Patterning of Soft Polydimethylsiloxane Elastomers Using Plasma Etching

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ABSTRACT: Soft polydimethylsiloxane (PDMS) elastomers spin coated on silicon wafers are patterned using plasma etching. The elastomers are formed by mixing vinyl-terminated PDMS (prepolymer) and crosslinker containing hydride sites. The elastomers are made softer by adding either excess prepolymer, excess crosslinker, or swelling agent. The different elastomers were etched using reactive ion etching, an old and well established processing technology in the microelectronic and MEMS industry. The etch rate and profiles of the etched surfaces were dependent on the process pressure. The dependence was different for the different elastomers. It was found that at low pressure, the etch rate was dependent on the shear modulus. At high pressures, the etch rate was dependent on the chemical composition of the elastomer. The results for the swollen elastomers were different from those for the nonswollen elastomers. The etch rate was lower and the profiles of the etched cavities were different. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 888–895, 2011

Key words: polydimethylsiloxane (PDMS) elastomers; reactive ion etching (RIE); storage modulus; etch rate; etched profiles

INTRODUCTION

Plasma etching of polydimethylsiloxane (PDMS) elastomers has been investigated in several earlier works.[1–7] In these works, commercially available elastomer kits were used. Garra et al.,[1] Vlachopoulou et al.,[5] and Balakrisnan et al.[7] used Sylgard-184 from Dow Corning. Szmigiel et al.[2–4] used MED-6215 from Ultrasil Technology. The elastomer kits used are not pure PDMS. Sylgard-184 contains 10–30% silica particles according to the material data safety sheet. The material data safety sheet for MED-6215 do not say anything about the content. However, from XPS spectra around the Si 2p area,[3] it is seen that the untreated sample contains some SiO_2. Both Sylgard-184 and MED-6215 must, therefore, be considered as composite materials. Earlier work[8] has showed that plasma etching of Sylgard-184 gave different results from PDMS elastomers without any filler. There are different technologies available for plasma etching.[9] All technologies have in common that the substrates are exposed to vacuum and an oscillating electric field excites the process gas to ions, radicals, and electrons. The earlier work about plasma etch of PDMS include both reactive ion etching (RIE),[1–4,7,8] inductively coupled plasma (ICP),[4,5] and microwave barrel etcher.[6] The oldest and simplest technology is barrel etcher. When etching in a barrel etcher, the sidewalls will be isotropic. There is no directional control of the etch rate. When using RIE, it is possible to achieve vertical sidewalls. RIE has the advantage that the technology is well established in microelectronic and MEMS industry and the equipment is readily available. ICP was introduced in the mid 90s and has many advantages over RIE. ICP allows more control over the plasma parameters and a higher plasma density compared to RIE. However, the equipments for ICP are in general much more expensive than RIE.

The commercially available elastomer kits contain additives to get the desired properties. The addition of silica particles in Sylgard-184 results in a higher shear modulus. The surface of the silica particles are covered by vinyl groups. These groups will react with the hydride sites on the crosslinker molecules. Hence, the particles will be part of the elastomer network. When no fillers are present in PDMS elastomers, the main parameters affecting the shear modulus are the molar mass of the components and the crosslinker density. The shear modulus of elastomers made from high molar mass components has contribution from molecular entanglements. This contribution can be disregarded when the molar mass is below a critical value.

Devices based on PDMS are produced in a wide range of applications. Microfluidic devices are in

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many cases made from PDMS,[10] either PDMS only or in combination with other materials. A wide range of methods for making such devices are available, also some that include plasma etching.[1] Optical devices are also a field in which PDMS has been used. PDMS is a well suited material for waveguides.[11] Other examples are metalized light-modulators,[12,13] variable optical attenuator,[14] and display modulators.[15] Such devices require a soft elastomer as the elastomer is deformed by electrical fields. A hard elastomer would require very high voltages to be sufficiently deformed. For waveguide applications, the PDMS must be robust to avoid breakage in the optical path. Chang-Yen et al.[11] used Sylgard-184 as building material. For high volume production of such small devices, cost effective processing technologies are desired. In microelectronic and MEMS industry devices are made on a wafer scale. Each wafer is processed and in the end cut into single devices. Such an approach is well suited for high volumes. RIE is one of several process technologies widely used for high volume wafer production.

In this work, we have carried out etching of soft PDMS elastomers using RIE. RIE has been performed on elastomers with different shear modulus. The elastomers are formed by mixing vinyl-terminated linear PDMS and crosslinker containing hydride sites. The shear moduli of the elastomers are changed by varying the ratio of prepolymer to crosslinker and by adding a swelling agent. Mechanical properties of the elastomers were measured to achieve a better understanding of the variations in etching results for the different elastomers. Plasma etching was found to be dependent on both the process conditions and the elastomer composition.

