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COMPARATIVE ASSESSMENT OF DIFFERENT SACRIFICIAL MATERIALS FOR RELEASING SU-8 STRUCTURES

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Abstract. A range of materials are tested and compared as sacrificial layers for releasing SU-8 structures from the substrate following their manufacture. Four metals (chromium, chromium/gold, copper and aluminium) and three polymers (polymethylmethacrylate, polyimide and polystyrene) were investigated. Factors assessed included; quality of the SU-8 structures (by SEM examination before and after release), effect of changing etchant concentrations, monitoring of the undercutting process (through depth measurement with time), and the time taken for the successful release process. Overall, it was found that the metals were the best choice as sacrificial layers under SU-8 for structures up to 200 μm , whereas polymers were the better for larger structures (up to 600 μm in this work).

1. INTRODUCTION

In recent years SU-8 has become a very popular and attractive negative-working photoresist for high-aspect-ratio structures and thick resist layers [1-3]. It is commonly used in a wide range of applications of micro- and nano-fabrication due to its physical properties, thermal and chemical stability, biocompatibility, and its low fabrication cost [4,5]. However, releasing the cured SU-8 structures from the substrate, following fabrication, is a critical step as it can be very time-consuming and may affect the integrity of the microstructures. This is particularly noticeable for microstructures where the contact surface with the substrate is large. Earlier research [6,7] investigated the performance of individual sacrificial layers for releasing SU-8 structures.

Two different photoresists (Shipley S1813 and Hoechst AZ P4620) were used as sacrificial layers in conjunction with SU-8 photoresist, which was used as electroplating mould in MEMS manufacture [6].

The authors of [6] claimed that the use of photoresists as sacrificial layers offered several advantages, including the reduction of process steps and hence cost. However, photoresist-based sacrificial layers fail when used in conjunction with SU-8, as it has a tendency to attack photoresist sacrificial layers. Increasing the softbake temperatures for the sacrificial layers was able to improve this situation. The optimised temperatures were 175 °C and 200 °C respectively for S1813 and AZ P4620. In both cases the bake time was 3 hours. NANO Removal

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PG was used to remove the sacrificial layer and exposed SU-8 at a temperature of 50-80 °C. Any residual SU-8 was removed by O₂ plasma ashing.

In some more recent work [7], polystyrene was used as the sacrificial material underlying the SU-8. Toluene was used, initially to dissolve the polystyrene for spin-coating, and again after the SU-8 processing to etch the sacrificial polystyrene layer and release the structures. This approach was designed primarily for releasing large SU-8 structures with a substrate contact area of ~50 cm². Additionally, the toluene caused almost no damage or swelling effect to the released SU-8 structures. For smaller SU-8 structures (in the range 10-500 nm), other materials were also investigated and included two metals (aluminium and chromium); one negative-tone photoresist, HNR 120 from Arch Chemicals Inc.; two positive-tone photoresists, HIPR 6512 from Arch Chemicals Inc. and Shipley S1813 and OmniCoat from MicroChem Corporation (MCC). The latter product was recommended by MCC who also manufactured the SU-8.

The work reported here adds to the earlier investigations, by carrying out comparative tests between a range of materials. The materials used were: chromium (Cr), chromium/gold (Cr/Au), copper (Cu), aluminium (Al), polymethylmethacrylate (PMMA), polyimide and polystyrene. Samples using the above materials were prepared and the effect of their processing on the SU-8 microstructures was assessed by SEM examination before and after their release. In addition, the time taken to release the structures from the silicon wafer substrates was also compared.

2. EXPERIMENTAL

The experiments for releasing SU-8 structures were divided into two main categories; metals and polymers. In both cases they were coated onto 100 nm silicon wafer substrates. The metals used were deposited in three different thicknesses in the nm range. Specifically, the layers for Cu were 150, 350 and 600 nm; for Al 25, 65 and 100 nm; for Cr 100, 225 and 410 nm; and finally for Cr/Au were 150 nm/100 nm, 150 nm/250 nm and 150 nm/500 nm.

Three polymers were initially tried (PMMA, polystyrene and polyimide). However, it was observed that the polyimide resist reacted with the SU-8. The solution to the above problem could be, according to Song and Ajmera [6], to change the processing cycle and to apply high hard baking temperature (around 200 °C), however it was not used any further in the work reported here.

Deposition of the Cr, Al and Cu to different thickness on the silicon wafers was performed using a Nordiko plasma sputtering system. A single chromium/gold layer was also deposited using a CVC plasma sputtering system. Better adhesion between chromium/gold was observed for the CVC-coated substrates. Films deposited using the Nordiko were found to delaminate during the release process.

