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Joshi, Shivani; Savov, Angel; Shafqat, Salman; Dekker, Ronald

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Investigation of “fur-like” residues post dry etching of Polyimide using Aluminum hard etch mask

Shivani Joshi[a], Angel Savov[a], Salman Shafqat[c] and Ronald Dekker [a,b]

[a] *Department of Microelectronics, Delft University of Technology, Delft, The Netherlands*
[b] *Philips Research Eindhoven, Eindhoven, The Netherlands*
[c] *Department of Mechanical Engineering, Eindhoven University of Technology, Eindhoven, The Netherlands*

Abstract

The authors found that oxygen plasma etching of polyimide (PI) with aluminum (Al) as a hard-etch mask results in lightly textured arbitrary shaped “fur-like” residues. Upon investigation, the presence of Al was detected in these residues. Ruling out several causes of metal contamination that were already reported in literature, a new theory for the presence of the metal containing residues is described. Furthermore, different methods for the residue free etching of PI using an Al hard-etch by using different metal deposition and patterning methods are explored. A fur-free procedure for the etching of PI using a one step-reactive ion etch of the metal hard-etch mask is presented.

Keywords: polymers, reactive ion etching, sputtering, residues, PI residues

1. Introduction

Polyimides (PI) are thermosetting ring chain polymers comprising of repeating chains of imide monomers. Polyimides are extensively used in Micro-Electro-Mechanical Systems (MEMS) devices because of their outstanding properties such as excellent chemical resistance [1], high thermal stability [2], high mechanical strength [3][4] and good dielectric properties [5][6]. Polyimides are used as

*Corresponding author
Email address: shivani.joshi@philips.com (Shivani Joshi[a], Angel Savov[a], Salman Shafqat[c] and Ronald Dekker [a,b])

sacrificial layers, structural layers, isolation layers and as substrate material in flexible/stretchable electronic circuits [7]. A good example of a technology that makes use of many of the aforementioned properties of PI is the Flex-to-Rigid technology (F2R) [8]. F2R was developed to assemble complex electronic systems, such as for ultra-sound imaging, on the tip of smart-catheters. It enables the fabrication of arbitrary shaped Si islands containing sensors and electronics that are fabricated by through-wafer DRIE etching and that after etching remain suspended in the silicon wafer by tiny PI tabs (Fig. 1). The rigid silicon sensor islands are interconnected using stretchable metal interconnects embedded in the PI. Here the PI acts as a substrate as well as an isolation layer for the interconnects. Dry etching of this PI layer renders the embedded interconnect layer free. These free standing interconnects [9] can bend out of plane when stretched, increasing the stretchability of the device in Fig. 1.

Polyimides can be of two types: photosensitive and non-photosensitive. In this paper, only non-photosensitive polyimides will be addressed. Patterning of the PI can be achieved by using either a resist mask or a hard-etch mask. In general, the selection of a mask is based on firstly, the selectivity of the etch process towards the mask, and secondly, its ease of integration as a masking material in the flowchart. Polyimide is usually dry-etched in gas mixtures primarily containing oxygen. This makes the selectivity of the etch towards resist very poor so that consecutively the mask erodes rapidly during the plasma etching. The selectivity of the PI etch with a photoresist mask is 1:1, implying that the PI and resist layers are etched at an equal rate. This requires the resist mask to be at least as thick as the underlying layer of PI. Thick resists, however, have a limitation for high resolution lithography, for both positive and negative tone resists. An alternative that has been adopted in the microfabrication industry, is the use of a hard-etch mask. In this approach, a metal layer like Al, Ti, Mo etc. is used as a mask for the patterning of the underlying PI layer, where the mask is unaffected by the plasma chemistry of the PI etch. However, according to literature [10], the adhesion of a metal layer to an untreated surface of polyimide is poor due to its low specific surface energy. For a proper adhesion