MATERIALS AND METHODS

Sample preparation and spin coating

The chemicals linear vinyl-terminated polydimethylsiloxane (DMS-V22 from Gelest Inc.), methylhydrosiloxane-dimethylsiloxane copolymer (HMS-151 from Gelest Inc.), trimethyl terminated PDMS (low molar mass silicone oil (10 cSt) from Sigma Aldrich), and catalyst (Platinum/Vinylsiloxane catalytic complex, PC072 from United Chemical Technologies) were used as delivered. Elastomers were made by mixing V-22 and HMS-151 in different ratios and adding catalyst. Swollen elastomers (gels) were made by adding the silicone oil before cure. By varying the ratio of the different components, different shear moduli were obtained. Table I shows the compositions and the shear modulus of the different samples. After mixing the components, the liquids were shaken for 5 min and left another 5 min to let trapped air bubbles diffuse out.

Three milliliters of the freshly mixed liquid was spin coated onto polished 100 mm silicon wafers. The samples Gel50 was spin coated 30 s at 350 rpm. The sample Gel100 was spin coated 30 s at 400 rpm. The rest of the samples were spin coated 40 s at 550 rpm. The spin-coater was programmed to use 10 s for acceleration up to the desired rotational speed and 15 s to slow down to zero. The samples were cured at least 24 h at 25°C after spin coating.

Masking and etching

A 100 mm glass wafer was cut into glass slides 10 mm wide. The glass slides were coated by carbon-fluor polymer on one side in CHF_3 plasma at 100 mTorr and 100W RF power for 5 min. The coated side was in contact with the PDMS film during the etching process. The purpose of the carbon-fluor polymer was to reduce adhesion between the film and the mask. In an earlier work,[8] there were problems with high adhesion between the mask and the PDMS film. The adhesion led to damages in the film when the mask was removed after etching. The glass slides were placed on the substrate with a spacing of 3 mm.

The etching was done in a PlasmaTherm SLR720 Reactive Ion Etcher (RIE). The RIE tool was configured to be able to process four wafers at a time. One wafer slot was used for the substrate. The other three were covered by glass dummy wafers. To ensure the same chamber conditions for all wafers, the process chamber was cleaned with deionized water after each processing. After cleaning, the chamber was left to pump overnight and then further cleaned by running oxygen plasma for 15 min. Each wafer was etched for 30 min using 80% SF_6 and 20% O_2 as process gas. The RF power was 300W. The temperature was set to 25°C. The pressure was varied from 50 to 200 mTorr. After etching, the glass slides were manually removed from the substrate.

Characterization

The surface of the PDMS film was characterized using a Wyko NT-9100 white light interferometer. The equipment measured both the etched depth and the profile of the etched surface. The etch rate was calculated by dividing the etched depth by the process time. The etched depth was measured in the middle of the etched area and at the edge of the etched area. It was measured in five locations on each wafer. The reported etch rate is the average of the measured results for each wafer.

TABLE I
Composition and Shear Modulus of the Elastomers

Sample Name	V-22 (wt %)	HMS-151 (wt %)	Silicone oil (wt %)	G' (kPa)
EV50	93	7	–	50
EV100	92	8	–	100
BE200	90	10	–	200
EH100	69.5	30.5	–	100
EH50	61.5	38.5	–	50
GEL100	54	6	40	100
GEL50	35.3	3.7	63	50

Dynamical mechanical measurements

The storage (G') and loss (G'') moduli were measured using oscillating parallel plates (50 mm diameter, 1 mm thickness) in a Physica MCR 300 rheometer. The elastomers were cured in the rheometer using a fixed curing profile (25–125°C at 10 K/min, 125°C for 20 min, and cooling down to 25°C at −1 K/min). The measurements were carried out at 25°C, with constant amplitude (1–2%) and angular frequencies ranging from 0.1 to 100 rad/s.