The polymers were deposited to the appropriate film thickness was achieved using a spin-coating process. Specifically, for the PMMA sacrificial layers, the different thicknesses were achieved thus:

- (i) for the 100 nm thickness layer, 3 ml of solution (4% ELV solvent) was spin-coated at a speed of 6000 rpm for 1 min. The wafer was then baked on a hot plate at 90 °C for 20 min.
- (ii) for the 250 nm thickness layer, 5 ml of solution (6% ELV solvent) was spin-coated at a speed of 4500 rpm for 1 minute. The wafer then was baked at 90 °C for 30 min.
- (iii) for the 420 nm thickness layer, 5 ml of solution (8% ELV solvent) was spin-coated at a speed of 4300 rpm for 1 min and baked at 90 °C for 40 min.

For the application of polystyrene as sacrificial layers on silicon wafers, a polystyrene solution was prepared by dissolving 0.7 grams of polystyrene film with 5 ml of toluene. Dissolution was aided by agitation in an ultrasonic bath. For 1.2, 1.6, and 5.8 µm thick polystyrene films spin speeds of 4000, 2000 and 1000 rpm respectively were employed for 1 minute. Subsequently the films were baked at 90 °C for 15 minutes.

After the sacrificial layers were cured, the SU-8 was applied thus:

- (i) 3.5 ml of a SU-8 solution (50/50 SU-8/GBL with 5% photo-promoter) onto the sacrificial layer and spun at 600 rpm for 150 seconds.
- (ii) Soft baking was 75 °C for a duration of 7 minutes.
- (iii) Exposure using hard contact on Karl Suss MA6 mask aligner for 50 sec.
- (iv) Post-exposure bake was 5 min.
- (v) Development time was 30 sec in EC solvent.
- (vi) An oxygen plasma descum was used to remove any traces of the remaining non-crosslinked SU-8 resist.

The resulting SU-8 structures had a thickness of 12 µm.

The samples were then immersed in their appropriate etchant for measured units of time, following which the level of undercut around the structures was measured. Measurements were made using an

Table 1. Sacrificial materials used and their etchants.

Metals and Plastics	Cu	Al	Cr	Cr/Au	PMMA	Polystyrene
Etchant Solutions	Cu Etchant (Rookwood Electronic Materials Ltd.)	Al Etchant (Rookwood Electronic Materials Ltd.)	Cr Etchant 18 (Rohmand Haas Electronic Materials Ltd.)	Au Etchant (Rookwood Electronic Materials Ltd.)	Acetone	Acetone

optical microscope with an integral digital camera. The system was calibrated using an NPL-standard grid pattern. Two structure-types were measured; large block ($600\ \mu\text{m} \times 600\ \mu\text{m}$) and smaller lines ($18\ \mu\text{m} \times 100\ \mu\text{m}$). The complete list of materials and their etchants are shown in Table 1.

3. RESULTS AND DISCUSSIONS

Fig. 1a shows the SU-8 structures where Cr was used as sacrificial layer before the wafer was sunk in the chromium etchant solution. Fig. 1b illustrates the undercutting of the SU-8 structures after 10 minutes in the solution. In all experiments the small structures were released first, however the experimental data of this paper were obtained through measurements of the large blocks. Initially pictures were taken in the optical microscope and the thickness of the layers was measured.

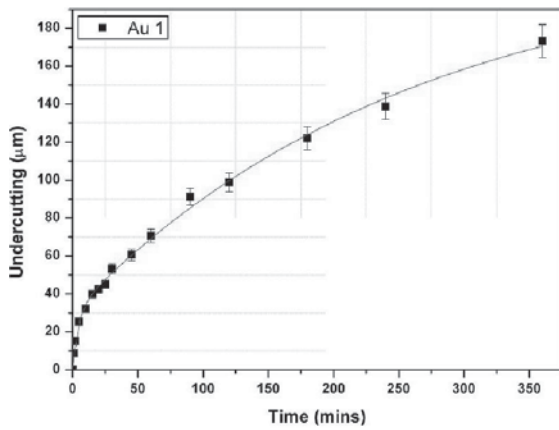
Fig. 2 demonstrates the effect of changing etchant concentrations on the undercutting rate for gold and chromium. Figs. 2a and 2c correspond to 100% concentrations of the gold and chromium etchant respectively, whereas Figs. 2b and 2d present a comparison to different etchant concentrations for 100%, 50%, and 25% for gold and chromium, respectively. For all concentrations there is high rate of release in the first 15 to 20 mins and afterwards this rate reduces. From the comparison it can be observed that as the concentration increases the undercut is higher. Specifically, in the case of gold a dilution of the etchant by 50% showed a two-fold decrease in side way etching rate suggesting a reactant limited system. A further reduction in gold etchant concentration to 25%, however only resulted in a slight reducing in side ways etch rate. One explanation for this result is that an increase in etchant solvation has allowed an increase in the diffusion rate of the reactants.

