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Reliability aspects of a radiation detector fabricated by post-processing a standard CMOS chip

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ABSTRACT

This paper describes various reliability concerns of the newly developed INGRID detector. This radiation detector is fabricated by waferscale CMOS post-processing; fresh detectors show excellent performance. Since the microsystems will be used unpackaged they are susceptible to all kinds of environmental conditions. The device passed tests of micro-ESD, radiation hardness, dielectric strength; but humidity tests show one weakness of SU-8 as a structural material. Already after 1 day of exposure to a humid condition the structural integrity, as measured by a shear stress test, is dramatically lowered. Dry storage of these devices is therefore a necessity. KMPR photoresist shows promising results as an alternative structural material.

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1. Introduction

We have recently shown the 3D integration of a miniaturized gas-gain radiation detector on CMOS chips [1]. For radiation imaging applications gas-gain grids are commonly used. These grids are punctured metal membranes suspended some micrometers over an anode plane, inside a gas volume, filled with for example a helium/isobutane mixture. Ionizing radiation (e.g. a cosmic ray particle) that crosses the gas volume over the grid can liberate electrons that are then driven towards the anode by an electric field. The high electric field (~100 kV/cm) between grid and anode causes an electron avalanche in this region and hence an exponential increase in the number of free electrons that reach the anode. A charge sensitive amplifier connected to the anode records arrival time, position and pulse height. When a microchip is used as the anode [2,3] the signal is picked up directly at the origin, reaching very high sensitivity (Fig. 1). The chip in this figure has an array of (charge collecting) bond pads each connected to a pre-amplifier and buffer. With a manually mounted grid, misalignment between holes in the grid and the sensing array on the chip leads to Moiré effects. These are overcome, by processing the punctured grid on top of the chip through wafer post-processing.

This paper briefly describes micro-ESD, a failure mechanism known from the separately mounted predecessor of the microsystem that has been tackled during the development phase of this integrated microsystem. The bulk of this paper focuses on the degradation of these microsystems before its first use. These microsystems will be used without a package and are thus susceptible to degradation due to environmental conditions such as high humidity.

2. Materials and processing details

The process details of the INtegrated Grid (INGRID) can be found in [1] (and references therein). In this study we used aluminum as material for both anode and punctured grid. The 55 µm high support pillars are made of SU-8, a negative tone photoresist commonly used in MEMS fabrication [4]. We use SU-8_50, since this can be spun in the thickness range suitable for our microsystems. SU-8 processing takes place below ~95 °C making it suitable for CMOS post-processing; and developed SU-8 is radiation hard [5].

Note that multiple thinner layers, using e.g. SU-8_5 or SU-8_10, may also be spin coated to make a thicker layer and subsequently expose the film. The composition of the different kinds of SU-8 is different, mainly the amount of solvents that determine the viscosity. For each kind of SU-8 and each application the process flow has to be optimized. The impact of using a different kind of SU-8 on the reliability of the INGRID microsystem was NOT part of this study.

In Section 5 we report initial studies on detectors made with alternative electrode or electrode covering materials and KMPR as functional material to replace SU-8.

3. Micro-ESD

A problem associated with all gas-filled proportional chambers is sparking. When an electron avalanche reaches Raether's limit [6], it may evolve into a discharge. We assume that the density and energy of the participating electrons becomes high, forming

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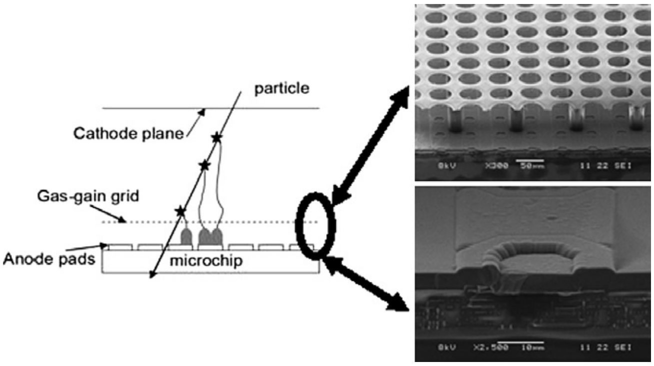

Fig. 1. Left schematic of the functioning of a gas-gain ionizing particle detector. Top right: SEM picture of the integrated microsystem. Bottom right: anode pixel pad on underlying CMOS chip.

a conductive plasma. This creates a conducting filament between the participating electrodes until the local field strength has dropped below the level that electrons are extracted from the grid electrode. As a result, a good fraction of the (capacitively) stored charge is transferred. This small electrostatic discharge (micro-ESD) can destroy one or several readout pixels by melting or evaporating the anode pads or even cause a breakdown in the electronic circuitry. Depending on the operating point of the microsystem, we find typical system lifetimes between a few hours and a few months.

