etching time, $t = 5$ min in (a) 2D and (b) showing the relatively flat surface having randomly distributed hillocks and valleys.

Fig. 7.4. AFM figures for 65 mT, 300W and 18 sccm O₂ flow rate and etching time, $t = 5$ min in (a) 2D and (b) showing the relatively flat surface having randomly distributed hillocks and valleys.

Fig. 7.5. AFM figures for 95 mT, 300W and 18 sccm O_2 flow rate and etching time, $t = 5$ min in (a) 2D and (b) showing the relatively flat surface having randomly distributed hillocks and valleys.

It can be further observed from Fig. 7.6 that initially the valleys coarsen together and form the bigger valleys and hence, the etched surfaces possess self-similar structure in time scale as described by Zhao *et al* [50] for Si etching. Viewing the 3D AFM images of Fig. 7.6, it can be postulated that the valleys shown in 2D images of Fig. 7.6 are the valleys in the morphology and peaks are the random distribution of hillocks. As the etching continues, the width and depth of the valleys increase or decrease based on different pressure scenarios. In case of 50 mT pressure, the initial roughness increases from 10 min to 20 min etching time, subsequently the roughness decreases up to the etching time of 30 min and finally, it increases at 35 min as shown in Fig. 7.7. It is interesting to point out that the nature variation of PFR

with etch time is similar to the nature of variation of SWR vs. depth at low pressure, which has been described in Chapter 6. The initial increase of planar film roughness in Fig. 7.7 is due to the presence of noise as has been described by Drotar *et al* [48] and the decrease of roughness is due to the shadowing effect. The final increase of roughness in Fig. 7.7 is due to the first order reemission effect. Since the variation of PFR with etching time is similar to the variation of SWR with vertical depth at lower pressure effect, therefore, the same mechanism described in Chapter 6 can be applied to the explanation of PFR variation with etch time. At 65 mT pressure, PFR also increases to a maximum and then decreases continuously as shown in Fig. 7.8. This variation PFR with etching time at 65 mT is similar to the variation of SWR with vertical depth at 65 mT pressure which has been described in Fig. 6.5, Chapter 6. At higher pressure of 95 mT, the PFR vs. etching time varies exactly in the same manner as SWR varies along the vertical depth as shown in Fig. 7.9. Though the data points for Fig. 7.9 are scattered, similar fitting trend has been considered as has been considered for Fig. 6.5 for 95 mT pressure in order to correlate the SWR vs. etch depth with PFR vs. etch time. It can be observed from 2D and 3D AFM figures, shown in Fig. 7.10 that self-similar structures are evolved with etching time as described earlier for 50 mT pressure. It is interesting to note that the widths of the peaks were reduced and that of valleys were widened, if we assume the valleys as the valleys and the grain heights as peaks. This is only possible because of the continuous shadowing effect mechanism which has been already described in Chapter 6. However, in comparison with the self-similar structures from Fig. 7.4 for 50 mT case, it can be observed that the depth of the valleys were increased towards

higher etching times, thus supporting our earlier explanation for the reemission effect mechanisms.

(i)

(ii)

(a) for $t = 10$ min

(i)

(ii)

(b) for $t = 15$ min

(i)

(ii)

(c) for $t = 20 \text{ min}$

(i)

(ii)

(d) for $t = 25 \text{ min}$

(e) for $t = 30$ min

(f) for $t = 35$ min

Fig. 7.6. AFM figures with the variation of etching time from $t = 10$ min to $t = 35$ min for 50 mT, 300W and 18 sccm O_2 flow rate in (i) 2D, showing hillocks and valleys and (ii) 3D, showing peaks and valleys.

Fig. 7.7. Variation of PFR with etching time at the pressure of 50 mT, 300 W RF power and oxygen flow rate of 18 sccm.

Fig. 7.8. Variation of PFR with etching time at the pressure of 65 mT, 300 W RF power and oxygen flow rate of 18 sccm.

Fig. 7.9. Variation of PFR with etching time at the pressure of 95 mT, 300 W RF power and oxygen flow rate of 18 sccm.

(i)

(ii)

(a) for $t = 10$ min

(i)

(ii)

(b) for $t = 15$ min

(i)

(ii)

(c) for $t = 20 \text{ min}$

(i)

(ii)

(d) for $t = 25 \text{ min}$

(e) for $t = 30$ min

(f) for $t = 35$ min

Fig. 7.10. AFM figures with the variation of etching time from $t = 10$ min to $t = 35$ min for 95 mT, 300W and 18 sccm O_2 flow rate in (i) 2D, showing hillocks and valleys and (ii) 3D, showing peaks and valleys.

7.3 Summary and conclusions

It was confirmed that with the increase of pressure, the PFR linearly decreases with an opposite trend of SWR. At low pressure the resulting higher roughness is due to the reemission of the reacting particles towards the valleys of the morphology and at higher pressure, the decrease in roughness is due to the shadowing effect. It has been experimentally proved that the variation of PFR with etch time is similar to the variation of SWR with etch depth in all of the pressure ranges. In other words, the evolution of PFR at low pressure is the combination of shadowing effect as well as reemission effect, and at higher pressure is due to the continuous shadowing effect.

Chapter 8

Monte Carlo Simulation Studies of Sidewall Roughness Evolved during Reactive Ion Etching

It has been described in previous chapters that one of the consequences during RIE in fabrication process is the evolution of sidewall roughness (SWR). It has also been discussed that SWR *varies* with vertical etch depth; in other words, SWR is not constant along the vertical etch depth of the waveguides and in fact, depends upon the etch parameters. In this chapter evolution of SWR will be further studied with the help of Monte Carlo simulation. Our main aims for this study are as follows.

- To study the evolution of SWR with time
- To study different etching modes such as shadowing and first order reemission etching that influence SWR

However, Monte Carlo simulation study in our case is limited to low pressure RIE. Moreover, the simulation covers the behavior of reactant particles within the surface features and the behavior of reactant particles within the plasma during RIE is beyond the scope of this research.

8.1. Monte Carlo Method

Monte Carlo method is an algorithm for solving various kinds of problems by using random numbers, which are the sequence of numbers with the property that it

is not possible to predict the number despite knowing all its previous values. In practice, a pseudo-random number, that is a sequence of numbers which appears to be random but in fact is generated by a finite computation, is used instead of random numbers. The method is useful for obtaining numerical solutions to problems which are too complicated to solve analytically.

In the most general form the algorithm of the Monte Carlo method can be presented in the following sequence [77]:

- a. Generate a random number (several random numbers).
- b. Select a new possible state of the system from the list of all possible states depending on the generated random number.
- c. Accept or reject the transition in the selected possible state depending in the generated random number.
- d. Repeat steps (a) – (c).

More information on Monte Carlo method can be obtained from the following references [78-81].

8.2. Simulation model

Monte Carlo simulation of SWR evolution was written in Matlab code, which can be found in Appendix A. In order to study the evolution of SWR, a three dimension model is designed instead of a two dimension model. Basically, the model simulates the incidents occurring within surface features during RIE.

In this model, low pressure RIE has been considered. At low pressure RIE, the mean free path of the gas particle is large. Hence, collision among particles within the surface feature can be neglected, thus enabling the etching particle to travel in a straight line until it hits the surface at another point. For low pressure RIE, the incoming etching particles are incident at more oblique angles with respect to the sidewall and at more normal angles with respect to the planar etch front [1]. The model assumes that the directions of the incoming etching particles have a uniform probability density function and the probability distribution of the direction has a constant value, which will be defined later.

In this model the particle hits at the surface of the target materials, which has to be etched. A given particle will either etch the surface at one point of incidence or is reemitted. For simplicity, the model assumed that the incident particle will be specularly reemitted. For specular reemission, the angle against the surface normal of the reemitted incident particle is the same as the angle of the incoming incident particle.