RESULTS AND DISCUSSION

Chemical composition and mechanical properties

Figure 1 shows the chemical reaction during the crosslinking. The vinyl group reacts with a hydride site on the crosslinker molecule. All elastomers in this work are made from the same starting materials. Some of the factors determining the modulus of the elastomers are the same for all elastomers such as molar masses of the precursors, molar mass distribution and the functionality of the crosslinker. The molar mass of the precursors are 9400 g/mole for V-22 and 1900–2000 g/mole for HMS-151 according to the product catalog. The moduli shown in Table I are all dependent on the composition of the elastomers. The modulus has no contribution from chain entanglement as the molar mass is well below the critical entanglement molar mass which is reported to be 10.1–12.5 × 10^3 g/mole.[16]

Figure 2 shows the shear modulus for the nonswollen elastomers as function of the composition. The highest modulus is achieved when 10% crosslinker is used. This corresponds to a stoichiometric ratio close to 1. This elastomer composition has the lowest number of free polymer chains and the highest crosslink density. A lower stoichiometric ratio (excess vinyl groups) leads to a lower modulus; small excess of vinyl groups leads to high decrease in the modulus. This is due to the fact that each excess vinyl group corresponds to increased number of free polymer chain ends. As the excess of vinyl groups increases (reducing stoichiometric ratio), some of the vinyl-terminated chains will not be bonded at any end and will remain free as a swelling agent. Going the other way, the elastomers have a different character. Small deficit of vinyl groups results in a little reduction in the crosslink density. All the vinyl groups will be used, and there will be some free hydride sites. As can be seen in Table I and Figure 2, it requires a high excess of crosslinker

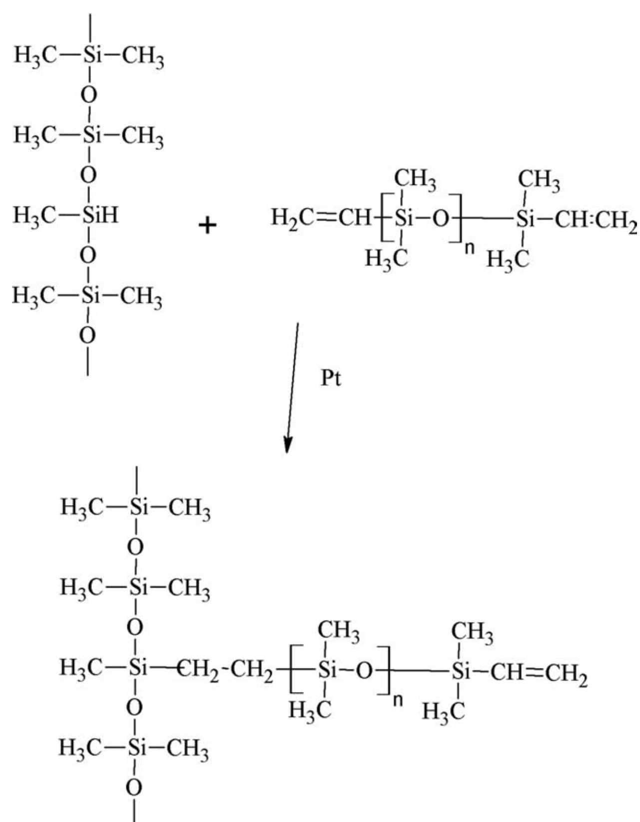

Figure 1 The chemical reaction during the crosslinking of the PDMS elastomers. The linear vinyl-terminated is attached to the hydride site in the crosslinker molecule. The reaction is catalyzed by platinum.

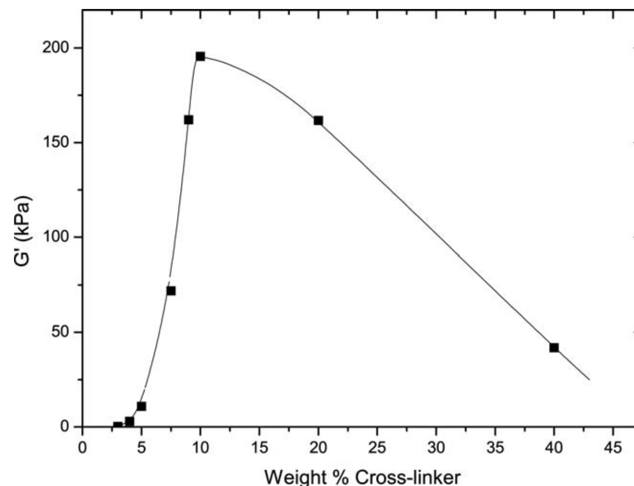

Figure 2 Shear modulus of the PDMS elastomers as function of the amount of crosslinker.

