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Silica Microchannel Fabrication Using Fluorine Based Rie With Alas A Mask

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Abstract: The silica microchannel fabrication process has been executed with Al thin films as the sacrificial layer during Reactive Ion Etching Process (RIE). CF_4/Ar and SF6/Ar plasmas has been used to investigate etch rate of Al thin films and silica wafer. Energy Dispersive X-ray (EDX) analysis has been used to investigate the chemical residues on the surface of silica wafer after RIE process. The transparency of the silica waveguide has also been inspected based on fringe visibility with the aid of the Twyman-Green Interferometer (TGI) for interferometry measurements. The results demonstrate that CF_4/Ar plasma etches silica faster than SF6/Ar plasma while keeping the etching rate of Al minimal. Residues are also less visible on the surface of the microchannel when applying CF_4/Ar plasma.

Key words:

INTRODUCTION

Microfluidics is one of the major application areas in Microsystems Technology (MST). Microfluidics aims at investigating and developing miniaturized devices which can sense, pump, mix or control small volumes of fluids or to be integrated with optical components to be applied in optofluidic applications (Psaltis, D., *et al.*, 2006). Microchannels are an important component of microfluidics and the primary process for microchannel fabrication is RIE (Reactive Ion Etching), which uses chemical and physical component of an etch mechanism to achieve anisotropic profiles, fast etch rates and dimensional control (Studer, V., *et al.*, 2002). Therefore a good establishment etching system needs to be achieved to maintain high etch rate for the required material to be etched and having a low etch rate for the sacrificial layer during RIE.

Aluminum is suitable to be used as a sacrificial layer due to its low reaction with plasma etching and easier to remove after RIE. The removal of aluminum after RIE is executed with Al etch which is an elemental compound of H_3PO_4:H_2O:CH_3COOH: HNO_3:/ 80:15:3:2. Flourin elements are known for its application of etching. Obtaining a vertical etching profile can be troublesome due to uncertain florin ion bombardment on the surface. Carrier gasses are needed for this vertical profile control. The ion bombardment also causes residues to remain on the surface especially at the depth and edges of the microchannel since these are the areas where direct contact occurs. Residues also remain on the surface of the aluminum during RIE but this is removed directly with Al etch. However, there are still aluminum residues remaining on the surface of the silica after RIE. Verifying the residues is executed usingthe Scanning Electron Microscope with an energy dispersive X-Ray analysis system (SEM/EDX). The visibility of the microchannel when illuminated with laser beam is analyzed with the Twyman-Green Interferometer Setup (TGI). Fringe Visibility was used to measure the contrast of bright and dark fringe when under the observation of the TGI setup. The fringe visibility will aid in determining the transparency of the silica microchannel. Transparent silica microchannels are important in interferometry applications. In this work, etching rates of Al and silica have been done using CF_4/Ar and SF6/Ar plasmas. Residues determination with the two types of plasma etching is via EDX. The visibility of the beam illuminated on the microchannel is analyzed using fringe visibility of the TGI Setup.

Experimental Details:

The silica microchannel was fabricated on silica wafer which has a thickness of 500 μm and a diameter of 100 mm. The complete microchannel fabrication process is shown in Figure 1. Silica wafer was first immersed in Buffer Oxide Etch (BOE) for 30 seconds. Sample wafer was then cleaned with acetone ($(CH_3)_2CO$) followed directly with isopropyl (IPA) and deionised water (DI). The cleaning process ensures the removal of residues, unwanted particles, smudges or fingerprints before the fabrication process. Al layers are then deposited on the surface of the bare silica wafer using the Physical Vapour Deposition (PVD). The Al deposition process involved the Al foil to be wrapped onto the heating coil in the process chamber and heated and vaporized on top of the silica wafer. The deposition process was executed under low pressure (10^{-5} to 10^{-6} torr). The deposition rate is closely related to the heating power which is controlled by the electric current. Thickness of one Al layer is approximately 700 Å. The Al layers act as the sacrificial layer during RIE process. Positive photoresist was