The probability of an incoming particle sticking to the surface of the target materials is s_0 ($0 \leq s_0 \leq 1$), where s_0 is called the zeroth-order sticking coefficient; while, n^{th} order reemitted particle is called as $(n+1)^{\text{th}}$ order particle. The sticking probability of an n^{th} order particle is s_n ($0 \leq s_n \leq 1$), and there is a probability of $1-s_n$ for which this particle does not stick, but reemits instead.

The model further assumed that the incident particle loses a considerably large amount of energy upon the first impact when it hits the surface of the target materials. Therefore, the incident particle is only allowed to reemit once, and the particle will stick if it hits the surface at another point after being reemitted ($s_1 = 1$).

Different etching modes are modeled by varying simulation parameter s_0 . Shadowing and first-order reemission etching models are simulated by setting $s_0 \approx 1$ and $s_0 \approx 0$ respectively.

Fig. 8.1. Schematic of simulation model

The schematic diagram of the model is illustrated in Fig. 8.1 and the simulation is elaborated as follows:

- The model is represented by an array of matrix ($a \times b \times c$)
- The matrix sizes in x , y , and the initial surface mean height in z direction are the input and the z size is set as maximum number of initial surface in z direction plus 2.
- The initial surface formed after the RIE step changes due to the arrival of particles. Therefore, the initial surface becomes an unknown factor in the simulation model. Thus, the initial surface, which is either absolute smooth or Gaussian type was varied.

- d. The absolute smoothness constitutes a flat initial surface which means, there is no variation of height in z direction, while Gaussian initial surface has normally distributed height in z direction.
- e. Each matrix cube has a value of 0, 1, and 2 which represent empty space, target particle and mask respectively.
- f. The mask covers half of the x - y plane, from top of the initial surface until the top x - y plane.
- g. The incident particle is introduced randomly on the top x - y plane which is not covered by the mask.
- h. The incident particle travels in straight line with a certain angle until it hits non-zero valued particle.
- i. The angle is defined by θ (the angle between the incident particle trajectory and z direction) and α (the angle between incident particle projection in x - y plane and x direction)
- j. θ value is uniformly distributed from 0 to a value of θ_{max} which can be varied as per the input, while α value is uniformly distributed from 0 to 2π .
- k. Although the value of θ_{max} can be varied according to the input, it should be noted that the model is applied for the low pressure RIE. According to Agarwal *et al.* incoming particles are incident at more oblique angle to sidewall and at almost normal incidence to planar surface [1] in low pressure. Thus, θ_{max} should have a narrow value (e.g. 5°)
- l. The general formula for the probability density function for the *uniform distribution* is

$$\begin{aligned}
 f(x) &= 0 & \text{for } x < a \\
 f(x) &= \frac{1}{b-a} & \text{for } a \leq x \leq b \\
 f(x) &= 0 & \text{for } x > b
 \end{aligned} \tag{1}$$

The total probability, $P = \int_a^b f(x)dx = 1$

The uniform distribution of direction of angle, α in x-y plane varies from 0 to 2π and of angle, θ in y-z plane varies from 0 to θ_{max} .

Hence, the probability density functions for the *uniform distribution* in x-y and y-z planes respectively are

$$f(\alpha) = p_1 = \frac{1}{2\pi - 0} = \frac{1}{2\pi} \quad \text{for } 0 \leq \alpha \leq 2\pi \tag{2}$$

and

$$f(\theta) = p_2 = \frac{1}{\theta_{max} - 0} = \frac{1}{\theta_{max}} \quad \text{for } 0 \leq \alpha \leq \theta_{max} \tag{3}$$

where θ_{max} is in radian.

The probability for θ_{max} is always either 0 (for a constant value) or 1 (for range of values). It is to be noted that probability density function is mentioned in this thesis, but not probability.

- m. At the point of incidence, if the incident particle hits the mask, it will be reemitted. However, if it hits the target particle, a random number is generated to decide whether the incident particle will etch (change the matrix value from 1 to 0) or reemit.

- n. If the generated random number has a value less than the input sticking coefficient, the incident particle will stick and etch after hitting the target particle.
- o. If the generated random number has a value more than the input sticking coefficient, the incident particle will not stick and reemit. Fig. 8.2 illustrates Monte Carlo simulation for etching.

Fig. 8.2. Monte Carlo simulation illustration: incident particle bounce off the surface and etches the surface elsewhere

- p. New etching particle can be introduced only when the particle etches the surface or is out of the matrix.
- q. Time was increased by 1 unit for every first time of simulation when the incident particle hits non-zero valued particle. The data of the matrix array were subsequently saved according to the input time interval. The simulation runs until N step is achieved and one step is equal to 1 unit of time

- r. When the simulation was completed, SWR of the saved data would be analyzed later. The simulation flow chart is shown in Fig. 8.3 on the next page.

8.3. SWR Analysis

SWR was analyzed by calculating root mean square (RMS) roughness in x direction of the model along the etch depth. The RMS roughness, denoted by w , is estimated as follows:

$$\langle w \rangle_N = \left\{ \frac{1}{N} \sum_{i=1}^N [h_i - \langle h \rangle_N]^2 \right\}^{\frac{1}{2}}$$

where h_i is the surface height at the i -th position in x direction with a total number of N positions, and $\langle h \rangle$ constitutes the average surface height which is given by the following equation.

$$\langle h \rangle_N = \frac{1}{N} \sum_{i=1}^N h_i$$

The calculated SWR was then plotted against the vertical etch depth (z direction) for further analysis.

Fig. 8.3. Flow chart of Monte Carlo Simulation

8.4. Evolution of SWR with Different Etching Modes

Simulations with different parameters were carried out to investigate the effect of different etching modes such as shadowing and first-order reemission

etching on SWR. These etching modes were simulated by varying the zeroth-order sticking coefficient (s_0) as simulation parameter. Shadowing and first-order reemission etchings were simulated by using $s_0 \approx 1$ and $s_0 \approx 0$ respectively, as mentioned in previous sections in this chapter.

Four different simulations, namely Simulation 1, Simulation 2, Simulation 3, and Simulation 4, were performed to study SWR created by different etching modes and the simulation parameters are mentioned below.

- Simulation 1 (Shadowing)
 - Matrix size : $150 \times 200 \times 300$
 - Initial surface : Gaussian
 - $\theta_{\max}$: 1°
 - s_0 : 1
- Simulation 2 (Shadowing and First-Order Reemission Etching)
 - Matrix size : $150 \times 200 \times 300$
 - Initial surface : Gaussian
 - $\theta_{\max}$: 1°
 - s_0 : 0.5
- Simulation 3 (Shadowing and First-Order Reemission Etching)
 - Matrix size : $150 \times 200 \times 300$
 - Initial surface : Gaussian

- $\theta_{\max}$: 1°
- s_0 : 0.2
- Simulation 4 (First-Order Reemission Etching)
 - Matrix size : $150 \times 200 \times 300$
 - Initial surface : Gaussian
 - $\theta_{\max}$: 1°
 - s_0 : 0.05

Extreme cases of shadowing and first-order reemission etching were represented by Simulations 1 and 2 respectively. Both Simulations 2 and 3 modeled a combination of shadowing and first-order reemission etching in which shadowing was the dominant etching mode in Simulation 2 and first-order reemission etching was the dominant etching mode in Simulation 3. The dominant etching modes in Simulation 2 and 3 were decided by comparing the total number of target materials being etched either by shadowing or first-order reemission etching and the simulation codes have been provided in a table in Appendix B. Results of simulations 1 - 4 are presented in next paragraph.

Fig. 8.4 shows the result of Simulation 1 in which SWR increases initially and becomes fairly constant along the etch depth when etching is shadowing dominated. Initial increase of SWR is due to the presence of noise and the details about noise will be discussed later.