Three methods seem promising to improving the robustness of the system against sparks in the gas volume. First a highly resistive layer over the anode prevents the instantaneous drain of the charge [7]. Secondly replacing the single grid by a stack of two grids allows the potentials to be chosen such that the discharges take place between the grids. Finally, micro-ESD is significantly reduced when He-based mixtures are used.

Both operation in He-based gases, and the use of high resistive protection layers, have recently proven to satisfactorily suppress micro-ESD occurrence.

4. Moisture-induced degradation

Another one of the potential problems that can be anticipated for this microsystem is the degradation of the functionality as a result of storage or transport. Mass manufacturing of our detector may take place in a warm and/or humid climate. The most important issue we foresee is exposure to high relative humidity (RH) possibly in combination with the rapid cooling down during shipment in an aircraft. Humid air can cause hygroscopic swelling resulting in a change in the device geometry (larger distance between anode and cathode) leading to changing functionality. Moisture absorption affects the stress in SU-8 – this is reported to be reversible until the point where delamination takes place [8]. When the isolating properties of SU-8 are reduced too much the device may not hold the operating voltage of 500 V.

4.1. Experimental details

We chose to first test at a moderate temperature of 30 °C and high relative humidity (RH) of 95%. Humidity tests we performed on single cell INGRID microsystems processed on dummy wafers. Additional test structures were processed without the suspended grid (only SU-8 pillars on the Al anode). The adhesion strength of the SU-8 on the Al anode was measured by a shear stress test pushing at 5 μm above the base of the pillar.

The small-signal conductance and capacitance of the cells is measured at 0 V as a quick screening test. A functional test was performed to see if electron multiplication was still possible in the microsystem after humidity testing.

4.2. Adhesion strength

First we have compared the shear force needed to break the SU-8 away from the Al for a fresh samples and one that was stored for 3 weeks at 30 °C 95% RH. For comparison we also added samples with different SU-8 treatment since the SU-8 properties are reported to depend on the processing details. Our normal SU-8 process only includes a post exposure bake. Including a hard bake after development is reported to eliminate cracks, improve mechanical stability [9] and adhesion [10]. (For the time-zero functionality of our microsystem a hard bake is not per se required.) As can be seen from Table 1 for SU-8, on Al the adhesion strength is reduced by more than 90% resulting from the hard bake immediately after the development. The dramatic reduction in adhesion strength might be due to the fact that the SU-8 becomes harder and more fragile [8]. On the other hand a temperature step on the finished microsystem just before the shear test seems to have negligible impact on the adhesion strength. It is clear that a well defined reproducible process is needed, and this includes specifications for storage time and condition (see Table 1).

Summarizing, our normal process (without hard bake) is the best for adhesion strength. It is furthermore clear that the 21 days exposure to high RH was far too long. Fig. 2 shows the shear force test results for normally processed SU-8 devices before and after storage at 30 °C and 95% RH. As this test is destructive, the experiments were done on different samples. Although the device-to-device variation is significant it is clear that already after 1 day of exposure the adhesion of the SU-8 to the Al has been reduced by about 50%. After 2 weeks of exposure severe cracking of the SU-8 and stress in the aluminium are visible in an optical microscope (Fig. 3). After 3 weeks in some samples the grid has disappeared partially or completely and the SU-8 starts to delaminate from the Al, starting at the outer ring where the SU-8 was not covered by Al. In two samples the majority of the pillars has also vanished, which could be observed with the naked eye. In the test samples of only SU-8 pillars on the Al electrode the pillars have already vanished after 7 days of exposure (shortest exposure in this experiment). Scanning electron microscope (SEM) inspection shows significant swelling of the SU-8 pillars (Fig. 4). After 3 weeks of exposure the diameter of the pillars increased by 5% from 30 μm to 31.5 μm. The height of the pillars increases approximately 2.5% but this is more difficult to determine from the SEM micrographs.