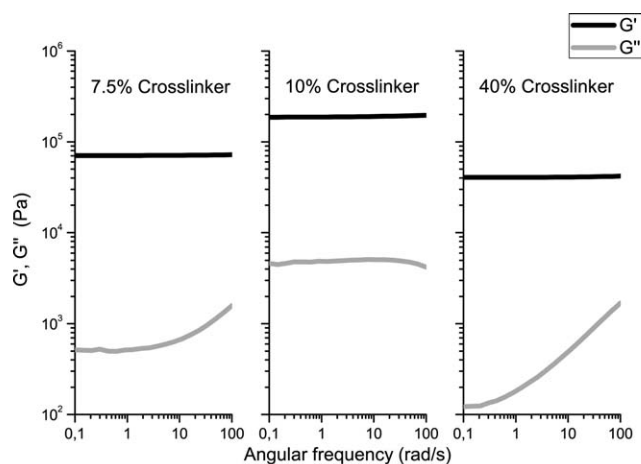

Figure 3 Mechanical spectra of three non-swollen elastomers based on V-22 and HMS-151.

to reduce the modulus to 50 kPa. At high excess of crosslinker, many free chain ends will be present in the elastomers.

Figure 3 shows mechanical spectra of three non-swollen elastomers. The left spectrum has a composition between the sample EV50 and EV100. The middle spectrum has the same composition as the sample BE200. The right spectrum has the same composition as the sample EH50. All three spectra show a flat curve for the storage modulus G' which indicates that the samples have reached their gel-points. The middle spectrum shows a flat curve for loss modulus G'' which indicates that there is no free chain ends present in the elastomer. The other two spectra show G'' increasing with the frequency. This indicates that there are free chain ends present in the elastomer. The variation in G'' is significantly larger for the right spectrum than the left. This indicates a higher number of free chain ends.

Another way of softening PDMS elastomers is to add swelling agent. In this case, the swelling agent is low molar mass silicone oil (viscosity 10cSt). The swelling agent is not chemically bound to the elastomer network. Hence, the mobility of the swelling agent molecules is high due to the low molar mass of the swelling agent. No measurement of the molar mass of the swelling agent has been performed. However, based on information from similar silicone oils, it is expected to be around 1000 g/mole. Figure 4 shows the shear modulus as function of the amount of swelling agent. There is a linear decrease with increasing amount of silicone oil. The point with 0% swelling agent has the same composition as the sample BE200.

Etching results

Figure 5 shows cross sections of the etched samples. The samples BE200, EV50, and EH50 all show a similar trend on increasing the pressure. At 50 mTorr, the etched surface has a flat or negative profile. At 100 mTorr, the etched surfaces have flat profiles. At 150 mTorr and 200 mTorr, all etched surfaces have positive profiles. This trend can be explained by different plasma conditions at different pressures.[8] The reduction in etch rate from 150 to 200 mTorr may be explained by a limitation in the plasma equipment. The amount of reflected RF power increases as the pressure is increased up to 200 mTorr. Hence, optimal plasma conditions are not present at this pressure. The samples EV100, EH100, and Gel100 had a profile following the same trends as the softer samples. The profiles of those samples were approximately in between the softer (EV50, EH50, and Gel50) and the harder (BE200) sample.

Comparing the sample BE200 and the other two nonswollen elastomers, it is seen that softening the elastomer by adjusting the stoichiometric ratio strongly affects the etching results. Comparing the sample BE200 and the sample EV50, it is seen first that the effect of adding excess vinyl groups leads to more variation in the profile of the etched surface. At 50 mTorr, the profile of the sample BE200 is flat, whereas the sample EV50 has a clearly negative profile. Second, it is seen that the addition of excess vinyl groups leads to a lower etch rate. The sample EV50 has a higher concentration of unreacted vinyl groups than the sample BE200. The incoming fluorine radicals from the plasma will easily react with the vinyl group and form a fluorinated chain end. The presence of excess vinyl groups lead to higher consumption of fluorine radicals without forming a gaseous reaction product compared to the balanced elastomer. Hence, the etch rate is lower.

The profiles of the sample EH50 and BE200 are more or less the same; flat at low pressure and positive at high pressure. The main difference between

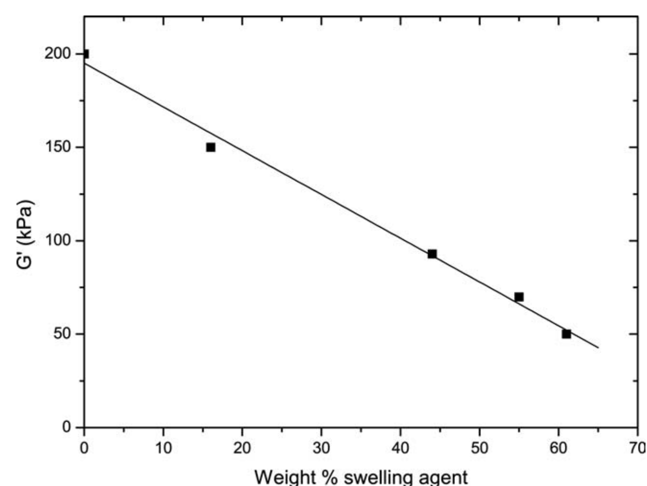

Figure 4 Shear modulus as function of the amount of swelling agent in the sample.