Fig. 8.4. Simulation 1: SWR along vertical etch depth for $s_0 = 1$

Fig. 8.5. Simulation 1: x-y cross section at different vertical etch depth ($z = 50, 100, \text{ and } 150$) for $s_0 = 1$

It is shown in Fig. 8.6 that as the shadowing sticking coefficient decreases to 0.5, spreading of SWR was obtained from simulation 2 and SWR increased slightly as the etch depth increased. It is further obtained from Fig. 8.7 that as the etch depth increased the valley became slightly deeper compared to simulation 1 (Fig. 8.5) when the shadowing coefficient decreased. In other words, when the first order reemission was dominant, the depth of the valleys of the morphology increased.

Fig. 8.6. Simulation 2: SWR along vertical etch depth for $s_0=0.5$

Fig. 8.7. Simulation 2: x - y cross section at different vertical etch depth ($z = 50, 100$, and 150) for $s_0 = 0.5$

Fig. 8.8. Simulation 3: SWR along vertical etch depth for $s_0 = 0.2$

Fig. 8.9. Simulation 3: x - y cross section at different vertical etch depth ($z = 50, 100$, and 150) for $s_0 = 0.2$

As shown in Fig. 8.8 (Simulation 3), SWR was spread along the vertical depth of the waveguides as compared to Simulation 2 (Fig. 8.6) and the depth of the valleys (Fig. 8.9) increases with increasing etch depth as compared to depth of valleys for $s_0 = 0.5$ (shown Fig. 8.7) when the first order reemission is completely dominant.

Fig. 8.10. Simulation 4: SWR along vertical etch depth for $s_0=0.05$

Fig. 8.11. Simulation 4: x-y cross section at different vertical etch depth ($z=50, 100, \text{ and } 150$) for $s_0=0.05$

It is further observed from Fig. 8.10 that as the shadowing sticking coefficient is nearly zero, (Simulation 4) SWR increases as compared to Simulations 2 and 3

along the etch depths. It is also obtained from Fig. 8.11 that valleys were much deeper as the etch depth increases.

Fig. 8.12. Comparison of SWR along vertical etch depth of Simulations 1, 2, 3, and 4.

Fig. 8.13. Comparison of x-y cross section of Simulations 1- 4 at position $z = 100$

Fig. 8.14. Comparison of y-z cross section of Simulations 1-4 at position $x = 100$

Figs. 8.13 and 8.14 depict the sidewall morphology views in z and x directions respectively. It can be observed that as zeroth order sticking coefficient decreases, sidewall surface becomes rougher and deeper valleys are created since shadowing effect becomes less dominant because of the dominance nature of the first-order reemission effect.

In comparing all the simulation results, it was further found that SWR increased with a wider distribution spread as zeroth-order sticking coefficient decreased (Fig. 8.12). Therefore, this implied that with the decrease of zeroth-order

sticking coefficient the first-order reemission effect became more dominant than the shadowing effect which has been discussed in previous chapters. When the shadowing effect became less dominant compared to the first-order reemission etching, the behavior of the variation of SWR along the etch depth started from fairly constant to an increasing trend. In other words, near the shadowing regime, the variation of SWR along the etch depth was nearly constant and near the first-order reemission regime, the SWR variation increased along the etch depth. Moreover, SWR increased with a steeper slope along the vertical etch depth as the first-order reemission etching became more dominant.

When $s_0 \approx 1$, etching is dominated by shadowing effect and incoming particles will stick and etch the surface. At low pressure, the incoming particles are incident at more oblique angles to sidewalls, resulting in non-roughening etching. Considering the morphology of the surface, the peaks of the surface will receive more incoming particles as shown in Fig. 6.6 (a-b). Incoming particles will etch away the peaks and a smoother surface is formed. Thus, shadowing is a smoothening mechanism in etching.

When $s_0 \approx 0$, etching was dominated by reemission effect and incoming particles were not stuck, but were reemitted from the point of incidence to the valleys of the morphology and etch the surface. Hence, after reemission the valleys of sidewall surface receives more reemitted incoming particles compared to the peaks as shown in Fig. 6.6 (c). Therefore, the increase of roughness must be due to etching

at the valleys and is further due to reemission at the point of incidence after peaks are etched. Thus, a rougher surface with deeper valleys is evolved in this condition.

8.5. Variation of other simulation parameters

Besides zeroth-order sticking coefficient, there are two other simulation parameters that can be varied according to the input value keyed in the model. The two simulation parameters are the initial surface and θ_{max} (the maximum value of the angle between the incident particle trajectory and z direction).

Simulations with smaller matrix size were performed by separately varying the parameters as stated in the above paragraph. For the initial surface parameter, the input was varied either from absolute smoothness or Gaussian surface, while fixing the rest of the parameters. For the angle between the incoming particle and z direction, θ_{max} was varied with the input value of 5° , 4° , 3° , 2° , and 1° while fixing other parameters.

The objective of this project was to study the evolution of SWR with time and different etching modes. It was found that variation of both parameters provides no significant influence to the trends observed in the SWR evolution with time and different etching modes. Hence, both of these parameters have not been discussed in this report.

8.6 Comparison of simulated data with experimental results

The experimental data at low pressure (35 mT) RIE was compared with the simulation results. For this purpose, the variation of SWR plot along the vertical depth was redrawn here (Fig. 8.15) as has been stated in previous chapters. It was observed that SWR increases initially, becomes fairly constant (Region-I), and finally increases (Region-II) as shown in Fig. 8.15.

Fig. 8.15. SWR dependence on etch depth for pure O₂ at low pressure (35 mT).

In the following paragraph, experimental results are presented and discussed.

- (a) The experimental study assumes that SWR is proportional to the planar film roughness (PFR) of the etch front, and SWR recorded as a function of etch depth is treated as a tracking indicator of the etched surface roughness as a function of etch time. In other words, the assumption behind the above statements is that the etched SWR at a particular etch depth does not change as the etching continues further and further.

- (b) The experimental study proposed that SWR behavior obeys the shadowing effect at lower vertical depth while it obeys first-order reemission effect at higher etch depth (Fig. 8.15).

Simulation results carried out in this research work show that SWR becomes archive as the etch depth increases but does not vary with etch time, thus supporting the above experimental results discussed in (a).

Through Monte Carlo simulation study and reference to Figs. 8.12-8.14, it had been found that SWR increased initially because of noise and became constant along the etch depth as the etching was shadowing limited (sticking coefficient, $s_0 = 1$), which further supported the shadowing model by Drotar *et al* [71]. However, as the sticking coefficient, s_0 decreased along the etch depth, the SWR increased. In other words, reemission effect was more dominant, which supported the experimental finding, point b. The increase of SWR due to decrease of s_0 was attributed to the fact that the number of reemitted particles, N_r increased along the etch depth as etching continued, which is shown in Fig. 8.16 and N_s in Fig. 8.16 is the number of shadowing particles. As the shadowing became less dominant, N_s decreased. N_0 is the number of particles which neither contribute in shadowing nor in reemission and it does not have any impact on SWR. It is further obtained from Fig. 8.12 that when the sticking coefficient, s_0 decreased along the etch depth, the noise during initial etching also increase. Noises are the small undulations which are created because of the random arrival of incident particles during etching [73]. The increase of noise due to decrease of s_0 along the etch depth can be further due to

increase of N_R . Therefore, our simulation results agree well with the experimental results and *assertions*.

Fig. 8.16. Variation of fraction of ions with shadowing sticking coefficient, s_0 , where N_S , N_R and N_O are number of particles taking part in shadowing, reemission and neither shadowing nor reemission effects.