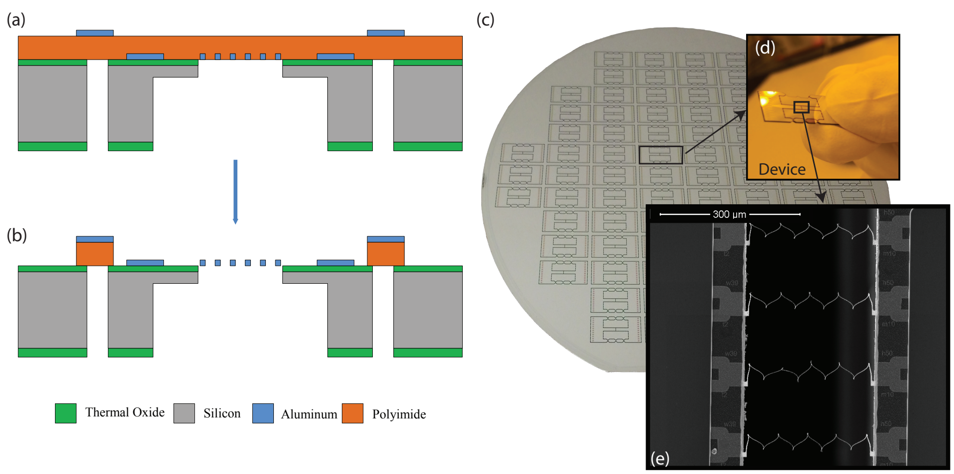

Figure 1: On the left, flowchart depicting the cross section of a wafer for the fabrication of free standing interconnect structures where (a) the metal interconnects are embedded in PI. After the reactive ion etch (RIE) etch of PI, (b) the interconnects are rendered free standing while the PI tabs remain. On the right, (c) a 6-inch test device wafer with the sacrificial PI tabs holding the devices together after the RIE etch. These tabs can be easily cut using laser to (d) isolate the device from the wafer. Upon stretching of the device, as seen in the SEM (e) the free standing interconnects bend out of plane to enhance the stretchability. The device is elaborately explained in the literature from Shafqat. S et. al. [9]

of the metal mask to the PI, a short Ar^+ sputter-etch of the polymer surface is recommended [10]. The metal layer is then patterned using a resist mask in the desired shape for the etching of PI. Prior to the etching of PI, the photoresist is stripped in acetone.

The plasma chemistry used for the etching of PI consists of oxygen radicals which break the unsaturated groups within the chemical structure of the polyimide. After etching of the polyimide using an Al metal mask, residues on the etched areas were observed. These residues were arbitrary in shape, and appeared light in texture, and for ease of understanding will be referred to as fur-like residues in this paper. Similar residues have been reported in the literature [11][12], wherein the silicon content in the self priming and silicone modified polyimides played a role. In this paper, however, we do not use either of the polyimides, thus eliminating the cause of the aforementioned occurence of the residues after reactive ion etching. The presence of these residues is undesirable as it leads to process instability and may interfere with the subsequent microfabrication steps. An interesting outlook towards these residues could be to harness them for the formation of nanowires, black silicon or in the Bosch process by controlling the residue size and density. However, it will be not be discussed in this paper as it is beyond the scope of this study.

The goal of this work is to investigate the origin/nature of these "fur-like" residues and provide solutions compatible with the microfabrication of PI-based electronics. Different hypothesis explaining the origin of the fur-like residues were tested by variations in process conditions and material analysis techniques (SEM, EDX, AFM and Raman). The experimental section describes the general processing flow employed to realize the PI-based devices/structures and motivation for the several short loop experiments and hypothesis. The subsequent sections discuss results for each hypothesis. Finally, devices are demonstrated that were fabricated through improved processing conditions, which eliminated the fur-problem, while maintaining compatibility with advanced MEMS-processing flows.

Experiment	Sample	PI surface preparation	Al mask deposition	Photoresist mask	Al mask etch	Hypothesis	Residues observed
1	R	Ar 100	Sputtering	**Yes**	PES	Metal redeposition	Yes
	A	Ar 100	Sputtering	**No**	Bl. PES		Yes
2	R	**Ar 100**	Sputtering	Yes	PES	Effect of PI surface preparation	Yes
	B	**N/a**	Sputtering	Yes	PES		No
3	R	Ar 100	Sputtering	Yes	**PES**	Metal mask inclusions	Yes
	C	Ar 100	Sputtering	Yes	**Dry**		No
4	D	N/a	**Sputtering**	Yes	PES	Mask deposition method	No
	E	N/a	**Evaporation**	Yes	PES		No

R	Reference sample
Ar 100	100 s Ar^+ sputter etch
PES	Wet chemical etchant (heated etch at 30°C) consisting of H_3PO_4, HNO_3 and CH_3COOH
Bl. PES	Blanket PES wet etch
Dry	Reactive ion etching (RIE) in a Cl_2 chemistry (5 mTorr) with 15 s overetch

Table 1: Short loop experiments designed for investigation of the PI residues with stated hypothesis and results for each experiment. Varying parameters of each experiment are depicted in red and bold text in the table.