The effect of chromium etchant concentration on the undercut rate as shown in Figs. 2c and 2d can

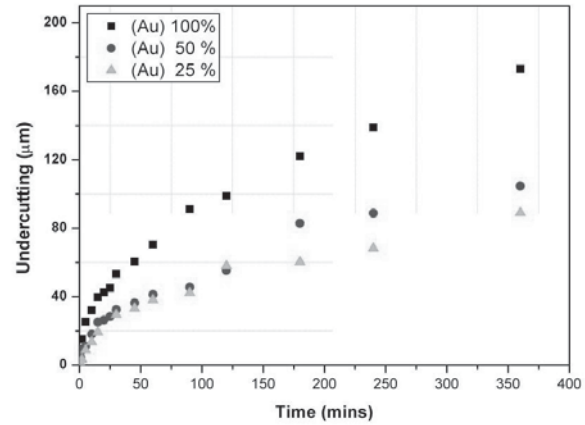


Fig. 1. Setup for undercutting measurements using optical microscope with integrated digital camera at (a) zero time and (b) after 10 min (example of Cr as sacrificial layer).

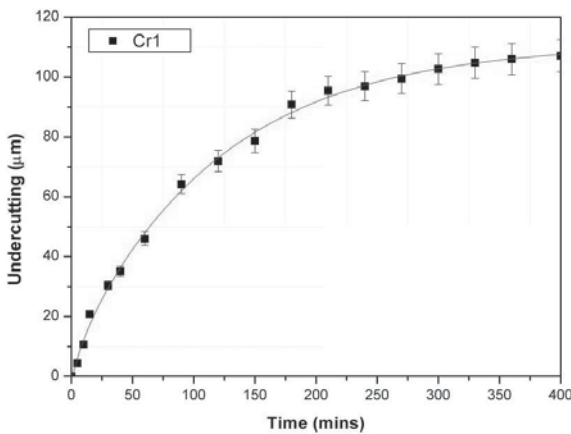
be more easily explained. In this case 100% for etchant only showed a moderated reduction in undercutting with reduction from 100% to 50% etchant concentration, suggesting a diffusion limited system.



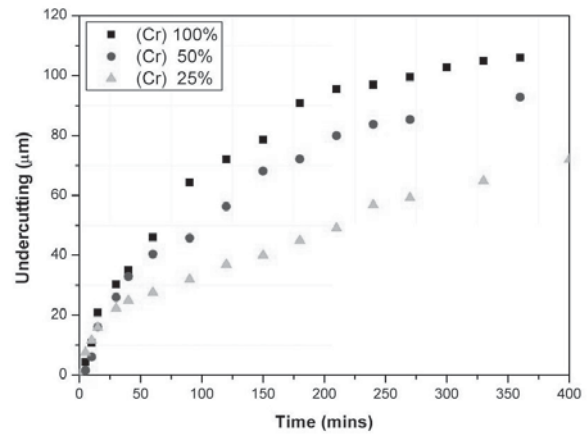
(a)



(b)



(c)



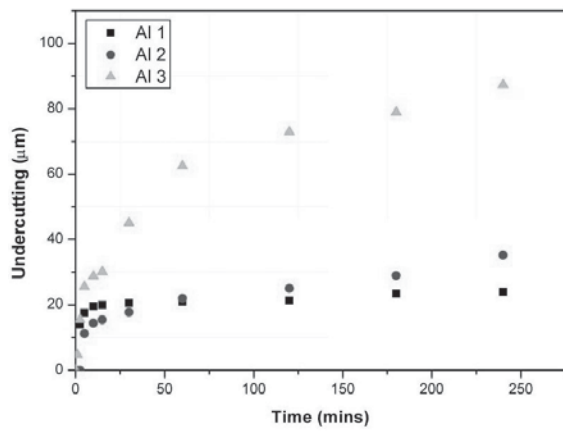
(d)

Fig. 2. Undercutting of SU-8 structures using as sacrificial layers: (a) Cr/Au with 100% gold etchant (b) Cr/Au with 100%, 50%, 25%; and, (c) Cr with 100% chromium etchant, and (d) Cr with 100%, 50%, 25%.