8.7 Summary and conclusions

Monte Carlo simulation study showed that as the etch time increased, at a particular etch depth, SWR was archived for later etch time for the new etch depths. The simulation study for shadowing model showed that SWR increased initially and became constant along the etch depth. The result therefore supported the assertions and mechanisms formulated in experimental observations for the shadowing region. It was further verified that as first-order reemission effect became more dominant SWR increased along the etch depth. The result was also in agreement with assertions and mechanisms formulated in experimental observations for the reemission region. It was observed that at lower s_0 , N_R was dominant whereas at higher s_0 , N_S was dominant. Combining these results, it could be concluded that

noise increased as s_0 decreased because of the increase of N_R , and SWR increased along the depth as s_0 decreased because of the increase of N_R .

Chapter 9

Reduction of roughness in polymer waveguides using O_2/N_2 gas mixtures during reactive ion etching

It has been reported that only pure oxygen plasma creates rougher devices [82] and the addition of nitrogen gas to oxygen plasma creates smoother devices. However, no quantitative information on roughness or smoothness has been reported. In this chapter, a systematic study of addition of nitrogen to oxygen gas was carried in order to solve the bottlenecks waveguiding/interconnect technology providing more vertical and smoother surfaces. Particularly, pressure effect on SWR of waveguides using O_2/N_2 plasma was studied and compared with the SWR results obtained using O_2 plasma. O_2/N_2 plasma etched films were characterized by ToF-SIMS to obtain the chemical information. Finally, optimum conditions for the fabrication of cladded waveguide are provided in this chapter.

9.1 Pressure effect on SWR

Pressure effect was studied by adding N_2 to O_2 plasma during RIE of FPE waveguides. Pressure was varied from 50 to 95 mT and the details of the RIE parameters are provided in Table 9.1. After RIE, Ti was stripped out by using Ti-etchant. SWR of the waveguides was measured by AFM in the tapping mode for all of the O_2/N_2 treated samples. It was observed that with the increase of pressure the standard deviation (sd) of SWR increases linearly as shown in Fig. 9.1. The increase of SWR with pressure is known as pressure effect and the mechanism which is

similar to the mechanism as has been described in previous chapters for pure O₂ plasma.

Fig. 9.1. Pressure effect on SWR in O₂/N₂ plasma

Table 9.1. Summary of RIE parameters in mixture O₂/N₂ plasma

Pressure (mT)	Power (W)	Gas flow rate (sccm)		SWR (nm) for O ₂ /N ₂ plasma	SWR (nm) for pure O ₂ plasma
		O ₂	N ₂		
50	300	18	46	32.451	34.1
65	300	18	46	34.666	36.6
95	300	18	46	41.551	49.1

9.2 Comparison of pressure effect on SWR treated with pure oxygen plasma

It can be seen from Fig. 9.2 that the SWR of sidewalls of waveguides (having the scanned surface area of 4.2 μm × 4.2 μm) treated with O₂/N₂ plasma is lower than that of the SWR in pure O₂ plasma. The results are also provided in Table 9.1. In other words, the addition of N₂ to O₂ plasma renders sidewall smoothness. The details about the increase of *smoothness* due to O₂/N₂ plasma can be further

explained by the surface characterizations described in section 9.4. Therefore, it was discovered that addition of N₂ to pure O₂ plasma *solves* one of the bottlenecks in passive device fabrications for OICs.

Fig. 9.2. Comparison of SWR with variation of pressures in pure O₂ and mixture of O₂/N₂ plasma.

9.3 Pressure effect of on variation of SWR

As mentioned in previous chapters that the SWR is neither constant along the depth of the waveguides nor can it be equated to the LER of the sidewall. Variation of SWR was measured by AFM at different pressures for the samples treated with O₂/N₂ plasma and details of the RIE parameters are provided in Table 9.1. SWR at different pressures was measured from top to the bottom of the sidewall at about vertical depth intervals of 400 nm (± 25 nm) for the scanning length of 4.2 μm. Fig. 9.3 shows the variation of SWR at vertical depth of the waveguides at different O₂/N₂ etching pressures. It was observed that at low pressure (50 mT) the variation of SWR in O₂/N₂ plasma is similar to the low pressure variation of SWR in O₂ plasma. Therefore, this variation of SWR can be expressed as the combination of both shadowing, and the first order reemission effects, and the details of the

mechanism have been described in the section 6.2 in Chapter 6. However, at 65 mT pressure in O_2/N_2 plasma the SWR declines continuously and this variation is similar to the SWR variation of 95 mT pressure in case of pure O_2 plasma. The detailed mechanism of this type of declining of SWR is known as continuous shadowing effect (CSE). At higher pressure of 95 mT, the decline of SWR is however, more prominent; in other words, the SWR declines *sharply* from 60.9 nm at 400 nm vertical depth to 25.2 nm at the vertical depth of 1600 nm only and thereafter, it remains almost constant up to the vertical depth of 2800 nm. After 2800 nm, the SWR values at the deep etched structures decreases further. The declining nature of SWR in O_2/N_2 plasma is better suited for the waveguide structures of up to intermediate depth than that of SWR variation in pure O_2 plasma. Therefore, the decline of SWR at higher pressure along the vertical depths can also be explained by the CSE as described for the pure O_2 plasma at 95 mT pressure, and the nature of *sharp* decline of SWR can be further and better explained by the surface

Fig. 9.3. Dependence of SWR on etch depth for O_2/N_2 plasma at 50 mT to 95 mT. All samples are etched at 300 W, O_2 and N_2 flow rate of 18 sccm and 46 sccm respectively.

characterization, which is described in section 9.4 below.

9.4 Surface characterization by SIMS

Samples treated with mixtures of O_2/N_2 plasma at various pressures are characterized by ToF-SIMS in order to obtain the chemical information from the surface. During RIE, the pressure in O_2/N_2 plasma are changed from 50 mT to 95 mT, keeping the RF power at 300 W, O_2 and N_2 gas flow rate at 18 and 46 sccm respectively. Finally, the results are compared with pure O_2 at higher pressure (95 mT) plasma treated samples. The reason for comparing higher pressure (95 mT) O_2 treated sample was that the variation of SWR was the largest in case of pure O_2 and hence, the effect on SWR and its variation can easily be realized at such a high pressure [83-84]. It was found that with O_2/N_2 plasma, the nitrogen containing bonds such as N^- , CN^- and CNO^- were furthermore observed in the planar films as shown in Figs. 9.5-9.7 as compared the high pressure O_2 plasma treated sample, shown in Fig. 9.4.

The depth profiles of O_2/N_2 plasma treated samples at various pressures were carried out and shown in Figs. 9.8-9.11. In this experiment, only CN^- peaks were chosen since the intensity of CN^- ions are higher as shown in the Figs. 9.5-9.7. It has been observed that at lower pressure (50 mT) the intensity of CN^- ions are higher at the interfaces and the intensity decreases with the sputtering time and remains more or less constant in subsequent etching times. In other words, the nitrogen containing bonds that were formed on the surface decreased initially with the depth from the surface and thereafter remained constant. The same phenomenon happens for the intermediate pressure (65 mT) and higher pressure (95 mT). Therefore, the decrease

of roughness at the sidewalls as well as in the planar films, which were described in previous sections, could be further due to the formation of nitrogen containing bonds.

Fig. 9.4. Negative ToF-SIMS surface spectra for pure O₂ samples at 95 mT, 300 W and 18 sccm O₂ flow rate.

Fig. 9.5. Negative ToF-SIMS surface spectra for pure O₂/N₂ treated samples at 50 mT, 300 W, 18 sccm, and 46 sccm O₂ and N₂ flow rate respectively.

Fig. 9.6. Negative ToF-SIMS surface spectra for pure O₂/N₂ treated samples at 65 mT, 300 W, 18 sccm and 46 sccm O₂ and N₂ flow rate.

Fig. 9.7. Negative ToF-SIMS surface spectra for pure O₂/N₂ treated samples at 95 mT, 300 W, 18 sccm and 46 sccm O₂ flow rate.