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then spin coated on the surface of the Al with a spin speed of 2500 rpm. Photoresist solution has to be uniformed on the entire layer of the Al surface. Pattern transfer of the microchannel is then transferred onto the sample with UV exposure. This step is highly important due to ensure that the exact same design of the microchannel is imprinted onto the photoresist. Al etch was then executed to remove the Al that has been exposed by the previous removal of the photoresist during the UV exposure. The exposed region of the Al is then etched with the inductively couple plasma (ICP RIE). There were two different set of parameters that were tested and analyzed to determine the most suitable fluorine based RIE for the fabrication of the silica microchannel. The main difference between the two set of parameters are the plasma gasses which were CF_4 and SF_6. Fluorocarbon (CF_4) when mixed with oxygen or argon acts as the plasma etchant to the surface of the silica wafer which also goes the same for sulfur hexafluoride (SF_6). The main parameter of the RIE process is stated in Table 1.

Table 1: RIE parameters

Parameter	Value
CF_4/SF_6	50 sccm
Ar	30 sccm
RF Bias	300 V
ICP Power	800 W

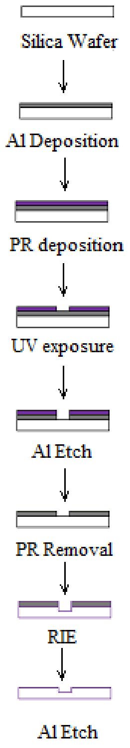


Fig. 1: Sample Preparation

Determining Remaining Residues:

During the RIE process, the plasma gases interact with the sample and form new chemical compounds, these compounds remain on the surface of the sample and may effect on the next processes for the sample. Therefore, the chemical residues on the surface of Al films and silica substrate are analyzed using Energy Dispersive X-ray analysis (EDX). Residues can become a hazardous distraction for silica waveguide applications dealing with light due to the importance of the transparency characteristic of the silica.

Transparency of the silica waveguide was also verified with a modified version of the Twyman-Green Interferometer (TGI). The TGI is a non-contact measuring instrument which performs microscale measurements with a resolution of up to 0.27 µm. The fringe produced from the TGI setup has a certain range of visibility. A transparent silica waveguide with less or no residues will provide high fringe visibility while too many residues can cause a low fringe visibility obtained from the TGI. A perfect fringe visibility would produce a '1' however it is very unlikely to obtain this due to the difficult overlapping of the beams and remaining residues on the surface of the microchannel. The intensity of the fringes have to be visible and measurable with the photodiode.

The fringe visibility of the TGI setup varies from 0.5 – 0.8 which is adequate enough for optical path difference calculation. Fringe visibility is calculated with Equation 1. The value of $I_{max}$is the intensity of the bright fringe while $I_{min}$is the intensity of the dark fringe. The photodiode will capture and record the intensity of the bright fringe followed by the dark fringe. Remaining residues will prevent the photodiode from measuring an accurate intensity measurement. This is where the importance of transparency plays a role.

$$V = \frac{I_{max} - I_{min}}{I_{max} + I_{min}} \quad (1)$$

RESULTS AND DISCUSSIONS

The Al and Silica etch rate was determined with the surface profiler. The results are shown in Table 2. The etch rate of the Al at three different locations are almost similar. CF_4/Ar and SF_6/Ar etches the Al layer at a rate of approximately 20 Å/s. However the etching rate of CF_4/Ar is nearly 20 Å/s more compared to the etching rate of SF_6/Ar.

Table 2: Aluminum and Silica Etch Rate with CF_4/Ar and SF_6/Ar = 50/30 sccm

	CF_4/ArEtch_rate A/s	SF_6/ArEtch_rate/s
Al 1	22.4	22.6
Al 2	23.3	23.1
Al 3	24.6	22.83
SiO_2	83.8	64.76

Figure 2 illustrates the EDX spectrum after RIE for CF4/Ar and SF6/Ar plasmas obtained at 10 keV. Figure 3 displays the list of chemical compounds on silica surface after RIE using CF4/Ar plasma. The figure shows that small amount of F (1.4%) and Al_2O_3 (0.74%) remain on surface of silica wafer. However, no carbon is present. Figure 4 displays the elemental compound analysis on silica surface after RIE using SF6/Ar plasma. The figure show that small amount of F (2.37 %) and Al_2O_3 (0.12) remains.