2. Experiment

The presence of the residues was first observed during the fabrication of a device wafer with test structures to characterize free-standing interconnects [9] (Fig. 1). The wafer has a pre-defined and patterned 5 μm PECVD SiO_2 hard etch mask (Novellus PECVD concept one) on the backside. This back oxide mask is used to completely etch through 400 μm of Si (30 min in SF_6 + C_4F_8 DRIE plasma, STS ICP tool) on certain predefined locations such as to render the top metal structures free from the underlying substrate (Fig.1-a). These top metal structures are embedded in a 5.2 μm thick PI (Dupont 2611) layer which is spin coated at 3000 rpm for 45 seconds and cured at 275°C for 3 hours in a nitrogen oven (KOYO Thermo Systems Co.Ltd., Japan). This polyimide layer acts as both sacrificial layer to embed and protect the final free standing interconnect structures, as well as for sacrificial tabs (Fig. 1-b). These tabs keep the devices attached to the wafer and are released by laser or scalpel cutting (Fig. 1-c and d). To obtain a good adhesion between polyimide and Al hard etch mask, the surface of polyimide is sputter etched in an Ar^+ ion plasma (50 sccm Ar gas, 300 W, 100 s in Veeco 2 Nexus, UHV system), where the charged

Ar^+ ions accelerate towards the PI surface creating micro roughness on the surface as well as chemically modifying the PI structure [13]. This is done to ensure a good chemical as well as mechanical bonding of metals like Al, Cu, Ni etc. to PI, which can be challenging due to its low surface energy [14]. Next, a 200 nm layer of Aluminum is sputter coated (Veeco 2 Nexus, UHV system with 99.99999% Al target purity and 2 nm/sec deposition rate) on top of the PI layer. This Al hard etch mask is patterned using a photoresist (PR) mask (HPR504 positive resist, OCG Microelectronic Materials n.v) with a thickness of 1.7 μm. The resist mask is spin coated on an automated spin coater (EVG 150, Austria), exposed with an energy of 160 mJ (ASML PAS5500, The Netherlands) and developed in a TMAOH based developer. The Al hard etch mask is wet etched in a commercially available wet etchant PES 77-19-04 (heated etch at 30°C) consisting of phosphoric acid H_3PO_4, nitric acid HNO_3 and acetic acid CH_3COOH with an etch rate of 95 nm/min. Subsequently, the resist mask is removed in acetone (CT60 spin coater, Gyrset). This hard etch mask is used to etch the PI in an O_2 plasma (6 min RIE in STS ICP tool). After the PI etch, "fur-like" residues were observed under the scanning electron microscope (SEM) around the free standing as well as not-free standing structures (Fig. 2-a and 2-b). This sample will be denoted as the reference sample (R) in all the preceding experiments.

For the investigation into the origin of these residues four different experiments are prepared. Each experiment is tested for a different hypothesis. The sample substrates in all the experiments were 6- inch Si wafers coated with 1 μm of plasma enhanced chemical vapor deposited (PECVD) SiO_2 (Novellus PECVD concept one). In all the experiments, the samples have been spin coated with 5.2 μm PI and the PI is etched with similar conditions as the reference sample. The process variations for different experiments along with the hypothesis and their consecutive results are listed in table 1. The choice for these process variations and their stated hypothesis is elaborated further in the discussion section. Each experiment is repeated three times. The results were the same for each.

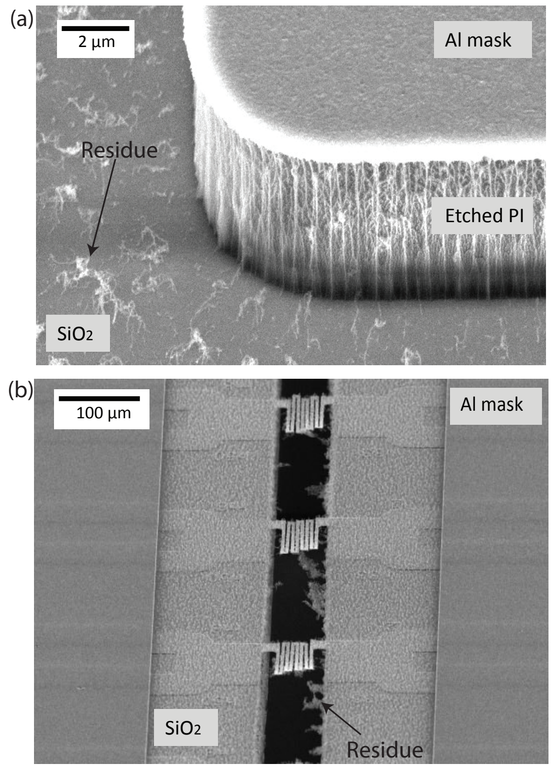

Figure 2: (a) Residue covered SiO_2 surface after reactive ion etch of PI with Al metal as the mask. (b) Free standing metal interconnect structures on the same wafer covered with "fur-like" residues after the PI etch.