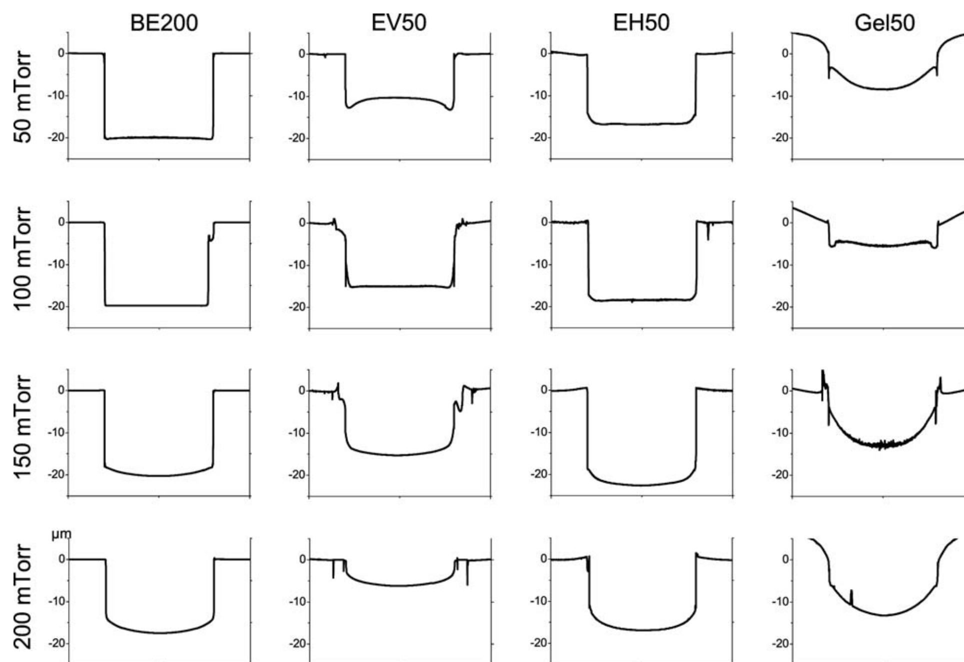

Figure 5 Cross section profile of the etched area. The profiles are measured using a white light interferometer.

the two elastomers is the shape of the corner of the etched areas. The sample BE200 has sharp corners, whereas the sample EH50 has rounded corners. This is explained by the different stoichiometric ratio. The sample EH50 has a high excess of crosslinker molecules. The excess is so high that some of the crosslinker molecules will not be bound to the elastomer network. Hence, the sample is a little swollen. The swollen molecules are mobile and will diffuse during the etching process. This may change the profile at the corners.

The swollen elastomer Gel50 has different profiles of the etched surfaces. For all pressures, the etched surface has a positive profile. This can be explained by the high mobility of the swelling agent. The swelling agent is not bound chemically to the elastomer network and the molar mass is low. Hence, the mobility is high. During the etching process, the samples are exposed to vacuum. The plasma radiates heat during the etching. This heat is absorbed by the substrate. Hence, the evaporation is enhanced. The evaporation of volatile compound from the swollen elastomer leads to diffusion of the swelling agent during etching. The diffusion of swelling agent molecules during the process leads to a constant supply of swelling agent molecules to the areas exposed to the plasma. The result is a positive profile for all pressures for highly swollen samples. There will also be a diffusion of swelling agent (relaxation) after the etching is complete, working toward a homogenization of the gel material. The extent of this diffusion has not been the focus of this study.

