Moisture resistance of SU-8 and KMPR as structural material for integrated gaseous detectors

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Abstract

This paper treats the moisture resistance of SU-8 and KMPR, two photoresists considered as structural material in microsystems. Our experiments focus on the moisture resistance of newly developed radiation imaging detectors containing these resists. Since these microsystems will be used unpackaged, they are susceptible to all kinds of environmental conditions. Already after one day of exposure to a humid condition the structural integrity and adhesion of SU-8 structures, measured by a shear test is drastically reduced. KMPR photoresist shows much stronger moisture resistance properties, making it a suitable alternative in our application.

Keywords: SU-8; KMPR; CMOS post-processing; adhesion strength; moisture; microsystems.

1. Introduction

Recently we showed a radiation imaging detector fabricated by IC compatible low temperature wafer post-processing [1]. This unpackaged microsystem is used in nuclear physics, high energy physics, astrophysics and radiology. This device uses 55 μm high isolating pillars as structural support for a 1 μm thick punctured aluminum grid, placed on top of a standard CMOS chip. Figure 1 shows a SEM picture of the device. SU-8 [2, 3] is an attractive candidate for fabrication of the support pillars [4] due to the low temperature process [3] and low residual stress in the underlying CMOS. Additionally it has good insulating properties [5] and it is radiation hard [6]. The prototypes fabricated with SU-8 50 show excellent radiation imaging performance.

As an alternative for SU-8 we also consider KMPR [3], a negative tone photoresist which is easier to strip, making it more suitable than SU-8 for electroplating molding [7]. The processing time for KMPR is shorter than SU-8 without risk of cracking. The maximum thickness of 100 μm that can be spin coated covers the range of interest for our system.

Humidity is a functional hazard for these micosystems, as the devices are not packaged. In this work we compare the structural integrity of microsystems using both SU-8 50 and KMPR support pillars after high-humidity bakes. The photoresist is tested on a variety of underlying thin films: PCVD Si_3N_4, PECVD a-Si:H, or pure aluminum. These materials are chosen because of their applicability at the chip surface of the radiation imaging system [8].

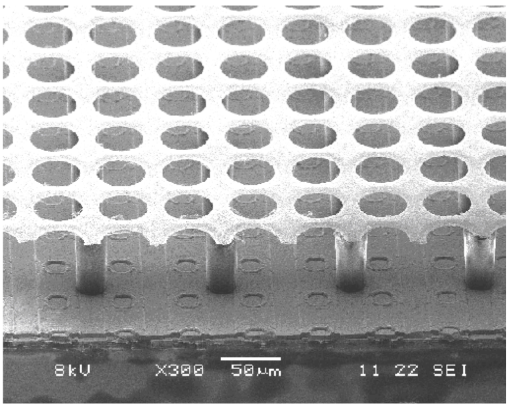

Figure 1. SEM picture of the detector, a punctured metal film placed over a CMOS chip is supported by insulating SU-8 or KMPR pillars.

2. Materials and processing details

The typical fabrication process for the SU-8 involves three days and comprises the following steps:

-SU-8 spin coating;

-Soft bake of the resist (10 minutes 50 °C, 10 minutes at 65 °C, 20 minutes at 95 °C and ramp down to room temperature);

-Expose the resist (24 seconds at 12 mW/cm^2, near UV broad band 350 nm-450 nm);

-Post exposure bake of the resist (5 minutes 50 °C, 5 minutes at 65 °C, 10 minutes at 80 °C and ramp down to room temperature).

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KMPR processing can be completed in one day, as follows:

-KMPR spin coat;

-Soft bake of the resist (15 minutes at 100 °C);

-Exposure of the resist (80 seconds at 12 mW/cm^2, near UV broad band 350 nm-450 nm);

-Post exposure bake of the resist (4 minutes 100 °C).

The processing of these photoresists follows the current advice of the resist manufacturers. Wet-development of the resist takes place after metal (sputter) deposition and patterning. More details about the complete fabrication process of the detector can be found in [1].

3. Results

Test structures were fabricated consisting of SU-8 or KMPR squares with 450 μm sides (unless stated otherwise) and 55 μm height. Their adhesion to the underlying layer was tested using a Dage 4000 shear tool. The shear machine increases the force linearly until structures delaminate from the substrate or the machine force limit is reached.

3.1. Adhesion strength

First we have studied the adhesion strength of SU-8 and KMPR over several underlying thin films. The underlying materials were chosen either because they are present at the surface of a CMOS chip (silicon nitride, aluminum, copper), or because we consider adding them in this microsystem. Figure 2 shows force needed to delaminate or break the non-exposed tests structures from different substrates. Clearly KMPR shows superior adhesion when compared with SU-8. For both SU-8 and KMPR we find that specific details of the processing (soft bake, hard bake, etc.) have an impact on the adhesion strength.