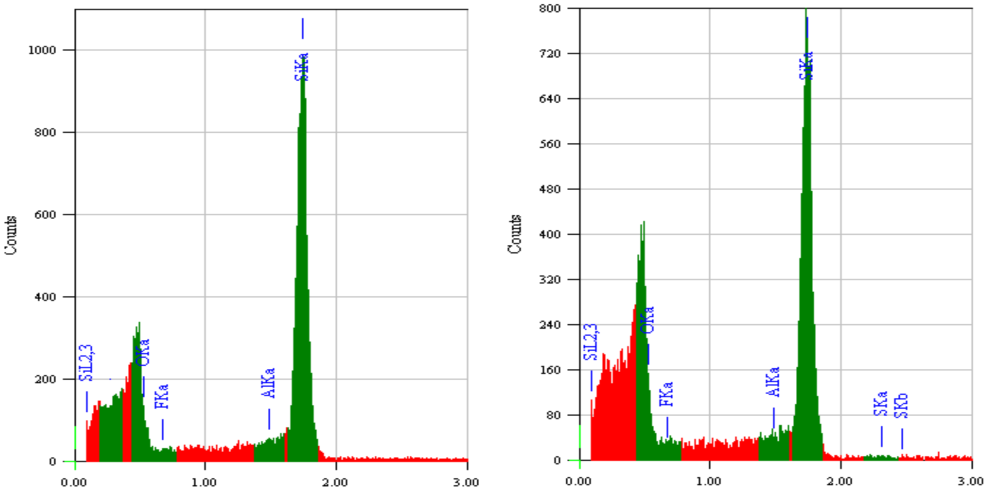
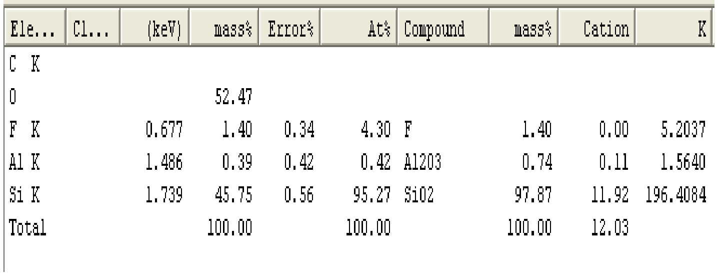
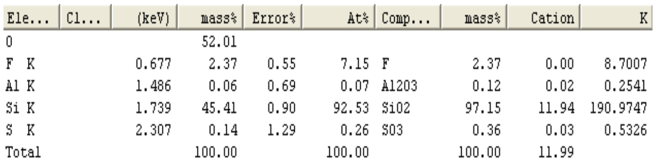


Fig. 2: EDX spectrum of elemental analysis for CF_4/Ar and SF_6/Ar plasma



Ele...	Cl...	(keV)	mass%	Error%	At%	Compound	mass%	Cation	K
C K									
O			52.47						
F K		0.677	1.40	0.34	4.30	F	1.40	0.00	5.2037
Al K		1.486	0.39	0.42	0.42	Al2O3	0.74	0.11	1.5640
Si K		1.739	45.75	0.56	95.27	SiO2	97.87	11.92	196.4084
Total			100.00		100.00		100.00	12.03	

Fig. 3: Element compounds on Silica Surface After RIE using CF_4/Ar obtained at 10 keV



Ele...	Cl...	(keV)	mass%	Error%	At%	Comp...	mass%	Cation	K
O			52.01						
F K		0.677	2.37	0.55	7.15	F	2.37	0.00	8.7007
Al K		1.486	0.06	0.69	0.07	Al2O3	0.12	0.02	0.2541
Si K		1.739	45.41	0.90	92.53	SiO2	97.15	11.94	190.9747
S K		2.307	0.14	1.29	0.26	SO3	0.36	0.03	0.5326
Total			100.00		100.00		100.00	11.99	

Fig. 4: Element compounds on Silica Surface After RIE using SF_6/Ar obtained at 10 keV

The transparency of the fabricated silica wafer was thenobserved with the TGI setup. Figure 5a displays the fringes of the silica waveguide etched with CF_4/Ar plasma. The outline of the microchannel with its reservoir can be seen with the sharp bend of the fringes due to the difference in depth.Figure 5b displays the fringes of the silica waveguide obtained with the SF_6/Ar plasma. Some of the fringes seem to be distorted due to remaining residues on the surface of the microchannel.