Fig. 9.8. Negative ToF-SIMS depth profile for pure O₂ samples at 95 mT, 300 W and 18 sccm O₂ flow rate.

Fig. 9.9. Negative ToF-SIMS depth profile for pure O₂/N₂ samples at 50 mT, 300 W, 18 sccm and 46 sccm O₂ and N₂ flow rate.

Fig. 9.10. Negative ToF-SIMS depth profile for pure O₂/N₂ samples at 65 mT, 300 W, 18 sccm and 46 sccm O₂ and N₂ flow rate.

Fig. 9.11. Negative ToF-SIMS depth profile for pure O_2/N_2 samples at 95 mT, 300 W, 18 sccm and 46 sccm O_2 and N_2 flow rate.

9.5 Optimum Conditions for cladded Waveguide fabrication

Based on the results and discussion obtained from pure O_2 and O_2/N_2 treated plasma treated samples, this section describes the optimum waveguide fabrication conditions in terms of sidewall smoothness.

- The addition of N_2 to O_2 plasma renders smoothness not only to the sidewall but also to the planar films.
- The low pressure plasma provides smoother sidewall area.

- For shallow etched structures (inorganic waveguides), low pressure provides the sidewall smoothness and for the deep etched structures (organic (e.g. polymeric) waveguides) higher pressure provides the smooth sidewalls.
- In general, addition of N_2 to O_2 plasma provides smoothness to the sidewalls compared to O_2 treated sidewalls; and low RIE pressure is better suited for shallow etched structures and high RIE pressure is more appropriate for intermediate to deep etched structures.

9.6 Summary and conclusions

Reduction of SWR was obtained for the waveguides treated with O_2/N_2 plasma as compared to pure O_2 plasma. Similar *pressure effect* was also obtained for the O_2/N_2 plasma treated waveguides. Furthermore, variation of SWR in case of O_2/N_2 plasma is similar to that pure O_2 plasma treated waveguides. Hence, the same mechanism can be used to explain the variation of SWR for waveguides treated with O_2/N_2 plasma. The reduction of SWR in case of O_2/N_2 plasma was further investigated by surface characterizations using ToF-SIMS. O_2/N_2 plasma treated thin films show that nitrogen containing bonds such as N^+ , CN^+ and CNO^+ are present in the planar etched thin films. Depth profiling of these films shows that the nitrogen containing bonds decreases from top to the deeper surfaces.

Chapter 10

Conclusions and Future Work

10.1 Conclusions

Three types of roughness evolved during RIE of FPE waveguides at different surfaces were classified and defined distinctly in order to remove the ambiguities which have been described so far in the literature. A methodology was developed to directly measure the SWR of the waveguides by AFM from the top to the bottom of the sidewalls. LER of different samples treated at various pressure conditions were also measured successfully by AFM and were compared with the corresponding SWR. It was confirmed that LER and SWR are different and should be treated as two different roughness parameters. The sd of roughness at the sidewall was measured by AFM. However, sd of SWR does not convey any information about the variation of SWR along the vertical depth of the waveguides. PFR of the RIE treated samples were also measured by AFM. It is noteworthy to mention that LER, SWR and PFR are all three different parameters in different planes of the surfaces.

FPE waveguides at different RIE conditions were fabricated successfully and the effects of RF power and pressure in the O₂ plasma have been investigated. Our study shows that higher RF power provides a more vertical profile and vertical etch rate decreases after reaching a maximum value as the RF power increases. With the increase of pressure the SWR increases and so does the vertical etch rate. The increase of SWR with pressure is known as *pressure effect*. This *pressure effect* can be explained by the angle of incidence of ions mechanisms. It is shown that a

smoother sidewall can be obtained at low pressure, which is due to the higher oblique to normal ions ratio at the sidewalls. Because of the shadowing effect of oblique ions and weak reemission, the resulting smoothness at the sidewall is obtained at the low pressure. However, the first order reemission is dominant at higher pressure and because of that the valleys of the morphology are etched providing higher roughness at the sidewalls.

Variation of SWR along the vertical depth of the waveguides was first observed by SEM, measured and confirmed directly by AFM. It was observed that the variation of SWR along the vertical depth of the waveguides does not vary significantly at all RF power ranges. The nature of variation of SWR remains almost the same in all power ranges. Systematic studies showed that SWR was not constant; in other words, variation of SWR was observed along the depth of the waveguides i.e. $SWR = SWR(z)$, where z represented the depth of the waveguides. The variation of SWR was significantly observed by varying the pressure. Our experimental findings indicate that SWR varies with etch depth, with opposite trends for low and high pressure etches at the critical depths. This is known as *Depth Effect*. This implies that O_2 low pressure RIE may provide smoother sidewalls for shallow polymeric structures, however, O_2 higher pressure RIE may be better suited for the deep etched structures. Furthermore, a complete description of etch roughening mechanisms based on the relative ratios of oblique and normal incidence of ions must consider the influence of etch depth in addition to that of pressure. Hence, pressure is found to be the most dominant factor to cause the variation of SWR along the depth of the waveguides.

The evolution of SWR of FPE waveguides using pure O₂ plasma was investigated. The variation of SWR along the vertical depth at lower pressure is due to the combination of both shadowing effect at which reemission effects are negligible showing the minimum or saturation of roughness values and the first order reemission effect. The increase of roughness for deep-etched structures at lower pressure must be due to reemission of etchants to the valleys of the morphology where the shadowing effect is completely negligible. However, variation of SWR at higher pressure is due to continuous shadowing effect both for peaks and valleys where the reemission is weak due to less energetic etchants. From the second order statistical roughness parameters, it was discovered that ACL along the vertical depth of the waveguides tracked SWR variation, suggesting that surface roughness evolution at the sidewalls during plasma etching possesses the same dynamics as that of etching of planar film. The average value of the roughness exponent α increases as the pressure increases, which suggests that higher pressure etching tends to produce surfaces which are locally smoother.

It was found that with the increase of pressure, the PFR linearly decreases and variation of PFR and SWR were opposite to each other. At low pressure the resulting higher roughness is due to the reemission of the reacting particles towards the valleys of the morphology and at the higher pressure, the decrease of roughness is due to the shadowing effect. It was experimentally proved that the variation of PFR with etch time was similar to the variation of SWR with etch depth in all of the pressure ranges. In other words, the evolution of PFR at low pressure is the

combination of shadowing effect as well as reemission effect and that at higher pressure is due to the continuous shadowing effect.

The Monte Carlo simulation study for shadowing model showed that SWR increased initially and became constant along the etch depth. The result is in agreement with experimental result for the shadowing region. It was further verified from the simulation that as first-order reemission effect became more dominant SWR increased along the etch depth. The result is also in agreement with experimental result for the reemission region. It was additionally discovered that at lower s_0 , N_R was dominant whereas at higher s_0 , N_S was dominant. Combining the above results, it can be concluded that noise increases as s_0 decreases because of the increase of N_R and SWR increases along the depth as s_0 decreases because of the increase of N_R .

Control of Roughness (Or Reduction of SWR) was obtained with the vertical profiles using O_2/N_2 plasma instead of using pure O_2 plasma. Similar *pressure effect* was also obtained for the O_2/N_2 plasma treated waveguides. Furthermore, variation of SWR in case of O_2/N_2 plasma was similar to that pure O_2 plasma treated waveguides. Hence, same mechanism can be used to explain the variation of SWR for waveguides treated with O_2/N_2 plasma. The reduction of SWR in case of O_2/N_2 plasma was further investigated by surface characterizations using ToF-SIMS. O_2/N_2 plasma treated thin films show that nitrogen containing bonds such as N^- , CN^- and CNO^- are present in the planar etched thin films. Depth profiling of these films shows that the nitrogen containing bonds decreases from top to the deeper surfaces.

10.2 Future Work

Future work can be divided into two parts. The first has the aim to study further REDUCTION of SWR using other techniques are as follows.

