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Moisture resistance of SU-8 and KMPR as structural material

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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 1 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.

1. Introduction

Recently we presented a radiation imaging detector fabricated by IC compatible low temperature wafer post-processing [1]. This unpackaged microsystem is applicable 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. Fig. 1 shows a SEM picture of the device. The widely applied photoresist SU-8 [2–5] is an attractive candidate for fabrication of the support pillars [6] due to the low temperature process [7] and low residual stress in the underlying CMOS. Additionally it has good insulating properties [8] and it is radiation hard [9].

Our radiation detector prototypes fabricated with SU-8 50 show excellent radiation imaging performance [1]. As an alternative for SU-8 we also considered KMPR [7], a negative tone photoresist which is easier to strip, making it more suitable than SU-8 for electroplating molding [10]. The processing time for KMPR is shorter than SU-8 without risk of cracking. The maximum thickness of 100 μm that can be obtained in a single spin-coating process covers the range of interest for our system.

Devices are typically operated inside a sealed chamber or with a continuous gas flow of a mixture like He/iC_4H_{10} or Ar/iC_4H_{10}. Still, humidity is a functional hazard for these micosystems, as the devices are unpackaged. During storage and transportation, humidity can affect the supporting photoresist pillars, leading to reduced flatness of the metal grid or even pillar detachment from the substrate. Clearly the detector functionality is then at stake.

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: Si_3N_4, a-Si:H, or pure aluminum. These materials are chosen because of their applicability at the chip surface of the radiation imaging system [11].

2. Materials and processing details

The starting substrates are 4-inch silicon wafers with 10 Ω cm resistivity. PECVD Si_3N_4 (200 nm), LPCVD a-Si:H (500 nm), or pure aluminum (1 μm) were deposited. The aluminum-covered wafers were cleaned in fuming nitric acid for 10 min. The Si_3N_4 and a-Si:H covered wafers were first cleaned in fuming nitric acid (10 min), followed by hot nitric acid during another 10 min and a short HF dip to remove native oxide.

Just before spin coating the photoresist a 10 min long dry baking at 120 °C is done.

Our non-standard fabrication process for the SU-8 involves 3 days (including the metallization step on top of the SU-8) and comprises the following steps:

- SU-8 spin coating;
- Soft bake of the resist (10 min 50 °C, 10 min at 65 °C, 20 min at 95 °C and ramp down to room temperature);
- Expose the resist (24 s at 12 mW/cm^2, near UV broad band 350–450 nm);
- Post-exposure bake of the resist (5 min 50 °C, 5 min at 65 °C, 10 min at 80 °C and slow ramp down to room temperature). Resist is allowed to relax overnight to reduce the amount of residual stress [12,13].

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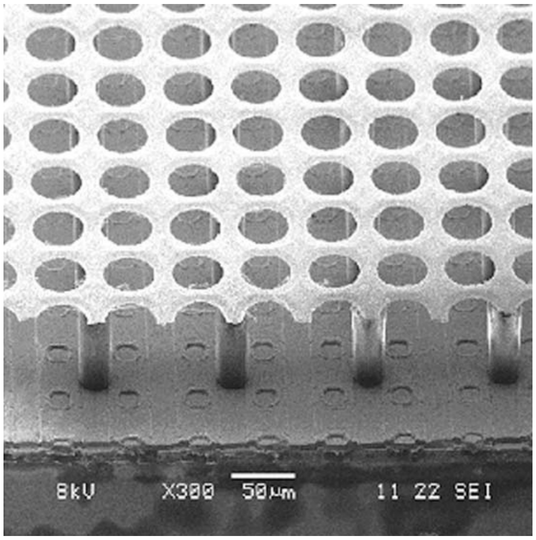

Fig. 1. SEM picture of the radiation detector. A punctured metal film is suspended over a CMOS chip by insulating SU-8 or KMPR pillars.

KMPR processing can be completed in 1 day, following the standard procedure [7]:

- KMPR spin coat;
- Soft bake of the resist (15 min at 100 °C);
- Exposure of the resist (80 s at 12 mW/cm^2, near UV broad band 350–450 nm);
- Post-exposure bake of the resist (4 min at 100 °C).

The unexposed parts of the SU-8 or KMPR photoresist are used as sacrificial material. The wet-development of this photoresist takes place after an aluminum metal layer has been deposited and patterned over it. More details about the complete fabrication process of the detector can be found in [1].

3. Results

The adhesion of the SU-8 [14] and KMPR to the underlying layer was tested using a Dage 4000 shear tool [15]. The shear machine increases the force linearly until structures delaminate from the substrate or until the machine's force limit is reached.