In the first experiment, the formation of residues due to metal redeposition is tested. The Al hard etch mask in the sample A is processed without a PR mask and blanket wet etched in PES (heated at 30 °C). This sample is compared with the reference sample (R) after dry etching of PI. Presence of residues in sample A after the PI etch contradicts with the first hypothesis. Since the residues are not formed due to metal redeposition, the role of sputter etch in PI sample preparation is next investigated. Therefore, in the second experiment two samples with and without surface treatment are compared with each other. The first sample is processed similar to the reference wafer and the second sample (B) is prepared without the PI surface preparation. No residues are observed in the sample B after the dry etching of PI contrary to the reference sample. The role of Ar^+ sputter etch is hence confirmed for the presence of these residues, where omitting this etch could be a simple and straightforward solution. However, as reported in literature [10][15], surface treatment of PI is necessary to obtain good adhesion with metal layers. The accelerated Ar^+ ions create roughness by sputter etching the top few nms of the PI layer. This roughened PI layer is coated with the Al metal mask layer and some metal particles possibly remain embedded even after the wet etching. To test this hypothesis of "metal inclusions", a third experiment is conducted with two samples. The first sample is prepared similar to the reference sample (R) and in the second sample (C) the hard etch Al mask is dry etched (Cl_2 plasma, SPTS CPX clustertool) with an overetch of 15 s to remove any residues from the top surface of the PI. Residues are not observed in the sample C after PI etch. To further test the role of metal deposition method in the formation of the residues, a fourth experiment is conducted. Both the samples in this experiment are processed similarly without PI surface preparation, except the method of metal deposition. The metal mask in the first sample (D) is sputter deposited while the mask is deposited using evaporation on the second sample (E). No residues are observed post etching on both the samples D and E.

The samples are investigated by means of Scanning Electron Microscopy in combination with X-ray Microanalysis (SEM/EDX). Electron micrographs were recorded with a FEI Nova600 NanoSEM microscope (voltage: 500-30 keV, resolution: 1.2 nm at 30 keV) using secondary (SE) and/or backscattered electrons (BSE). Additionally EDX spectra and element-mappings were recorded using the Oxford Xmax 80 EDX system. Polyimide layers with after a 5 nm sputter treatment was analyzed with atomic force microscopy (AFM) to determine the surface roughness. The measurements were performed with a Bruker Dimension FastScan, using a Si probe in the tapping mode in air. Data processing was performed with MountainsMap 6.0 from Digital surf. The sample was scanned on two locations, scan range 4 x 4 μm^2. Before scanning the surface was cleaned using a nitrogen gun. Raman spectra of the residues were recorded using a Renishaw InVia Raman spectrometer, with 514 nm excitation (Coherent Innova 70C laser). Laser intensities varying from 100 mW up to 500 mW were used, in combination with a 100x magnification with a numerical apperture of 0.85. Plasma etching of PI and Al was conducted in an inductively coupled plasma-reactive ion etching (ICP RIE chamber with He backside cooling) machine (SPTS CPX clustertool).

3. Results and Discussions

To begin the investigation, the composition of the residues in the reference sample was determined (Fig. 3-a). This was done using an energy dispersive x-ray analysis (EDX), which is an elemental analysis technique that uses x-ray energies given off by the atomic structure of the emitting elements to determine the composition of the present elements [16]. The analysis showed that next to Si, O_2, F, C etc. from the substrate underneath, an additional peak of Al was present (Fig. 3-b). The result of the analysis leads to an immediate and first theory of metal redeposition from the Al hard etch mask during the plasma etching of PI. According to this theory, the Al metal mask gets bombarded by ions during plasma etching and redeposits on the surface of the PI resulting in micromasking which can lead to grass formation. Previously, in literature [17][18], the presence of these grass-like residues could only be observed in the proximity of Al structures. However, in the reference experiment the residues were observed to be homogeneously distributed over the etched areas, and even on areas that were distant from the metal mask (Fig. 2). To challenge this theory, sample A in experiment 1 was processed the same way as the reference sample except that the metal hard etch mask was completely removed by wet etching and the PI was blanket etched (Table 1). This excludes the possibility of the mask being redeposited on PI. However, SEM inspection again showed the presence residues over the SiO_2 surface on both the samples. Upon EDX analysis, the presence of Al was again confirmed in the residues. This dismisses the redeposition and micromasking theory. Another possible explanation for