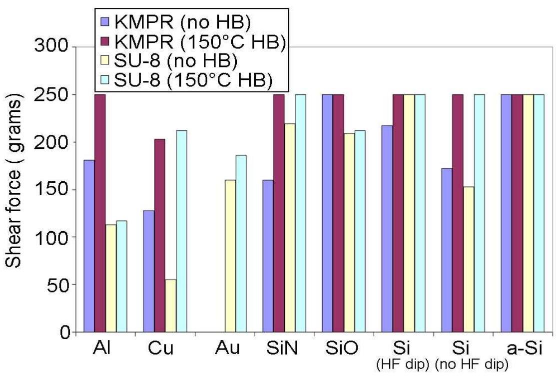

Figure 2. Adhesion strength of SU-8 and KMPR over different substrate materials before and after hard bake.

In all cases the SU-8 structures show delamination at the interface; in several occasions the KMPR structures are broken rather than delaminated. All silicon-based materials, which have an SiO_2 native oxide, show good adhesion, and much better than the investigated metals. In all cases a 150 °C hard bake increases considerably the adhesion for both SU-8 and KMPR.

3.2. Primer treatment

As the SU-8 adhesion on metals was relatively poor in the abovementioned experiment, an additional experiment was conducted with this photoresist. In standard semiconductor manufacturing, prior to photoresist coating the substrate surface is coated with a thin primer layer to increase the resist adhesion [9]. Two primers commonly used are trichlorophenylsilane (TCPS) and hexamethyl-disilizane (HMDS). The adhesion experiments were repeated using both primers, to investigate if the bond strength could be improved.

In the case of TCPS, wafers were first cleaned with oxygen plasma. Then the TCPS vapor primer was applied and baked at 200 °C during 30 minutes. For HMDS priming, wafers were cleaned in fuming nitric acid and hot nitric acid, the HMDS vapor primer was applied, without baking step. Finally SU-8 was spin coated on either primer following the process described in section 2.

Figure 3 shows the results of the adhesion of SU-8 on an aluminum substrate for different square test structures with dimensions of 450 μm, 200 μm and 100 μm side. Only small differences are observed: the adhesion is marginally increased with TCPS primer. HMDS primer has no effect.

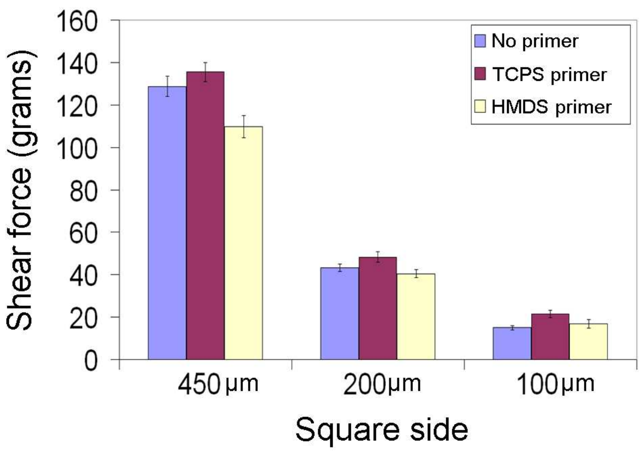

Figure 3. Adhesion of SU-8 on aluminum substrate for different primer treatments and different square sizes.

Remarkably the shear force does not increase proportionally with the test structures size. This points towards the influence of the internal stress of the SU-8 layer, more important in big structures than in small structures, playing a role in the adhesion strength.

3.3. Exposure to humidity

The reduction of the adhesion strength under exposure to a high relative humidity (95% RH at 30 °C) was studied for KMPR and SU-8 samples. SU-8 on aluminum shows a

50% reduction in adhesion strength after only one day, further decreasing to ~5% of its original value after 3 weeks of exposure. In some samples adhesion was completely lost and the top grid or the pillars even peeled off from the substrate during transport.

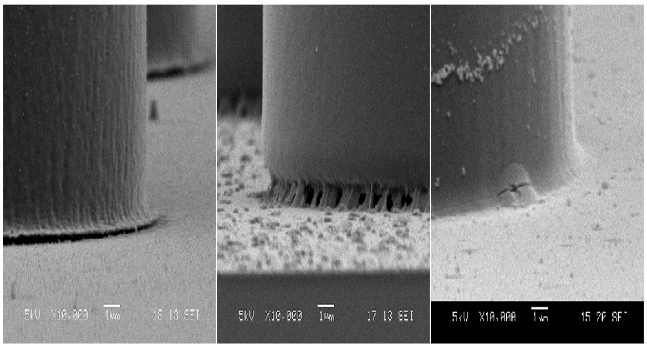

Figure 4. SEM picture of SU-8 and KMPR pillars. Left: SU-8 pillar before exposure to humidity. Middle: SU-8 pillar after exposure to humidity. Right: KMPR pillar after exposure to humidity.

SEM inspection (figure 4, left) shows that the SU-8 pillars have swollen as much as 5% after the humidity treatment, likely by water absorption [10, 11]. The SU-8 parts from the aluminum interface, as shown in figure 4 (center). On Si_3N_4 or Si, the adhesion of SU-8 is better; but the swelling is the same, causing a dramatic reduction in the adhesion already after 1 or 3 days. This is shown in figure 5.