A limited amount of samples was exposed to a lower humidity; at 30 °C with 85% and 75% RH. As expected a lower humidity shows reduced degradation. But at the shortest exposure of 7 days the adhesion strength at 75% RH is still reduced by 50% compared to the fresh sample.

4.3. Electrical test

The leakage current between the anode and the grid measured at −100 V and +100 V increase by a factor 2–5 after 2 weeks of

Table 1
“Force” needed to push over the SU-8 measured in a shear test pushing at 5 μm from the base

Normal procedure	125 g
normal + 120 °C hard bake	12 g
normal + 150 °C hard bake	7 g
normal + time delay + 150 °C	120 g
normal + 3 weeks @ to 30 °C 95% RH	4 g

Table 2
Measured and calculated capacitance at 10 kHz for two cell geometries using the best fit for the relative dielectric constant of SU-8 based on the observed swelling

Nr pillars	Measured capacitance		Calculated capacitance	
	Fresh (pF)	Exposed (pF)	Fresh (pF)	Exposed (pF)
10,500	61	72	61.8	71.8
21,000	67	78	66.3	77.5

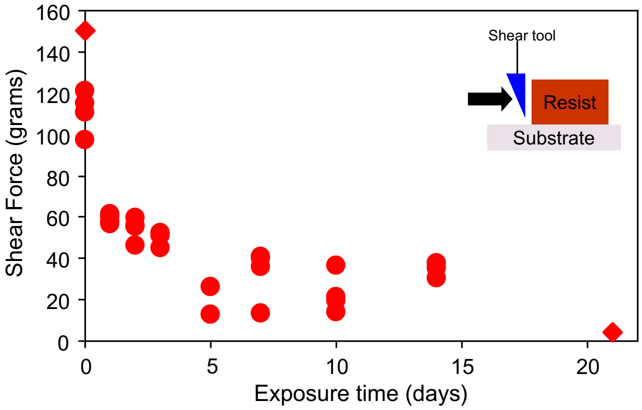

Fig. 2. Force needed to push over SU-8 (on Al anode) as a function of the exposure time at 30 °C, 95% RH. Inset shows a schematic of the test setup. Force was applied 5 μm from the base.

exposure. Since the cells were not flushed with nitrogen it is not clear whether this is caused by side-wall leakage or partially also by the humid air in the cell. A functional test after flushing the cell with the helium/isobutane mixture failed because the leakage current between the anodes became too high at 250 V between the electrodes and the cell collapsed. At this voltage no electron multiplication occurs yet. In other words, the microsystem failed the functional test after exposure to humidity.

The capacitance at 0 V was measured for two sets of devices of 19 mm diameter and 10,500 and 21,000 support pillars, respectively (Table 2). The supporting outer ring of SU-8 covered with the grid also contributes to the capacitance and is equal for both devices. An increase in capacitance of around 11 pF was measured after an exposure of 14 days. Simplifying the system to two parallel capacitors with air and SU-8 as the dielectrics we have calculated an increase in the dielectric constant from 6.35 for fresh SU-8 to 7.40 for SU-8 that has been exposed for 2 weeks. Such an increase is consistent with water absorption.

4.4. Impact of materials choice

Recently KMPR has caught the attention of researchers in the MEMS community as an alternative to SU-8. KMPR is an epoxy based I-line photoresist that can be more easily stripped than SU-8 [11]. Additionally it allows for shorter processing times due to the lower amount of solvents in the material compared to SU-8 allowing for shorter bake steps. It is less prone to cracking and has good adhesion properties. We have manufactured INGRID prototypes replacing the SU-8 support pillars by KMPR pillars. Secondly we have studied the possibility of replacing the aluminium traditionally used as anode and cathode by other metals and we have studied the adhesion properties of the pillars on other materials. Since only the anode pads need to be metal and not the entire bottom plane, scratch protection layers like Si_3N_4 or SiO_2 can also be used as the base for the pillars. A different base material may well lead to improved adhesion. A low temperature PECVD amorphous silicon (a-Si) layer effectively reduced the micro-ESD problem, so the adhesion of SU-8 on a-Si is also studied. For simplicity these materials are also called anode material in the