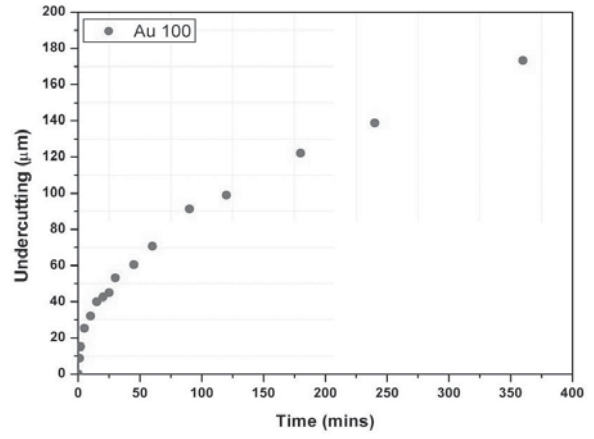
Fig. 3a shows the variation with time of the undercutting depth using as sacrificial layers Aluminium of three different thicknesses. For Al3 it can be observed that there is a high rate of undercutting depth in the beginning of the process which reduces subsequently. For Al1 and Al2 there is a reduced rate of undercutting as compared to Al3. For Al1 the undercutting progresses very slowly after the initial period of 15 minutes whereas for Al2 there is a slightly increased rate. The undercutting depth growth with time for a sacrificial layer Cr/Au (150/100 nm) is illustrated in Fig. 3b. A high initial rate is maintained for the first 30 minutes and subsequently it remains almost constant. Fig. 3c shows the re-

sults when Cr is used as sacrificial layer. There is an almost linear variation of the undercutting depth with time and there is little difference among the three different layer thicknesses. In Fig. 3d, Cu is used as a sacrificial layer, and it is apparent that there is a linear variation of the undercutting depth with an almost identical behaviour for the three layer thicknesses.

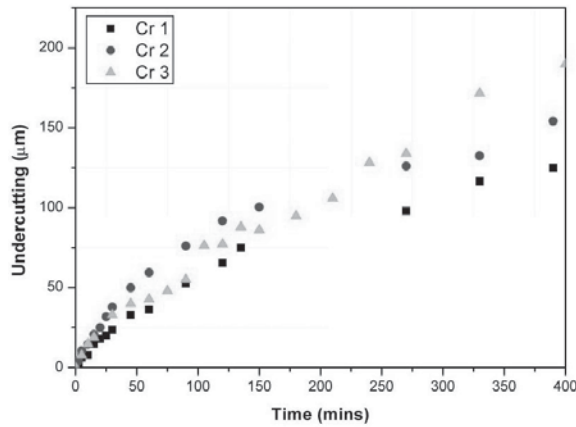
In Table 2 a comparison is carried out of the undercutting depth where metals were used as sacrificial layers of the same thickness. The results of three different times are presented and it can be observed that copper offers the fastest release rate followed by gold, aluminium and finally chromium.



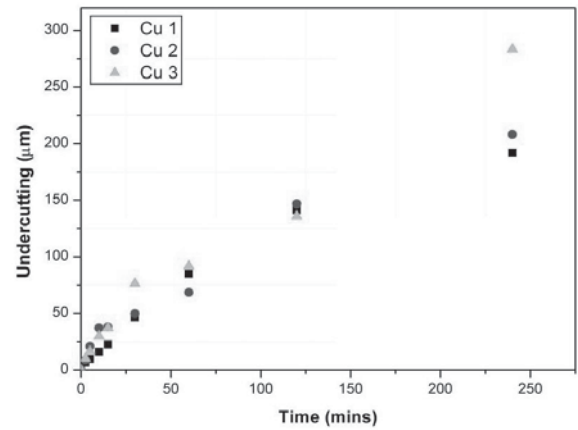
(a)



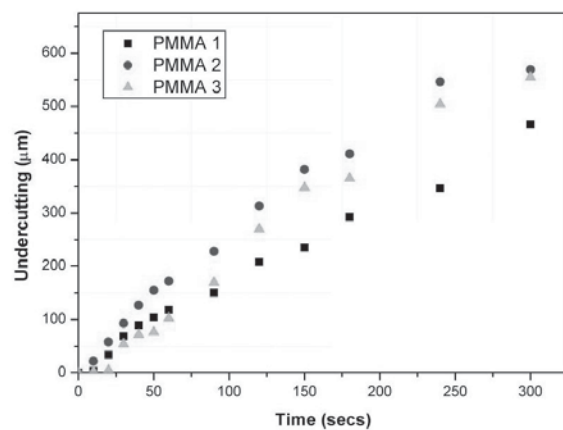
(b)