Figure 6 shows the etch rate as function of the pressure for all samples. The etch rate is measured in the middle of the cavity and at the edge of the cavity. All nonswollen elastomers have a reduction in the etch rate when the pressure is increased from 150 to 200 mTorr. This is as already mentioned an equipment limitation due to increased reflected RF power. At low pressures, the ion flux and the ion energy are high. As the pressure is increased, both the ion flux and ion energy decrease. The radical flux increases with increasing pressure.[8]

The sample BE200 shows little change in the etch rate in the middle of the cavity up to 150 mTorr. The etch rate at the edge of the cavity gradually decreases as the pressure is increased. The samples EV100 and EV50 both show increasing etch rate when the pressure is increased from 50 to 100 mTorr; from 100 to 150 mTorr the etch rate decreases. The etch rates in the middle of the etched cavity and at the edge of the cavity show a similar trend. The curves for the two cavity positions cross at 100 mTorr for both samples. The change in etch rate is higher for the sample EV50 than for the sample EV100. The samples EH100 and EH50 show increasing etch rate from 50 to 150 mTorr. The etch rate in the middle of the cavity increases more than the etch rate at the edge. The curves cross at 100 mTorr. The etch rate of all nonswollen elastomer increases when increasing the pressure from 50 to 100 mTorr. This is explained by increasing radical flux and still sufficient ion flux and ion energy. From 100 to 150 mTorr, the etch rate behaves differently on the different elastomers. The etch rate

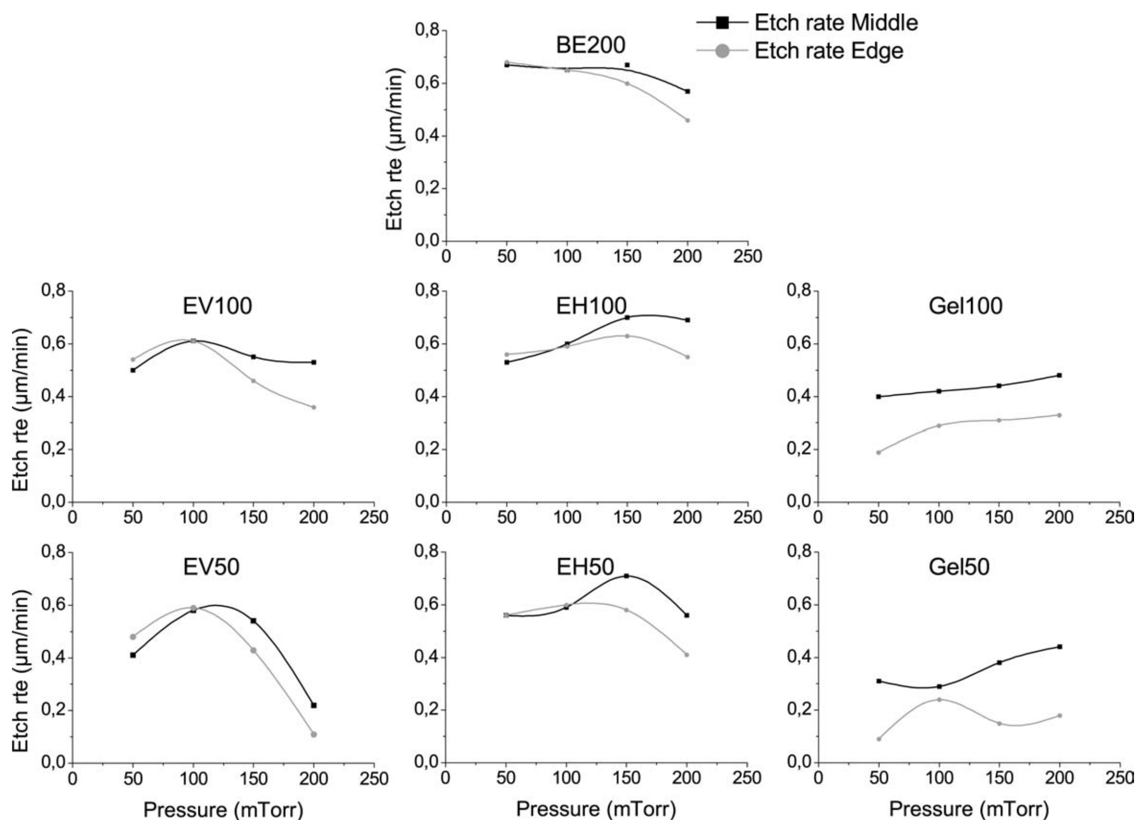

Figure 6 Measured etch rate plotted as function of the process pressure at different cavity positions.

decreases for the samples with excess vinyl groups. The etch rate is constant for the balanced elastomer. The etch rate increases for the sample with excess hydride sites.

The samples Gel100 and Gel50 show different results from that of the nonswollen elastomers. Both samples have a higher etch rate in the middle of the cavity than at the edge for all pressures. The sample Gel100 shows increasing etch rate with increasing pressure up to 200 mTorr at both cavity locations. The sample Gel50 shows more variation in the etch rate than the sample Gel100. However, it is not clear if this high variation is caused by the etching process itself or the manual placement of the mask. The sample Gel50 contains more swelling agent than the sample Gel100. Hence, a higher fraction of the volume has a high mobility. Exerting a force on such a film will induce movement of the mobile fraction.