In final detectors, 30 µm diameter pillars support the metal grid, but these pillars offered too small resistance against the shear test. For that reason test structures were shear-tested instead. These structures consisted of SU-8 or KMPR squares with 450 µm side (unless stated otherwise) and 55 µm height.

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 conventional CMOS chip (silicon nitride, aluminum and copper), or because we consider adding them in this microsystem. Fig. 2 shows the force needed to delaminate or break the non-exposed test structures from different substrates. Earlier work by Palacio et al. shows delamination of SU-8 under forces with the same order of magnitude (be it under different experimental circumstances) [16].

The figure shows that generally, KMPR shows better adhesion than SU-8. For both SU-8 and KMPR we find that specific details of the processing (soft bake, hard bake, etc.) have a considerable impact on the adhesion strength.

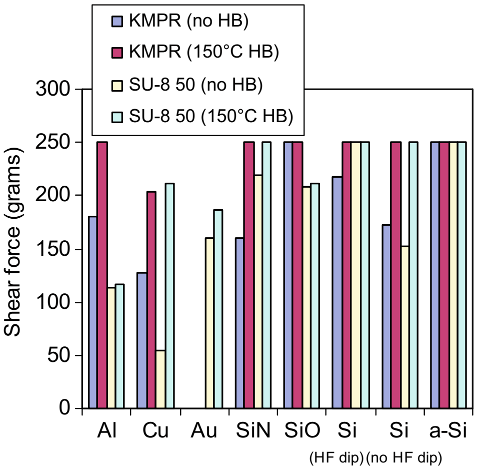

Fig. 2. Adhesion strength of SU-8 and KMPR over different substrate materials before and after hard bake. The measurement instrument's force limit is 250 g.

In all cases the SU-8 structures show delamination at the interface; KMPR structures on the other hand in several occasions break rather than delaminate. All silicon-based materials, which have a SiO_2 native oxide, show good adhesion, and much better than the investigated metals. In all cases a 150 °C hard bake increases the adhesion considerably 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 [17]. Two commonly used primers 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 min. 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.

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

The shear force does not increase proportionally with the test structures area, but it is almost proportional to the structure's side length. This proportionality is consistent with the observation (see Section 3.1) that SU-8 releases through (progressive) delamination from the surface.

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. There was at least 1 week delay between sample fabrication and first humidity exposure or shear force measurement. SU-8 on aluminum shows a 50% reduction in adhesion strength after only 1 day, further decreasing to ~5% of its original value after 3 weeks of exposure. In some samples adhesion was completely

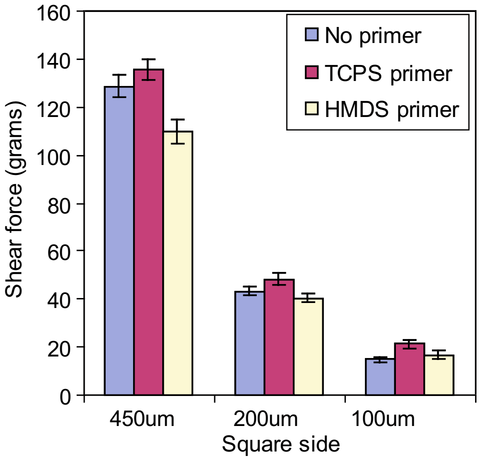

Fig. 3. Adhesion of SU-8 on aluminum substrate for different primer treatments and different square sizes.

lost and the top grid or the pillars even peeled off from the substrate during transport.

SEM inspection (Fig. 4, left and middle) shows that the SU-8 pillars have swollen evenly as much as 5% after the humidity treatment, likely by water absorption [18,19]. At the interface between substrate and photoresist the swelling is less pronounced as the resist cannot expand freely. The SU-8 seems to detach from the aluminum interface (at least at the outside of the pillar), as shown in Fig. 4 (middle). Only some threads keep the pillar in contact with the substrate. 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 quantified in Fig. 5.

KMPR samples exposed to a few days of high-humidity show a less dramatic reduction in the adhesion (Fig. 5). There even seems to be a slight improvement in the adhesion after 3 days exposure compared to the initial decrease after 1 day. This could be associated to a change in the material properties. It was observed that after 3 days humidity exposure the photoresist became more elastic and the shear tool deformed the resist test structure before delaminating it. However, it must be added that the quantitative results of identically treated samples vary with about 10% from wafer to wafer, so the difference between 1-day and 3-day adhesion is not very significant.