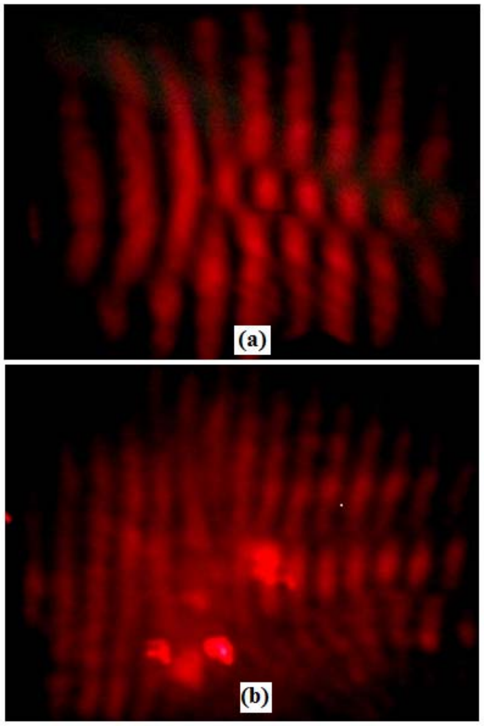
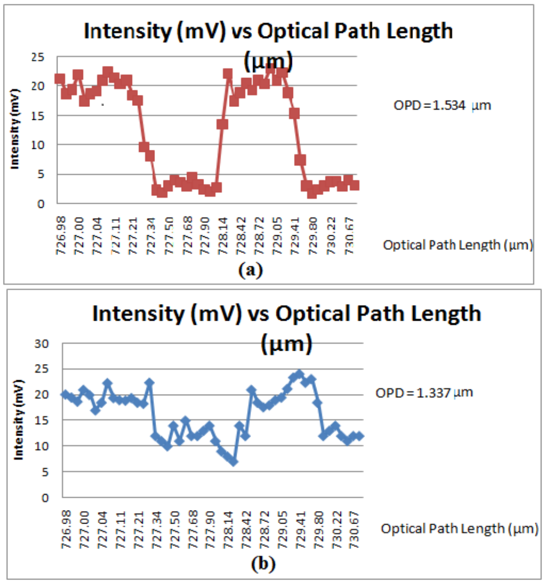


Fig. 5: (a) Fringes from TGI setup of the silica microchannel etched with CF_4/Ar plasma
(b) Fringes from TGI setup of the silica microchannel etched with SF_6/Arplasma

The remaining residue on the surface of the silica waveguide causes the visibility of the fringes to deteriorate. Based on the fringe intensity measurement which was recorded with a single photodiode as shown in Figure 6, fringe visibility for both CF_4/Ar and SF_6/Ar was 0.84 and 0.45. Figure 6b shows unclear outlines of the microchannel. This causes difficulty for the photodiode to detect the transition between a bright and dark fringe. When measuring fringe contrast, stability of the TGI setup plays an important role (Jaing, C.C., *et al.*, 2009). However, if the sample to be measured has a lot of residues, measurement will be difficult. Throughout the years, many methods of measuring thin films were executed. Some of the most common are the surface profiler, ellipsometer and Atomic Force Microscopy (AFM) (Piegari, A. and C. Masetti, 1985). The surface profiler needs the presence of grooves on the sample and it is a contact measuring instrument. The ellipsometer will have inaccurate measurements when measuring transparent samples due to internal reflection beam sensed by the detector. The TGI requires a smooth and highly reflecting surface for fringe acquirement. The fringe visibility determination in this work is however applied to a microchannel. The microchannel has depth difference similar to thin film but with a different surface profile. Fringes produce from the TGI based on the microchannel demonstrates this outline clearly and can also be seen in the intensity measurements. Results demonstrate that residues caused fringe visibility to fluctuate at a certain level that makes it difficult to obtain other possible measurements such as depth determination of the microchannel. In this work as can be seen in

Figure 6a the obtained fringe visibility demonstrates that SF_6/Ar is not suited for the fabrication of the silica waveguide when considering transparency characteristic for optical applications.