The etch rate for the different samples at the different pressures are compared in Figure 7. At 50 mTorr, the sample BE200 is etched faster than the other samples. The etch rate of the samples with excess vinyl groups is lower than for the samples with excess hydride sites. The etch rate is lower for the sample EV50 than the sample EV100. In contrast, the etch rate of the sample EH50 is higher than the sample EH100. The sample BE200 has the highest modulus and hence least flexibility in the elastomer network. At 50 mTorr, the ion flux and ion energy are high. The ion energy is not absorbed through movement of the polymer chains as in the softer samples with more free chain ends. Instead the ions in a higher degree contribute to break up the elastomer network. Hence, the etch rate for the harder sample is higher than for the softer samples. The samples containing excess hydride sites have a higher etch rate than the samples containing excess vinyl groups. The higher excess of hydride sites the higher etch rate. In contrast, the higher excess of vinyl the lower etch rate. Those observations are explained by the chemistry of the free chain ends. Fluorine radicals from the plasma will react with both the vinyl group and the hydride sites. The vinyl group will absorb two fluorine radicals and result in a fluorinated chain end. This leads to an extra consumption of fluorine radicals and, therefore, lower etch rate. A hydride site will react in a different way. The fluorine radical can extract the hydrogen and form hydrogen fluoride. The result is a radical site on the polymer chain. This radical site can either be a breaking point for the chain or react with other fluorine radicals. Increased excess of crosslinker leads to a higher number of free hydride sites. Hence, the etch rate increases with increasing excess of crosslinker.

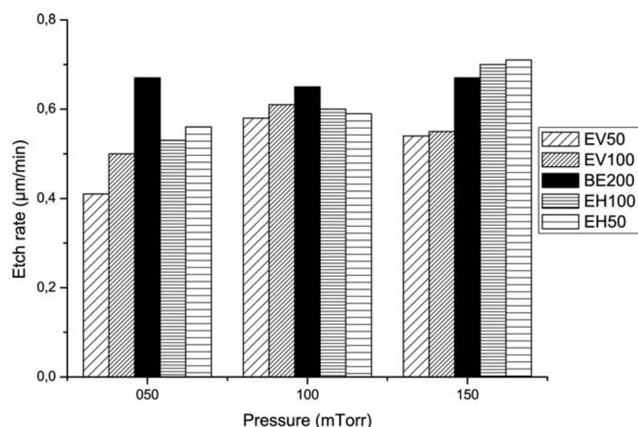

Figure 7 Etch rate for the different nonswollen elastomers compared at different pressures.

At 100 mTorr, the etch rate of the different elastomers show less variation. The etch rate for the sample BE200 is a little higher, whereas the difference in etch rate for the other samples are within the expected statistical variation. At 100 mTorr, the contribution from ions is lower compared to 50 mTorr. Both the ion energy and the ion flux are lower; the radical flux has increased. However, the ions still have a significant contribution to the overall etching. Hence, the softer samples all show increased etch rate as the pressure is increased from 50 to 100 mTorr.

At 150 mTorr, the etch rates show a completely different behavior. The etch rate for the samples containing excess vinyl groups are significantly lower than for the rest of the samples. At this pressure, the contribution from ions is low. The overall etching process is dominated by radicals. The ion energy and ion flux are reduced so the surfaces exposed to the plasma are not continuously excited by incoming ions. The result is a lower reaction rate between the radicals and the exposed surface. The vinyl group consumes fluorine radicals and the resulting fluorinated chain ends have a stabilizing effect. Hence, the etch rate is lower for samples with excess vinyl groups. There is little difference on the etch rate for the samples BE200, EH100, and EH50. However, there is a trend that is clear. The higher excess of hydride sites the higher etch rate. The silicon atoms bound to a hydrogen atom and one methyl group are less stable to chemical reaction than silicon atoms bound to two methyl-groups. Hence, the etch rates of samples containing excess hydride sites are higher than that of the balanced elastomer.

Other factors affecting the etching results

The manual placement of the glass slides used as mask before etching, and the removal of them after etching can affect the measured results. It is not possible to avoid using any force when placing the glass slides. The very mobile swelling agent molecules will then be forced to move from the areas underneath the glass slide to the areas that will be etched in the plasma. The effect is visible in Figure 8. The areas that are exposed to the plasma are more than 2 µm higher than the areas protected by the glass slides before etching starts. Hence, the measured etch rate can be lower than the true etch rate. When the glass slides are removed the effect is the opposite. There is some adhesion between the glass surface and the PDMS surface. When removing the glass slides, the stretching of the protected areas can force the swelling agent to move to the areas underneath the mask. This explains the not very flat protected areas for the swollen samples in Figure 5. This diffusion may not be disregarded for affecting the measured profiles of the etched surfaces.