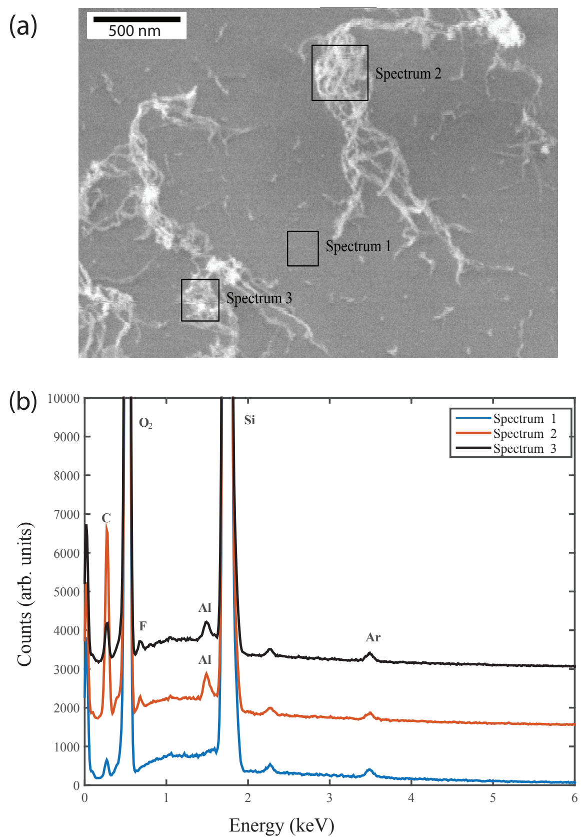

Figure 3: (a) A close up SEM image of the "fur-like" residues obtained after the PI etch in sample R with back overlaid rectangles representing the region of interest used for EDX scan of the corresponding spectrum, and (b) EDX analysis and comparison of residue free and covered areas depicting the presence of Al peaks for spectrum with residues. The spectra have been offset for clarity.

the residues is that the metal hard etch mask is not completely etched during the wet etching. Increasing the temperature or duration of the wet etch (200% over-etch) of metal mask did not change the results. To remove these residues the substrate was immersed in a PES etch shortly after the PI was etched by RIE. Upon inspection it was observed that the residues had been removed. This is one of the methods to eradicate the residues. However, this approach is not suitable for test device like structures as mentioned previously with free standing exposed metallic structures. Any kind of wet etchant leads to loss of the free standing structures due to the viscous forces acting upon them. This method is therefore only suitable for samples with no free standing structures and no exposed and active metal areas.

The etched mask area of the PI after the removal of the residues with PES etchant was studied under the SEM and a sparkly formation was observed on the PI (Fig. 4-a). Materials like polyimide are electrically non-conducting and normally show charging in a SEM. To prevent charging usually, a conducting surface coat must be applied to provide a path for the incident electrons to flow to ground. Unlike a normal polyimide, the polyimide at the locations of the metal mask did not show any charging, suggesting signs of certain metal inclusions on the top surface. The sparkles and the substrate underneath (PI) it was investigated with an EDX analysis, and traces of Al were again measured in the spectra (Fig. 4-b).

A second hypothesis based on these results was drawn; the formation of micro/nano pockets on the surface of the PI where the Al gets deposited while sputtering. These pockets/roughness is created during the Ar^+ ion sputter etch to improve adhesion of the metal to PI [13].

Upon wet etching of the patterned metal, the metal in these pockets is not etched due to the low surface energy of PI, like most of the polymers [19][20][21]. Increasing the duration of the wet etch in such a situation had no effect. This polyimide when etched in an oxygen plasma, gets etched except the pockets of Al which may act as micro-masks and eventually form “fur-like” residues in the end. The sputter etch is suspected to play a role in the formation of

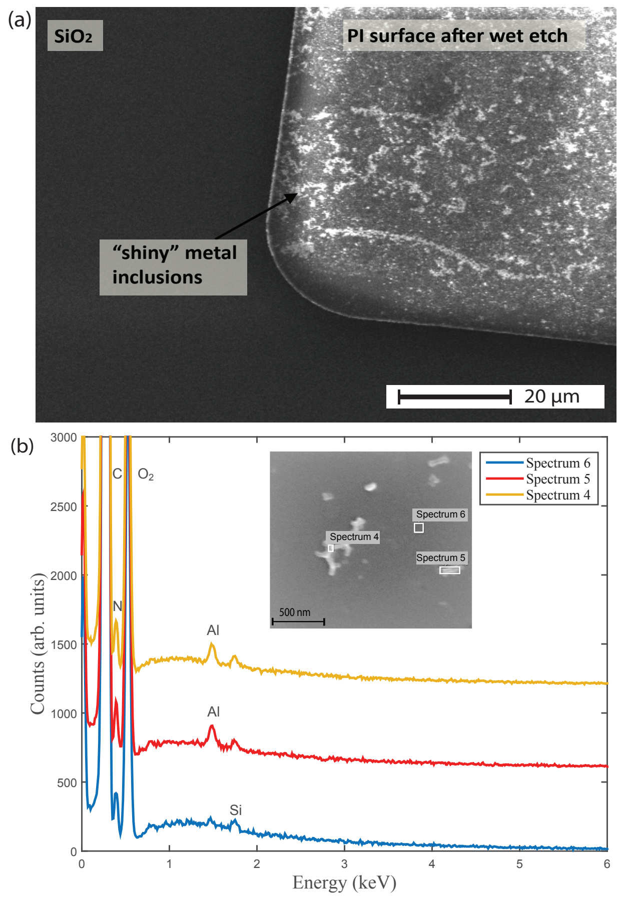

Figure 4: (a) Polyimide surface under the SEM after removal of the metal mask depicting sparkly inclusions in sample B. (b) EDX analysis of the shiny (spectrum 4 and 5) and non shiny (spectrum 6) areas on the PI represented in the inset image, depicting the presence of Al in small amounts. It is to be noted that the signal for EDX of these inclusions is very weak as the Al is possibly embedded within the PI. The spectra have been offset for clarity.