KMPR samples exposed to a few days of high humidity show a less dramatic reduction in the adhesion (figure 5). There even seems to be a slight improvement in the adhesion after 3 days exposure compared to the initial decrease after one day. The SEM picture of an exposed KMPR sample (figure 4, right) shows cracking at the base of the pillar, consistent with the observation that also for the exposed samples the KMPR breaks before showing delamination at the interface. Initial temperature cycling tests between 30 °C 95% RH and 0 or -10 °C also hint towards a significantly stronger robustness for the KMPR systems.

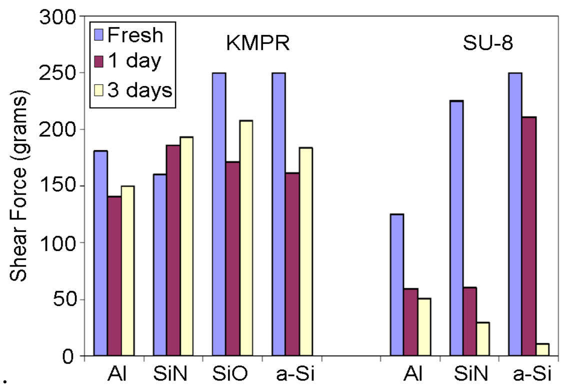

Figure 5. Adhesion strength of SU-8 and KMPR on different substrate materials; before humidity exposure (fresh) and after exposure to 95% relative humidity during one or three days.

To confirm the difference in moisture response between SU-8 and KMPR longer humidity exposure times were studied as well as other substrate materials.

Figure 6 shows that even after 15 days of exposure to 95% relative humidity the KMPR samples on aluminum substrate maintain the original adhesion strength.
For the SU-8 samples the same trend is found when the substrate is aluminum or a material with originally better adhesion, such as a-Si. We can conclude that adhesion loss is determined by the photoresist itself and not the substrate material

The 95% relative humidity conditions are the most aggressive for the photoresists. When samples are exposed to 75% or 85% relative humidity the adhesion reduces at a lower rate. Figure 7 shows a comparison between the three different humidity conditions for SU-8 on aluminum. After 21 days at 75% relative humidity adhesion is reduced to about one third of its original value. Unexpectedly adhesion is apparently reduced at a faster rate for 75% relative humidity than for 85% relative humidity. With the given sample-to-sample variation this may be insignificant.

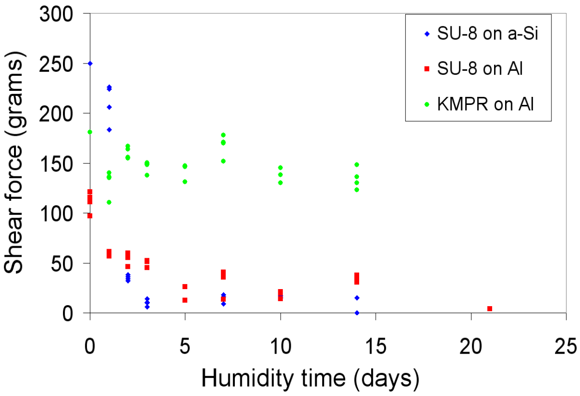

Figure 6. Adhesion strength of SU-8 over an aluminum substrate, a-Si substrate and KMPR over aluminum substrate when exposed to 95% relative humidity during several days.

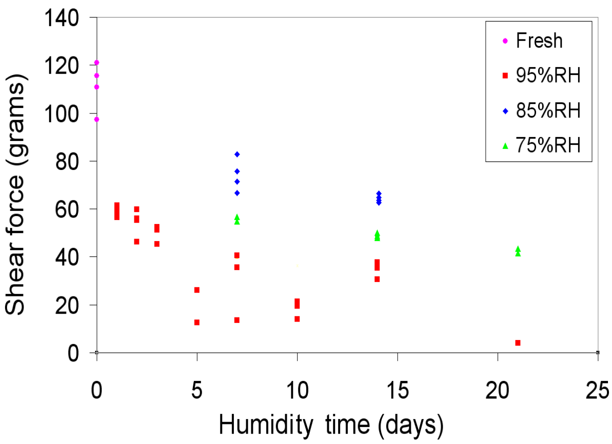

Figure 7. Adhesion strength of SU-8 over an aluminum substrate when exposed to different relative humidity percentages during several days.

4. Conclusions

We have shown that microsystems using SU-8 as structural material can encounter severe adhesion problems when exposed to even mild humidity conditions. The adhesion of SU-8, which is particularly poor on metals, is not improved significantly by the use of TCPS or HMDS primer.

When subjected to the same humidity conditions, KMPR photoresist shows superior performance. Its adhesion shows not significant degradation even after several days. In combination with other favourable properties, this finding makes KMPR a suitable candidate to replace SU-8 in our radiation imaging microsystem.

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