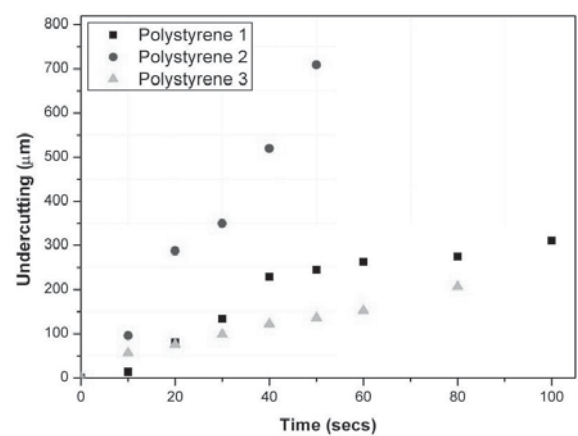
(c)



(d)



(e)



(f)

Fig. 3. Undercutting of SU-8 structures using as sacrificial layer with: (a) Al (A1=25 nm, A2=65 nm, A3=100 nm), (b) Cr/Au (150/100 nm), (c) Cr (Cr1:100 nm, Cr2:225 nm, Cr3:410 nm), (d) Cu (Cu1:150 nm, Cu2:350 nm, Cu3:600 nm), (e) PMMA (PMMA1:100 nm, PMMA2:250 nm, PMMA3:420 nm), and (f) Polystyrene (Poly1:1.2 μm, Poly2:1.6 μm, Poly3:5.8 μm).

Table 2. Comparison of the undercutting depth of SU-8 structures with time using metals as sacrificial layers with standard thickness 100 nm.

Time in etchant solution	Undercut in mm of SU-8 structures using metals as sacrificial layers with thickness 100 nm			
	Au	Cr	Cu	Al
45 minutes	60.5	32.8	69.7	53.7
90 minutes	91.2	52.5	122.5	70.4
240 minutes	138.8	85.4	192	87.33

In the present work, it was found out that gold presents reasonable adhesion properties with SU-8 resist. However, in the research paper presented by C. Luo *et al.* [7], it was claimed that gold by itself can not be used as a sacrificial material for releasing a functional SU-8 structure, because SU-8 has very poor adhesion to gold and then SU-8 cannot be uniformly coated on gold. The difference between the two conflicting results maybe attributed to the fact that in the present study the deposition of the gold layer was carried out in CVC instrumentation or the different level of contamination.

From the experimental work on the polymer materials, it was found out that the adhesion between SU-8 and these materials was very poor and the small structures started to detach from the wafer and were damaged by the time were placed in the acetone. The worst case was this of the polystyrene. On the other hand, for larger structures the polymer sacrificial layers demonstrated the advantage of very fast releasing times as compared to the metal ones. This is obvious in Figs. 3e and 3f. Also, the optimum film thickness for polystyrene and PMMA were 1.6 mm and 250 nm, respectively. It has to be mentioned that in the present work acetone was used for releasing the polystyrene films, as opposed to other works [7] where toluene was used instead.

4. CONCLUSIONS

In this work, different materials metals and polymers have been used as sacrificial layers for releasing SU-8 structures. The comparative assessment showed that from the metals copper provides the fastest release rate followed by gold, aluminium and finally chromium. The SEM pictures taken before and after the release of the SU-8 structures

showed that almost no damage was caused by the etchants used for diluting the sacrificial metals and polymers on the SU-8 structures. In addition, the effect of changing etchant concentrations on the undercutting rate for gold and chromium was demonstrated and was explained.

The method of using polymer films, like polystyrene and PMMA as sacrificial layers has the advantage of simplifying the process steps and also offers fast etching rate for releasing large areas of SU-8 structures above 600 μm . However, the drawbacks are the bad adhesion between the polymers and the SU-8 resist as well as they are not suitable for small structures in the range of few mm. In addition, SU-8 has a tendency to attack photoresist sacrificial layers like the polyimide resist.

Finally, this comparative assessment of different sacrificial materials for releasing SU-8 structures has a potential utilisation for MEMS applications where SU-8 is to be used as a structural material for monolayer or multilayer micro-structures.

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