- Post-RIE processing of polymer waveguides can be performed to further reduce the SWR, e.g. heating the waveguides under minimal birefringence condition.
- Though optimum conditions can be applied to reduce SWR and hence to reduce total propagation loss, UV curable polymers can be used to further reduce the SWR.

The second recommends the application of our work in new concepts of waveguiding e.g. Photonic Crystals (PCs). The PCs are the periodic structures with high refractive index contrast within a unit cell. Due to the presence of photonic Band Gap (PBG), localization of light is possible by creating a defect. A linear defect in PC leads to Photonic Crystals Waveguide (PCW). These devices are also fabricated by photolithography and RIE process. Hence, the concept of SWR and its variation together with the mechanisms can be better realized in such PCWs and PCW-based devices.

References

1. N. Agarwal, S. Ponoth, J. Plawsky and P. D. Persans, J. Vac. Sci. Tech. A, **20**, 1587 (2002).
2. K. K. Lee, D. R. Lim, H. Luan, A. Agarwal, J. Foresi, and L. C. Kimerling, Appl. Phys. Lett. **77**, 1617 (2000).
3. J.W. Bae, W. Zhao, J. H. Jang, I. Adesida, A. Lepore, M. Kwakernaak, and J. H. Abeles, J. Vac. Sci. Technol. B **21**, 2888 (2003).
4. Y. Wang, Z. Lin, X. Cheng, C. Zhang, F. Gao, and F. Zhang, Appl. Phys. Lett. **85**, 3995 (2004)
5. R.G. Hunsperger, Integrated optics: Theory and Technology, Springer Series in Optical Sciences, Springer-Verlag Berlin Heidelberg, 1985
6. K. K. Lee, D. R. Lim, A. Agarwal, K. Wada and L. C. Kimerling, Proc. Mat. Res. Soc. Symp. **637**, E3.4.1 (2001)
7. D. Marcuse, The Bell Syst. Tech. J., **48**, 3187(1969).
8. J.P.R. Lacey and F. P. Payne, IEE Proc., **137**, 282 (1990).
9. F. P. Payne and J.P.R. Lacey, Opt. Quant. Electron., **26**, 977 (1994).
10. F. Ladouceur, J.D. Love and T.J. Senden, IEE Proc. Optoelectron., **141**, 242 (1994).
11. J. H. Jang, W. Zhao, J.W. Bae, D. Selvanathan, S. L. Rommel, I. Adesida, A. Lepore, M. Kwakemaak, J. H. Abeles, Appl. Phys. Lett. **83**, 4116 (2003).
12. N. T. Agarwal, *Optical waveguides for on-chip interconnect applications*, Ph.D. thesis (2002)

13. H. Tanobe, Y. Kondo, Y. Kadota, K. Okamoto and Y. Yoshikuni, IEEE Photon. Tech. Lett. **10**, 235 (1998)
14. J. Sarathy, S. Chandrasekhar, A.G. Dentai, and C. R. Doerr, IEEE Photon. Tech. Lett. **10**, 1763 (1998)
15. L. Eldada, SPIE OE magazine, (2002).
16. L. Eldada and L. W. Shacklette, IEEE J. Select. Topics Quantum Electron. **6**, 54 (2000)
17. L. Eldada, [on-line].Available:
<http://www.wmrc.com/businessbriefing/pdf/optical2001/tech/Telephotonics.pdf>
18. J. W. Kang, J. P. Kim, W. Y. Lee, J. S. Kim, J. S. Lee and J. J. Kim, J. Lightwave Tech., **19**(6), 872 (2001)
19. M. Usui, M. Hikita, T. Watanabe, M. Amano, S. Sugawara, S. Hayashida and S. Imamura, J. Lightwave Tech., **14**, 2338 (1996)
20. S. W. Ahn, W. H. Steier, Y.H. Kuo, M.C. Oh, H.J. Lee, C. Zhang and H.R. Fetterman, Opt. Lett., **27**, 2109 (2002)
21. Y. Hida, Y. Inoue and S. Imamura, Electron. Lett., **30**, 959 (1994)
22. M.B. J. Diemeer, L.H. Spiekman, R. Ramsamoedj and M.K. Smit, Electron. Lett., **32**, 1132 (1996)
23. T. Watanabe, N. Ooba, S. Hayashida, T. Kurihara, and S. Imamura, J. Lightwave Tech., **16** (6), 1049 (1998)
24. J. F. Viens, C. L. Callender, J. P Noad and L. Eldada, Opt. Fiber Commun. Conf., 2, 133 (2000)

25. C. L. Callender, J. F. Viens, J. P Noad and L. Eldada, Opt. Fiber Comm. Conf., **2**, 133 (2000)
26. J. F. Viens, C. L. Callender, J. P Noad and L. Eldada, IEEE Photon. Tech. Lett., **12**, 1010 (2000)
27. C. L. Callender, J. F. Viens, J. P. Noad and L. Eldada, Electron. Lett., **35**, 1839 (1999)
28. J. T Yardley, L. Eldada, K. M. T. Stengel, L. W. Shacklette, R. A. Norwood, C. Xu, and C. Wu, Proc. SPIE, 3005, **155** (1997)
29. A. Mukherjee, B. J. Eapen, and S. K. Baral, Appl. Phys. Lett. **65**, 3179 (1994).
30. L.A. Hornak, and T. W. Weidman, Appl. Phys. Lett. **62**, 913 (1993).
31. S. Suzuki, M. Yanagisawa, Y. Hibino and K. Oda, J. Lightwave Tech., **12**, 790 (1994)
32. C. F. Kane and R. R. Kerchnavek, IEEE Photo. Tech. Lett., **7**, 535 (1995)
33. D. Burdeaux, P. Townsend, J. Carr and P. Garrou, J. Electron. Mater., **19**, 1357 (1990)
34. P.B. Chinoy and J.Tajadod, IEEE Trans. Compo., Hybrids Manufact. Tech., **16**, 714 (1993)
35. J. Kobashi T. Matsusra, S. Shigekuni and T. Maruno, Appl. Opt., **37**, 1032 (1998)
36. H. Franke, G. Knabke, and R. Reuter, Proc. SPIE, **682**, 191 (1986)
37. J. Kobashi, Y. Inoue, T. Matsusra, and T. Maruno, IEICE Trans. Electron., **E81-C**, 1020 (1998)

38. J. Kobayashi, T. Matsuura, Y. Hida, S. Sasaki and T. Maruno, J. Lightwave Tech., **16**, 1024 (1998)
39. A. J. Beuhler, D.A. Wargowski, K.D. Singer and T. Kowalczyk, IEEE Trans. Compon., Packag. Manufact. Tech., **B-18**, 234 (1995)
40. J. –J. Kim, and J. –W. Kang, Proc. SPIE, **3799**, 333 (1999)
41. H. J. Lee, M. H. Lee, M. C. Oh, J. H. Ahn and S. G. Han, J. Polym. Sci, Part A: Polym. Chem., **37**, 2355 (1999)
42. M. C. Oh, M. H. Lee, J.H. Ahn, H.J. Lee and S. G. Han, Appl. Phys. Lett, **72**, 1559 (1998)
43. J. H. Ahn, H. J. Lee, W. Y. Hwang, M. C. Oh, M. H. Lee, S. G. Han, H. G. Kim, and C. H. Yim, IEICE Trans. Electron., **E82-C**, 354 (1999)
44. Y. H. Min, M. H. Lee, J. J. Ju, S. K. Park and J. Y. Do, IEEE J. Select. Topics Quantum Electron. **7**, 806 (2001)
45. S. Imamura, R. Yoshimura and T. Izawa, Electron. Lett., **27**, 1342 (1991)
46. N. Agarwal, S. Ponothe, J. Plawsky, and P. D. Persans, Appl. Phys. Lett. **78**, 2294 (2001).
47. M. Ariga, Y. Sekido, A. Sakai, T. Baba, A. Matsutani, F. Koyama, and K. Iga, Jpn. J. Appl. Phys., Part 1 **39**, 3406 (2001).
48. J. T. Drotar, Y. –P. Zhao, T. –M. Lu, G. –C. Wang, Phys. Rev. B **61**, 3012 (2000).
49. Y. P. Zhao, J. T. Drotar, G. C. Wang, and T. M. Lu, Phys. Rev. Lett. **82**, 4882 (1999).
50. K. Walch, A. Meyyappan, S. Muckenhirn and J. Margail, Proc. SPIE, **4344**, 726 (2001)