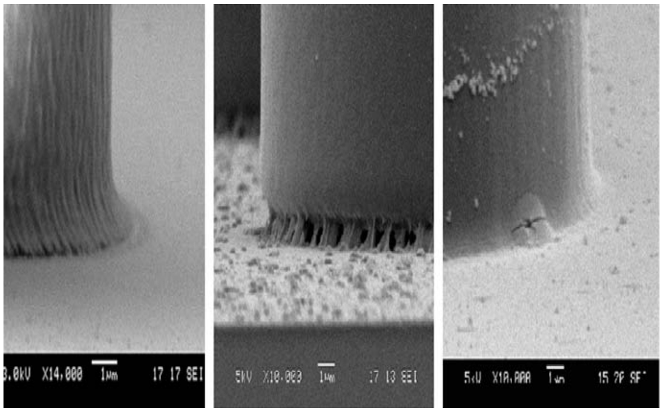

Fig. 4. SEM picture of SU-8 and KMPR pillars. Left: SU-8 pillar before exposure to humidity. Middle: SU-8 pillar after exposure to humidity. Right: KMPR pillar after exposure to humidity.

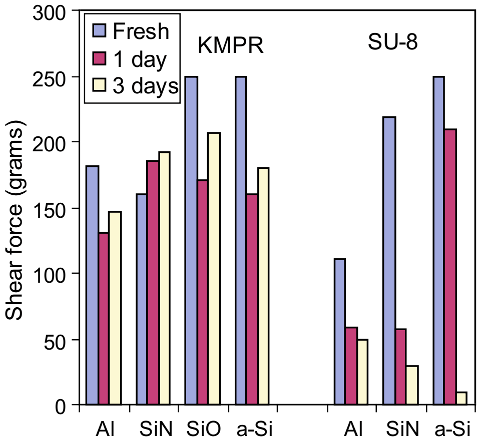

Fig. 5. Adhesion strength of SU-8 and KMPR on different substrate materials; before humidity exposure (fresh) and after exposure to 95% relative humidity during 1 or 3 days.

The SEM picture of a humidity-exposed KMPR sample (Fig. 4, right) shows a good contact between substrate and photoresist, suggesting better KMPR adhesion. 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.

Fig. 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 adhesion reduction trend is found whether the substrate is aluminum or a material with originally better adhesion, such as a-Si. We can conclude that adhesion loss is due to 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. Fig. 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 (~10%) this may be insignificant.

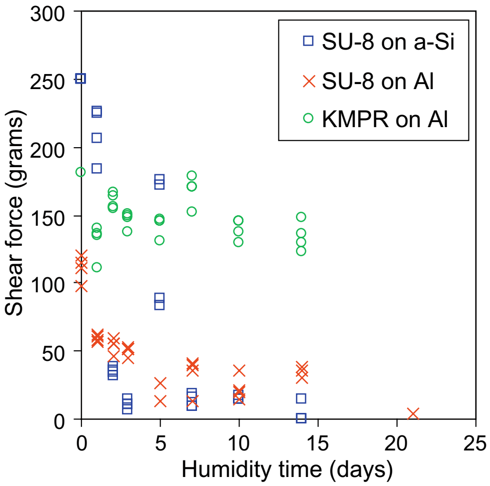

Fig. 6. Adhesion strength of SU-8 over an aluminum substrate, a-Si substrate and KMPR over aluminum substrate when exposed to 95% relative humidity during several days.

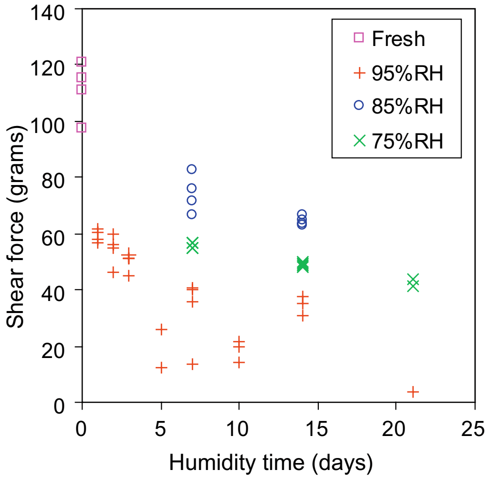

Fig. 7. Adhesion strength of SU-8 over an aluminum substrate when exposed to different relative humidity percentages during several days.

4. Conclusions

We have shown that microsystems using SU-8 as structural material can encounter severe adhesion problems when exposed to relatively 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 insignificant 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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