Conclusion:

This study has demonstrated that Reactive Ion Etching using CF_4/Ar plasma etches silica faster than SF_6/Ar plasma. The etch rate for Aluminum using CF_4/Ar and SF_6/Ar plasma is nearly equal to each other. CF_4/Ar etches silica faster while keeping the Al mask etching rate minimal, which implies that CF_4/Ar plasma is more suitable to be used when etching transparent silica wafers. EDX results show the presence of F and Al_2O_3 on silica surface after CF_4/Ar plasma while F, Al_2O_3 and SO_3 on silica surface after SF_6/Ar plasma. Fringe visibility determination with the TGI also demonstrates that RIE etching of SF_6/Ar is not suited for the silica microchannel fabrication.

REFERENCES

Abdelsalam, D.G. et al., 2011. Highly accurate film thickness measurement based on automatic fringe analysis. Optik - Int. J. Light Electron Opt. (2011)

Donati, S., 2008. Electro-Optical Instrumentation: Sensing and Measuring with *Lasers*: *Upper Saddle River: Prentice Hall*

Gravesen, P., J. Branebjerg, & O.S. Jensen, 1993. Microfluidics-A Review. J. Micromech. Microeng. 3: 168-182.

Hale, G.M. & M.R. Querry, 1973. Optical Constants of Water in the 200-nm to 200-µm Wavelength Region. Applied Optics, 12(3): 555-563.

Han, Y., N. Shikazono, & N. Kasagi, 2011. Measurement of liquid film thickness in a micro parallel channel with interferometer and laser focus displacement meter. International Journal of Multiphase Flow 3: 36-45.

Herk, A.V., 1972. Use of an XY oscilloscope in interferometric measurements of small displacements. Journal of Physics E: Scientific Instruments 1973 : 6.

Hernández, M., A. Juárez, & R. Hernández, 1999. Interferometric thickness determination of thin metallic films. Superficies y Vacío., 9: 283-285.

Jablonski, E.R., 1986. Interferometric Measurement of Angles. Measurement 4(4): 148-153.

Jaing, C.C., Y.L. Shie, C.J. Tang, Y.Y. Liou, C.M. Chang, & C.R. Yang, 2009. Determination of Displacement with a Piezoelectric Transducer Using Phase-Shift Algorithms. *OPTICAL REVIEW* 16(2): 170-172.

King, B.V., J.C. Kelly, & R.L. Dalglish, 1980. A strain monitor for ion implantation. Journal of Physics E: Scientific Instrument. J.Phys.E: Sci. Instrum.. 14.

Lunazzi, J.J., & M. Garavaglia, 1972. Fabry-Perot laser interferometry to measure refractive index of thickness of transparent materials or thickness of transparent materials. Journal of Phyics E: Scientific Instrument. J.Phys. E: Sci. Instrum. 6: 237.

Piegari, A. and C. Masetti, 1985.Thin film Thickness Measurement: A Comparison of Various Techniques. Thin Solid Films, 124: 249-257.

Psaltis, D., R. Stephen Quake & C. Yang, 2006. Developing optofluidic technology through the fusion of microfluidics and optics.NATURE pp: 442.

Šantić, B., 2010. Measurement of the refractive index and thickness of a transparent film from the shift of the interference pattern due to the sample rotation. Thin Solid Films 518: 3619-3624.

Shabana, H.M., 2004. Determination of film thickness and refractive index by Interferometry. Polymer Testing, 23: 695-702.

Sinha, J.K., & H.V. Tippur, 1997. Infrared interferometry for rough surfacemeasurements: application to failure characterization and flaw detection. Optical Engineering, 36: 8.

Studer, V., A. Pepin, Y. Chen & A. Ajdari, 2002. Fabrication of microfluidic devices for AC electrokinetic fluid. Microelectronic Engineering, 61-62: 915-920.

Tolansky, S., 1949. Multiple Beam Interferometry of Surfaces and Films, Oxford University press.

Vannoni, M., M. Trivi, & G. Molesini, 2006. Phase shift Interferometrywith a digital photocamera. European Journal of Physics Eur. J. Phys., pp: 117-124.

Yao, D.J., & P.Y. Chen, 2007. Room Temperature Microchannel Fabrication for Microfluidic System. *Proceedings of the 7th IEEE International Conference on Nanotechnology,* pp: 122-125.

Yeh, Y.L., 2008. Real-time measurement of glucose concentration and average refractive index using a laser interferometer. Optics and Lasers in Engineering, 46: 666-670.