51. A.J. Steckl and P.H. Yin, Appl. Phys. Lett., **60**, 1966 (1992)
52. K. Hosomi, M. Shirai, K. Hiruma, J. Shigeta, T. Katsuyama, IEICE Trans. Electron. E79-C (1996) 1579.
53. A.Matsutani, F. Koyama, and K. Iga, Appl. Phys. Lett. **66**, 64 (1995).
54. Y. Martin and H. K. Wickramasinghe, Appl. Phys. Lett. **64**, 2498 (1994)
55. Snyder A.W., and Love J.D. (1983). *Optical waveguide theory*, London ; New York : Chapman and Hall
56. J.A. Ogilvy and J.R. Foster, J. Phys. D: Appl. Phys. **22**, 1243 (1989)
57. C.Y. Poon and B. Bhusan, Wear, **190**, 76 (1995)
58. V. N. Koinkar and B. Bhushan, J. Appl. Phys., **81**, 2472 (1997)
59. S. Ganti and B. Bhushan, Wear, **180**, 17 (1995)
60. D. G. Hall, Opt. Lett., **6**, 601 (1981)
61. R.C. Hewson-Browne, P.C. Kendall and D.A. Quinney, IEE Proc., **136**, 281 (1989)
62. A. Yegorov, Proc. SPIE, **4750**, 192 (2002)
63. M. V. Bazylenko and M. Gross, J. Vac. Sci. Tech. A, **14**, 2994 (1996).
64. M. V. Bazylenko, M. Gross and M. Faith, Appl. Phys. Lett., **69**, 2178 (1996).
65. W.-S. Pan and A. J. Steckl, J. Electrochem. Soc. **137**, 213 (1990).
66. X. Mao, W. Chan, M. Shannon, R. Russo, J. Appl. Phys. **74**, 4915 (1993).
67. C. Momma, S. Nolte, G. Kamlage, F. Von Alvensleben, A. Tunnermann, Appl. Phys. A **67**, 517 (1998).
68. P. Pronko, S. Dutta, J. Squier, J. Rudd, D. Du, G. Mourou, Opt. Commun. **114**, 106 (1995).
69. J. Macklin, J. Kmetec, C. Gordon III, Phys. Rev. Lett. **70**, 766 (1993).

70. A. Voevodin, M. Donley, J. Zabinski, Surf. Coat. Technol. **92**, 42 (1997).
71. J. T. Drotar, Y. -P. Zhao, T. -M. Lu, and G. -C. Wang, Phys. Rev. B **62**, 2118 (2000).
72. J. W. Kang, J. S. Kim and J. J. Kim, Jap. J. Appl. Phys 40 (2001) 3215
73. J. H. Yao, and H. Guo, Phys. Rev. E, **47** 1007 (1993).
74. F. Ladouceur, J.D. Love, and T.J. Senden, *Electron. Lett.* **28**, 1321 (1992).
75. G. Rasigni, F. Varnier, M. Rasigni, J. P. Palmari, and A. Llebaria, *Phys. Rev. B* **25**, 2315 (1982).
76. Y. Zhao, G.-C. Wang and T.-M. Lu, *Characterization of Amorphous and Crystalline Rough Surface - Principles and Applications* **37**, Academic Press, (2001).
77. A. Touzik, H. Hermann, and K. Wetzig, Comp. Mat. Sci., **28**, 134 (2003)
78. I. Manno, *Introduction to the Monte-Carlo Method*, Akademiai Kiado (1999)
79. K. Ohno, *Computational materials science : from ab initio to Monte Carlo methods*, Springer (1999)
80. G. S. Fishman, *Monte Carlo : concepts, algorithms, and applications*, Springer-Verlag (1996).
81. D. Frenkel, and B. Smit, *Understanding molecular simulation: from algorithms to applications*, Academic Press, San Diego (2002).
82. K. Taguchi, T. Ueguchi and M. Ikeda, Technical Digest of CLEO, 2000, Nice France, Europe.
83. S. K. Pani, C. C. Wong, and K. Sudharsanam, Appl. Phys. Lett., 85 1295 (2004)

84. S. K. Pani, C. C. Wong, K. Sudharsanam, C. S. Premachandran and M. K. Iyer, J. Vac. Sci. Technol. (2006) (Accepted).

List of Publications

Journals

1. S. K. Pani, F. Tjiptoharsono, C. C. Wong, "Monte Carlo Simulation of Sidewall Roughening Evolved during Reactive Ion Etching," Communicated.
2. S. K. Pani, C. C. Wong, K. Sudharsanam, C. S. Premachandran and M. K. Iyer, "Evolution of variation of sidewall roughness of polymeric waveguides during reactive ion etching," **J. Vac. Sci. Technol.B** 24 (2006) p.163.
3. S. K. Pani, Y. Quiling , C. C. Wong, D. K. Y. Low, and X. Zhang, "Fabrication of buried hybrid sol-gel optical waveguides by femtosecond laser direct writing," **Thin Solid Films** 504 (2006) p. 336.
4. S. K. Pani, C. C. Wong, K. Sudharsanam, and V. Lim, "Direct measurement of sidewall roughness of polymeric optical waveguides," **Appl. Surf. Sci.** 239 (2005) p. 445.
5. S. K. Pani, C. C. Wong, and K. Sudharsanam, "Variation of sidewall roughness of polymeric waveguides during reactive ion etching," **Appl. Phys. Lett.**, 85 (2004) p. 1295
6. S. K. Pani, C. C. Wong, K. Sudharsanam, S. G. Mhaisalkar, V. Lim, S. Mohanraj, and P. V. Ramana, "Effect of Process Parameters on Sidewall Roughness in Polymeric Optical Waveguides," **Thin Solid Films**, 462– 463 (2004) p. 471

Conferences

1. D. K. Y. Low, X. Zhang, X. Hong, G.C. Lim, S. K. Pani and C.C. Wong, "Direct Writing of Embedded Waveguides in Hybrid Organic-Inorganic Sol-Gel by Femtosecond Laser Pulses," *International Congress on Application of Laser and Electro-Optics, Florida, USA (2005)* (To be presented).
2. S. K. Pani, C. C. Wong, K. Sudharsanam, C. S. Premachandran, M. K. Iyer, P. V. Ramana, and V. Lim, "Pressure and depth dependence of sidewall roughness of polymer optical waveguides during reactive ion etching," *In Proc. of Electronics Packaging Technology Conference, Singapore, December (2004)* p. 381.
3. S. K. Pani, C. C. Wong, K. Sudharsanam, S. G. Mhaisalkar, and P. V. Ramana, "Development of 16-channel Polymeric Arrayed Waveguide Grating," *In Proc. of Semi Technology Symposium, Singapore, August (2003)* p. A5