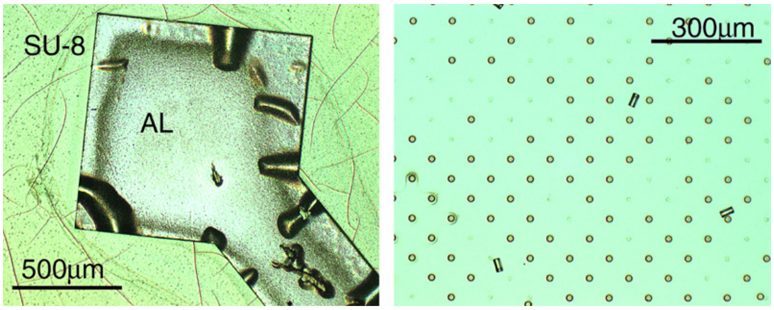

Fig. 3. Optical microscope images of exposed devices. Left: stress in the bondpad connected to the top electrode and cracks in the SU-8. Right: partially disappeared pillars and totally removed grid.

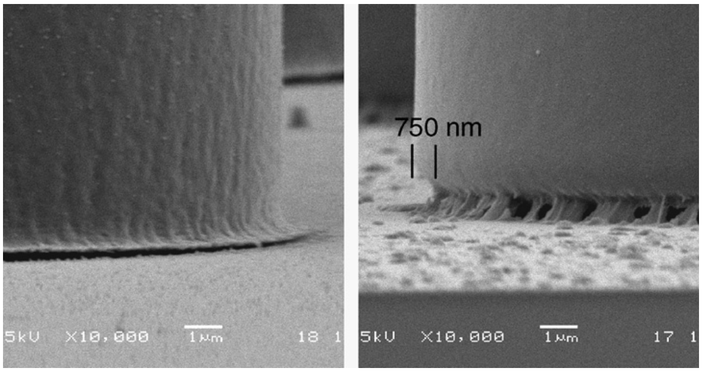

Fig. 4. SEM micrograph of SU-8 pillars on Al. Left: fresh sample, right: after 3 weeks at 30 °C, 95% RH. A gold coating was added to minimise charging during SEM.

upcoming figures. Fig. 5 shows the results of the shear force test using different anode materials for both SU-8 and KMPR pillars. Since our KMPR process is not yet mature we show the results of a process with and without hard bake. For SU-8 the adhesion improves if the pillars are on silicon nitride, silicon oxide as well as on amorphous silicon. In this case the adhesion is so good that the pillars can not be removed by the shear force test. For SU-8 in all cases the pillars tumble over when the force becomes too large, a sign of poor adhesion. In case of KMPR however the pillars are destroyed or broken before delamination can take place. So for KMPR Fig. 5 shows the force needed to destroy the pillars.

Fig. 6 shows the results of shear tests after exposure to 30 °C and 95% RH for 1 and 3 days for the different material combinations. For the SU-8 pillars the benefit of stronger adhesion for fresh samples in case of a silicon nitride or silicon base/anode material is already reversed after 1, respectively 3 days. After being exposed to a high humidity environment the KMPR pillars still break before the pillars show delamination. After an initial reduction of the strength of the pillars, prolonged exposure even seems to increase the resistance to breaking again.

4.5. Thermal cycling

We have also exposed a few samples to thermal cycling. All samples were cycled between 30 °C 95% RH and 0 °C for 1 day. The dwell time at high and low conditions was 1 h and the ramp-up and ramp-down speeds were 3 °C/min and −1.6 °C/min, respectively. The cooling down causes water condensation that might lead to corrosion of the aluminium anode. In case of SU-8 on Al indeed the adhesion strength was strongly reduced to 6 g in shear force test compared to 125 g for a fresh sample and 59 g after 1 day at 30 °C and 95% RH. SEM inspection however did not show obvious signs of corrosion. For SU-8 on other materials as well as for the KMPR samples a slightly better result was obtained after the cycling test than for a continuous exposure at 30 °C. This is in line with the shorter exposure time at the highest humidity condition.

The samples with SU-8 pillars on Al and a-Si were cycled a second day between 30 °C at 95% RH and −10 °C. The idea was that after condensation, water creeps into cracks or voids and expands when frozen; this could speed up the degradation. For the standard sample with SU-8 pillars on aluminium the degradation after the first day is already so large that the impact of freezing the detector does not cause a measurable decrease in adhesion strength. For the SU-8 pillars on the silicon substrate, the degradation is not larger than could be expected after 2 days exposure at high humidity.