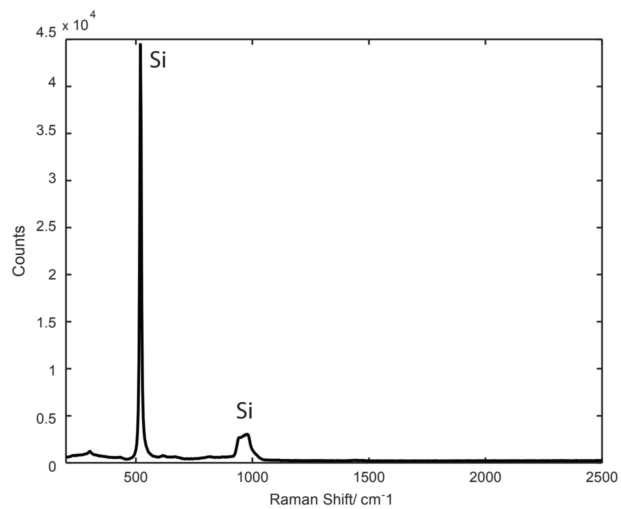

Figure 5: Raman bands for Al_4C_3 are reported for 532 nm excitation at 252.85 cm^{-1}, 288.65 cm^{-1}, 340.86 cm^{-1}, 493.00 cm^{-1}, 718.22 cm^{-1} and 864.40 cm^{-1} wavelengths [22][23]. However, Raman spectra of the residues show only the Si peaks from the substrate for sample A.

such pockets. To determine the role of sputter etch, sample B in experiment 2 is coated with 200 nm Al deposited by sputtering without sputter etch. This sample is blanket etched in wet etchant, followed by a blanket etch of the PI. Inspection of the samples in the SEM showed no residues. This confirms the second hypothesis, that the Ar^+ ion sputter etch does play a role in the residue formation.

In order to determine the cause of the formation of these residues, two more explanations were investigated. In the first theory, it is assumed that the surface of PI forms activated carbon after a short sputter etch in an Ar^+ plasma. This active carbon can react with the sputtered Al of the mask and form aluminum carbide (Al_4C_3). It is hypothesized that this compound does not get etched in the O_2 plasma etching of the PI and forms Al_4C_3 containing residues.

To detect the presence of Al_4C_3, Raman spectra of the residues in the reference sample were studied. According to literature, Raman bands for Al_4C_3 are reported for 532 nm excitation at 252.85 cm^{-1}, 288.65 cm^{-1}, 340.86 cm^{-1},

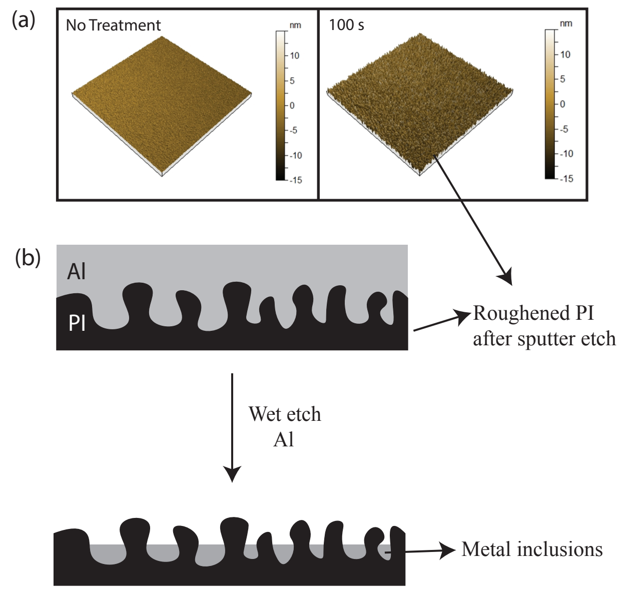

Figure 6: (a)Atomic force measurement (AFM) images of polyimide surface before and after 100 s Ar^+ sputtering with the root mean square (RMS) deviation of the surface being 0.5 nm and 4.7 nm, respectively. (b)Schematic of a rough PI surface with aluminum layer on top and the metal inclusions inside the roughened pockets of PI that remain after a wet etch due to partial wettability with the PES wet etchant.

493.00 cm^{-1}, 718.22 cm^{-1} and 864.40 cm^{-1} wavelengths [22][23]. Using similar excitation, and varying the laser intensities from 100 mW up to 500 mW (0.85 N/A), spectra for the residues and the substrate (SiO_2) underneath were obtained (Fig.5). No bands related to Al_4C_3 or carbon were detected, only bands related to silicon from the substrate are visible in the spectra, refuting this theory.