The other main problem during the work was strong adhesion between the glass slides and the PDMS surface. When removing the glass slides, flaws were introduced in the PDMS film. One example of such damages is shown in Figure 9. These damages are problems especially on the samples EV50 and EH50. These are the samples containing the highest amount of free polymer chain ends. The

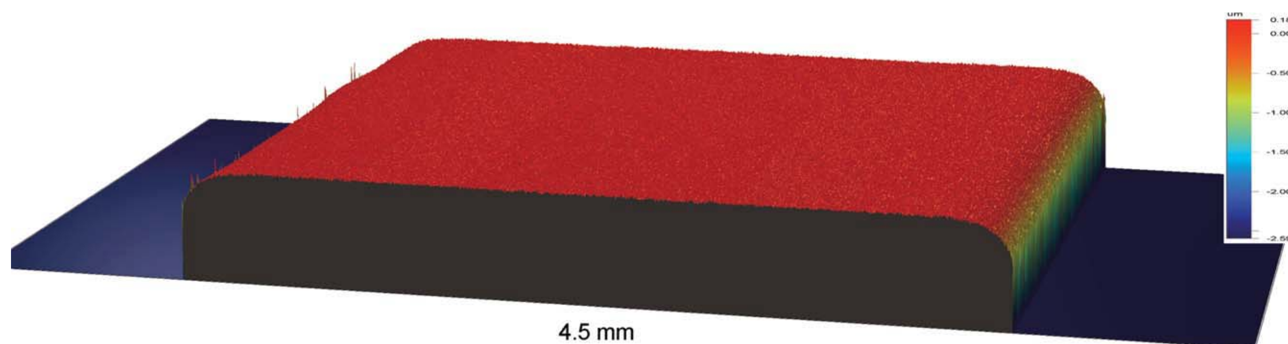

Figure 8 3D profile image of the sample Gel50 after placing the glass slides before etching. The blue areas on the side are the areas covered by the glass. The swelling agent is forced to the area between the glass slides when placing the mask. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

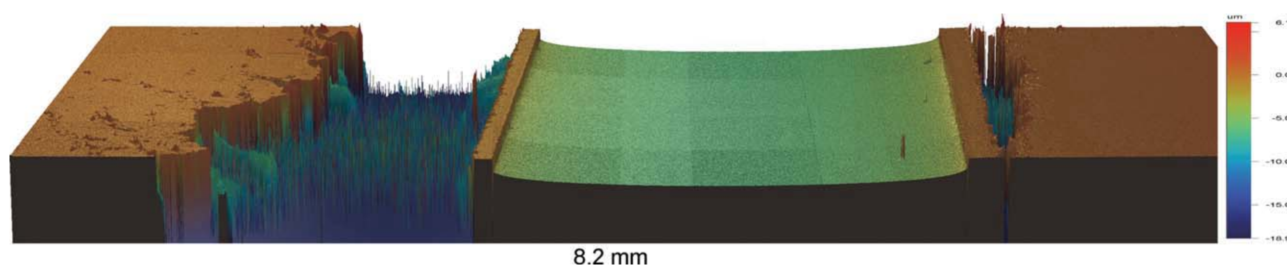

Figure 9 Damages in the PDMS film introduced when removing the glass slides after etching. The example is from the sample EV50. The green area is the etched cavity. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

sample BE200 and the two swollen elastomers Gel100 and Gel50 do not have this problem.

CONCLUSIONS

Soft PDMS elastomers spin coated on silicon wafers were made and plasma etched using RIE. The etch rate was dependent on the process pressure. The dependence was different for the different elastomers. The balanced elastomer had constant etch rate up on increasing pressure. The elastomers with excess vinyl groups had a maximum etch rate and the elastomers with excess hydride sites had increasing etch rate on increasing pressure. At low pressure, the etch rate is dependent on the shear modulus of the elastomers. Highest etch rate was measured for samples with the highest shear modulus. At high pressure, there was no dependence on the shear modulus. The chemical composition of the elastomers had an effect on the etch rate of the elastomers. Elastomers with excess vinyl groups had a lower etch rate than the balanced elastomers and the ones with excess hydride sites. Swollen elastomers had lower etch rate, but it is uncertain about the accuracy of the etch rate measurements because of the high mobility of the very low molar mass PDMS.

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