Appendix A

1. % SIDEWALL ROUGHNESS EVOLUTION (3D)

```

step = input('Number of step: ');
t = 0;

while t <= step

    if t ~= 0 & (tmax+t/int - ceil(tmax+t/int)) == 0
        for i = 1:nx
            for j = 1:ny
                for k = 1:zmax
                    mat(i,j,k,(tmax+t/int)) = matrix(i,j,k);
                    etch1(tmax+t/int) = netch1;
                    etch2(tmax+t/int) = netch2;
                    mask1(tmax+t/int) = nmask1;
                    mask2(tmax+t/int) = nmask2;
                    reflect(tmax+t/int) = nreflect;
                end
            end
        end
    end

    Start_V1 % calls file from Start_V1.m
    Trajectory_V1 % calls file from Trajectory_V1.m
    t = t + 1;

    if matrix(xhit, yhit, zhit) == 2
        nmask1 = nmask1 + 1;
        Specular_V1 % calls file from Specular_V1.m
        Trajectory_V1 % calls file from Trajectory_V1.m
    end
end

```

```
if matrix(xhit, yhit, zhit) == 2
    nmask2 = nmask2 + 1;
    continue
elseif matrix(xhit, yhit, zhit) == 1
    matrix(xhit, yhit, zhit) = 0;
    netch2 = netch2 + 1;
end
elseif matrix(xhit, yhit, zhit) == 1
    if rand < p
        matrix(xhit, yhit, zhit) = 0;
        netch1 = netch1 + 1;
    else
        nreflect = nreflect + 1;
        Specular_V1                                % calls file from Specular_V1.m
        Trajectory_V1                             % calls file from Trajectory_V1.m
        if matrix(xhit, yhit, zhit) == 2
            nmask2 = nmask2 + 1;
            continue
        elseif matrix(xhit, yhit, zhit) == 1
            matrix(xhit, yhit, zhit) = 0;
            netch2 = netch2 + 1;
        end
    end
end
end
end

end
```


2. % INITIAL POSITION AND ANGLE OF INCIDENT PARTICLE (Start.m)

$x = 0.5 + (nx - 1) * \text{rand};$

$y = ny/2 + 0.5 + (ny/2 - 1) * \text{rand};$

$z = z_{\text{max}} - 0.5;$

$\alpha = 360 * \text{rand};$

$\text{teta} = \text{tetamax} * \text{rand};$

$v = [(\sin(\text{teta} * \pi / 180) * \sin(\alpha * \pi / 180)) (\sin(\text{teta} * \pi / 180) * \cos(\alpha * \pi / 180)) (-\cos(\text{teta} * \pi / 180))];$

3. % TRAJECTORY OF INCIDENT PARTICLE (Trajectory_V1.m)

```

while matrix(ceil(x), ceil(y), ceil(z)) == 0
    x = x + vel*v(1);
    y = y + vel*v(2);
    z = z + vel*v(3);
    if x<0.5 | x>(nx-0.5) | y<0.5 | y>(ny-0.5) | z<0.5 | z>(zmax-0.5)
        break
    end
end

if x<0.5 | x>(nx-0.5) | y>(ny-0.5) | z>(zmax-0.5)
    continue
elseif y<0.5
    disp('not enough target particle in y direction')
    break
elseif z<0.5
    disp('not enough target particle in z direction')
    break
end

xhit = ceil(x);
yhit = ceil(y);
zhit = ceil(z);

x = ceil(x - vel*v(1));
y = ceil(y - vel*v(2));
z = ceil(z - vel*v(3));

```

4. % SPECULAR RE_EMISSION (Specular_V1.m)

```
if (x+1)>nx
    nx1 = [0 0 0];
else
    if matrix((x+1), y, z) ~= 0
        nx1 = [-1 0 0];
    else
        nx1 = [0 0 0];
    end
end
```

```
if (x-1)<1
    nx2 = [0 0 0];
else
    if matrix((x-1), y, z) ~= 0
        nx2 = [1 0 0];
    else
        nx2 = [0 0 0];
    end
end
```

```
if (y+1)>ny
    ny1 = [0 0 0];
else
    if matrix(x, (y+1), z) ~= 0
        ny1 = [0 -1 0];
    else
        ny1 = [0 0 0];
    end
end
```

```
if (y-1)<1
    disp('not enough particle in y direction')
    break
else
    if matrix(x, (y-1), z) ~= 0
        ny2 = [0 1 0];
    else
        ny2 = [0 0 0];
    end
end

if (z+1)>zmax
    nz1 = [0 0 0];
else
    if matrix(x, y, (z+1))~=0
        nz1 = [0 0 -1];
    else
        nz1 = [0 0 0];
    end
end

if (z-1)<1
    disp('not enough particle in z direction')
    break
else
    if matrix(x,y, (z-1)) ~= 0
        nz2 = [0 0 1];
    else
        nz2 = [0 0 0];
    end
end
```



```

no = nx1 + nx2 + ny1 + ny2 + nz1 + nz2;

if no == [0 0 0]
    if nx1 == [-1 0 0] & nx2 == [1 0 0]
        if (x+1) == xhit
            n = nx1;
        elseif (x-1) == xhit
            n = nx2;
        end
    elseif ny1 == [0 -1 0] & ny2 == [0 1 0]
        if (y+1) == yhit
            n = ny1;
        elseif (y-1) == yhit
            n = ny2;
        end
    elseif nz1 == [0 0 -1] & nz2 == [0 0 1]
        if (z+1) == zhit
            n = nz1;
        elseif (z-1) == zhit
            n = nz2;
        end
    else
        continue
    end
else
    n = no;
end

n = (2*dot(-v,n)/(n(1)^2+n(2)^2+n(3)^2)) .* n;
v = n + v;

x = x - 0.5;

```

$y = y - 0.5;$

$z = z - 0.5;$

5. % CONTINUE SIMULATION (Continue.m)

```
disp('Type 1 for Yes and 0 for No')
answer = input('Continue Monte Carlo simulation? ');

if answer == 1
    tmax = size(mat, 4);
    Sidewall_V1
elseif answer == 0
    tmax = size(mat, 4);
    disp('Done =')
else
    disp('Wrong input')
end
```

6. % RMS CALCULATION (RMS_V1.m)

```
time = input('Time: ');
```

```
for k = nz:-1:1
```

```
    for i = 1:nx
```

```
        h(k,i) = 0;
```

```
        for j = 1:ny
```

```
            if mat(i,j,k,time) == 1
```

```
                h(k,i) = h(k,i) + 1;
```

```
            else
```

```
                continue
```

```
            end
```

```
            if h(k,i) == ny
```

```
                kmin = k+1;
```

```
                break
```

```
            end
```

```
        end
```

```
        if h(k,i) == ny
```

```
            break
```

```
        end
```

```
    end
```

```
    if h(k,i) == ny
```

```
        break
```

```
    end
```

```
end
```

```
for k = nz:-1:kmin
```

```
    for i = 1:(ny-50)
```

```
        height(k,i) = h(k,i+25);
```

```
    end
```

```
end
```



```
for k = nz:-1:kmin
    for i = 1:(ny-50)
        hfinal(k,i) = height(k,i) - ny/2;
    end
end

rms = std(hfinal, 1, 2);
```

Appendix B

Table: Number of target particles being etched by shadowing, and the first order reemission etching mode for Simulations 1, 2, 3, and 4. N_S , and N_R are the number of target particles being etched by shadowing and first order etching mode respectively.

Simulation No.	t (1,000,000)	N_S	N_R
1	1	1,000,000	0
	2	2,000,000	0
	3	3,000,000	0
	4	4,000,000	0
2	1	500,222	257,136
	2	1,000,563	529,382
	3	1,500,019	810,680
	4	1,999,891	1,096,697
	5	2,500,366	1,388,286
3	1	200,480	360,361
	2	400,483	756,172
	3	800,203	1,166,613
	4	1,000,067	1,583,742
	5	1,200,742	2,009,972
	6	1,400,642	2,442,155
4	1	50,209	402,795
	2	100,272	806,924
	3	150,436	1,212,266
	4	200,456	1,621,919
	5	251,042	2,017,310
	6	300,684	2,424,531
	7	350,953	2,838,358
	8	401,163	3,222,896