In another theory, the surface roughness of the PI created after Ar^+ ion sputtering is speculated to encompass Al inclusions within the roughened PI grooves. The roughness is measured with an atomic force microscope (AFM) where the root mean square (RMS) deviation of the surface is measured to be 0.5 nm and 4.7 nm before and after 100 s sputter etch, respectively (Fig. 6-a). The reference plane for the calculation of this parameter is the mean plane of the measured surface. The metal inclusions in the grooves do not get etched in the Al etch as the wettability of PI is low, making wetting of the grooves difficult during the PES etch (Fig. 6-b). In order to support this theory, the metal mask is dry etched in a Cl_2 chemistry plasma (5 mTorr) instead of the wet etchant. An over etch of 15 s is used to ensure the removal of the entrapped Al in the roughened PI. This does not effect the underlying PI layer. In the next step the PI is etched in exactly the same way as in the previous experiments and the surface is inspected in the SEM. SEM analysis showed no presence of residues on the surface, confirming this theory. This final result was tested on the sample C reported in experiment 3 (Table 1), where all the fabrication steps remained the same as the reference sample except the patterning of Al mask on top of PI. After the PI etch, the results show "fur free" free standing interconnect structures as shown in Fig. 7.

The role of metal mask deposition method was also studied in experiment 4. In this experiment sample E was prepared by depositing metal mask via evaporation instead of sputtering (sample D). After the PI etch, no residues were observed in the SEM. However, the sample surface has no sputter etch prior to the mask deposition as this cannot be incorporated in the metal evaporation tool. Although this is a solution, the metal- PI adhesion issue cannot be addressed using this deposition technique. Hence, this was not considered as a desirable solution.

4. Conclusions

In this paper, residues formed after dry etching of polyimide using Al hard etch mask have been investigated. Generally, according to literature [10], a short sputter etch is used on the surface of PI before depositing Al as a hard etch mask for a better adhesion. The metal mask deposited after the sputter etch forms metal "inclusions" on the top roughened PI layer. These inclusions do not get etched in the wet etch of the metal mask due to the low surface energy of PI [15]. This leads to the formation of metal containing residues after

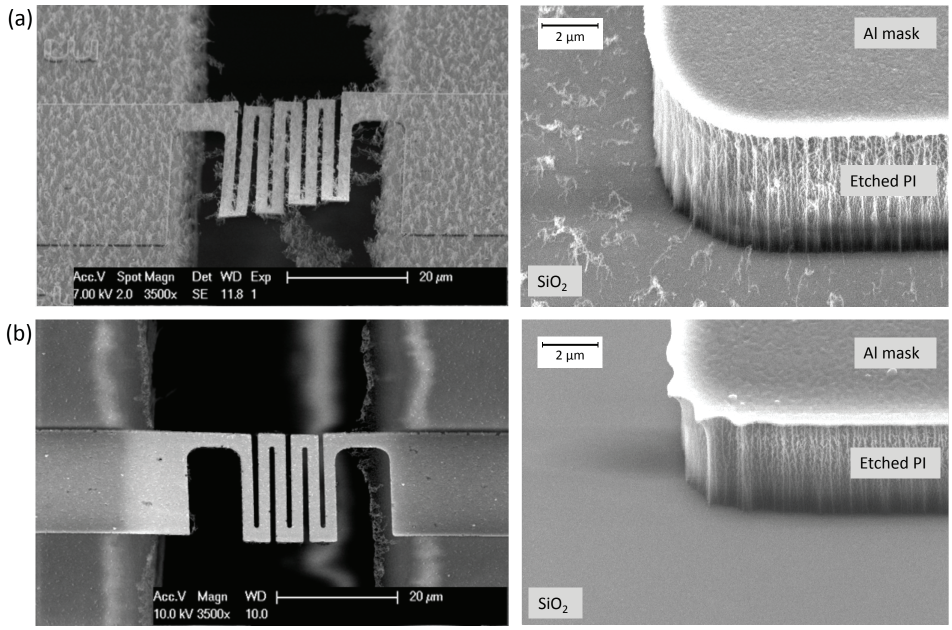

Figure 7: SEM image comparison of (a) sample R with residue covered free standing as well as not free standing structures after PI etch, with (b) free standing as well as not free standing structures of sample C after PI etch using dry etching of PI.

the PI etch. The residues can be removed using wet etchant like PES, but this approach becomes unfeasible if there are exposed active metal structures. A one-step dry etch of the metal mask solution has been investigated in this paper using a slight overetch. This slight overetch is enough to erode the top layer of the PI and remove the metal inclusions, thus enabling a "fur-free" PI etch.

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References

[1] W. Li, J.-L. Zhang, H.-M. Li, T. Ding, L. Wang, Preparation of ultrafiltration membranes with aromatic polyimide, in: Fifth International Membrane Science & Technology Conference, Sydney, Australia, 2003, pp. 1–6.

[2] M. Bessonov, Polyimides–thermally stable polymers, Consultants Bureau, 1987.

[3] M. Ghosh, K. Mittal, Polyimides: fundamentals and applications,(1996), Marcel Dekker: New York, NY).(b) M. Tomikawa, S. Yoshida, N. Okamoto, Polym. J 41 (2009) 604.

[4] L. Rothman, Properties of thin polyimide films, Journal of the Electrochemical Society 127 (10) (1980) 2216–2220.

[5] A. B. Frazier, Recent applications of polyimide to micromachining technology, IEEE Transactions on Industrial Electronics 42 (5) (1995) 442–448.

[6] E. Sacher, Dielectric properties of polyimide film. ii. dc properties, IEEE Transactions on Electrical Insulation (2) (1979) 85–93.

[7] J. C. Yeo, C. T. Lim, et al., Emerging flexible and wearable physical sensing platforms for healthcare and biomedical applications, Microsystems & Nanoengineering 2 (2016) 16043.

[8] B. Mimoun, V. Henneken, A. van der Horst, R. Dekker, Flex-to-rigid (f2r): A generic platform for the fabrication and assembly of flexible sensors for minimally invasive instruments, IEEE Sensors Journal 13 (10) (2013) 3873–3882.

[9] S. Shafqat, J. Hoefnagels, A. Savov, S. Joshi, R. Dekker, M. Geers, Ultrastretchable interconnects for high-density stretchable electronics, Micromachines 8 (9) (2017) 277.

[10] D. L. Pappas, J. J. Cuomo, K. G. Sachdev, Studies of adhesion of metal films to polyimide, Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films 9 (5) (1991) 2704–2708.

[11] B. Mimoun, H. T. Pham, V. Henneken, R. Dekker, Residue-free plasma etching of polyimide coatings for small pitch vias with improved step coverage, Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics: Materials, Processing, Measurement, and Phenomena 31 (2) (2013) 021201.

[12] Y. Tsang, C. Miller, T. Lii, Investigation of polyimide residue due to reactive ion etching in o 2, Journal of the Electrochemical Society 143 (4) (1996) 1464–1469.

[13] S. Joshi, A. van Loon, A. Savov, R. Dekker, Adhesion improvement of polyimide/pdms interface by polyimide surface modification, MRS Advances 1 (01) (2016) 33–38.

[14] U. Buder, A. Berns, R. Petz, W. Nitsche, E. Obermeier, Aeromems wall hot-wire anemometer on polyimide substrate featuring top side or bottom side bondpads, IEEE Sensors Journal 7 (8) (2007) 1095–1101.

[15] E. Liston, L. Martinu, M. Wertheimer, Plasma surface modification of polymers for improved adhesion: a critical review, Journal of adhesion science and technology 7 (10) (1993) 1091–1127.

[16] S. J. B. Reed, S. J. B. Reed, Electron microprobe analysis, Vol. 2, Cambridge University Press Cambridge, 1975.

[17] G. Schwartz, L. Rothman, T. Schopen, Competitive mechanisms in reactive ion etching in a cf 4 plasma, Journal of The Electrochemical Society 126 (3) (1979) 464–469.

[18] T. H. Fedynyshyn, G. W. Grynkewich, T. B. Hook, M.-D. Liu, T.-P. Ma, The effect of aluminum vs. photoresist masking on the etching rates of sili-

con and silicon dioxide in cf 4/o 2 plasmas, Journal of The Electrochemical Society 134 (1) (1987) 206–209.

[19] S. Süzer, A. Argun, O. Vatansever, O. Aral, Xps and water contact angle measurements on aged and corona-treated pp, Journal of applied polymer science 74 (7) (1999) 1846–1850.

[20] A. Bellel, S. Sahli, P. Raynaud, Y. Segui, Z. Ziari, D. Eschaich, G. Dennler, Improvement of the polyimide surface wettability using siox films deposited in a decr reactor from hmdso/o2 mixtures, Plasma Processes and Polymers 2 (7) (2005) 586–594.

[21] H. Hiraoka, S. Lazare, Surface modifications of kapton and cured polyimide films by arf excimer laser: applications to imagewise wetting and metallization, Applied Surface Science 46 (1-4) (1990) 264–271.

[22] B. Chen, L. Jia, S. Li, H. Imai, M. Takahashi, K. Kondoh, In situ synthesized al4c3 nanorods with excellent strengthening effect in aluminum matrix composites, Advanced Engineering Materials 16 (8) (2014) 972–975.

[23] J. L. Kennedy, T. D. Drysdale, D. H. Gregory, Rapid, energy-efficient synthesis of the layered carbide, al 4 c 3, Green Chemistry 17 (1) (2015) 285–290.