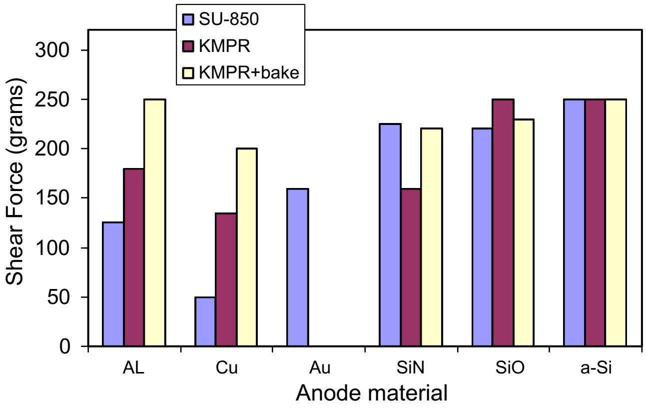

Fig. 5. Adhesion strength of SU-8 and KMPR on several anode materials. Note that 250 g is the maximum force that can be detected. In most cases the KMPR pillars break before delamination.

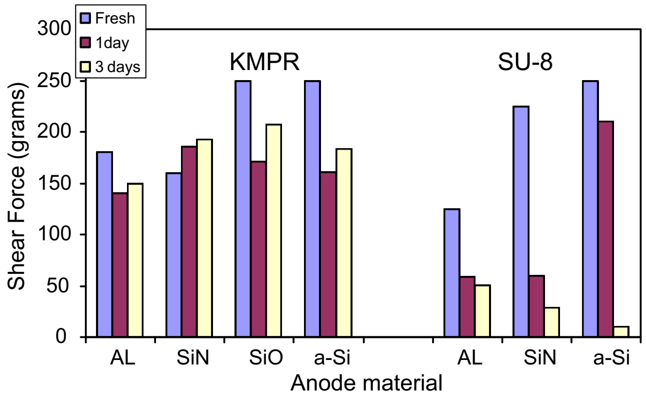

Fig. 6. Adhesion strength of KMPR and SU-8 on several anode materials before and after exposure to 30 °C and 95% RH. For the SU-8 the adhesion to the substrate is the weakest link, in case of KMPR the pillars break. Two-hundred and fifty grams is the maximum force that can be detected.

5. Discussion

The effect of exposure to a high humidity environment on the microsystem is mainly caused by the hygroscopic swelling of the SU-8 and the resulting stress and delamination at the interface. No evidence of interface degradation due to the corrosion of the aluminium was found in high humidity but a cycling test showed accelerated degradation. More cycling tests are needed to investigate problems when the absorbed water in the SU-8 inside cracks expands when frozen. The leakage current increases after the SU-8 absorbs water preventing the detector from operating in a regime where charge multiplication can take place. Further tests should reveal if a simple *I–V* measurement can be used as a reliability predictor. The capacitance of the cell increases with the increase of the relative dielectric constant and the diameter of the pillars but this effect is counteracted by the increase in the height of the pillars. In combination with the device-to-device variation of several pF, the cell capacitance is not very suitable as a screening test for a good cell.

The improved adhesion of SU-8 on other anode materials disappears, or even turns into a reduction, after only short exposures. The hygroscopic swelling in this case causes a rupture at the site of adhesion. Using KMPR to build our microsystem might reduce the degradation in high humidity environments since it seems less prone to absorb water giving better adhesion even after exposure to moisture. Future studies will have to clarify if a microsystem build using KMPR as structural material can also withstand more rigorous thermal cycling tests.

This study was focussed on the adhesion strength of SU-8 pillars on a metal anode or intermediate layer. Replacing the punctured grid with another metal or stack will cause new technological and reliability challenges.

6. Conclusion

We have shown that severe reliability issues exist related to the exposure of integrated radiation detectors. The choice of materials compatible to CMOS post-processing poses new reliability challenges. Microsystems containing SU-8 as structural material can be expected to show moisture-related stability problems. The addition of an amorphous silicon layer acting as a high resistive ESD protective layer more than doubles the adhesion strength of SU-8 pillars but this benefit is lost after only a few days exposure to a high humidity environment. KMPR shows promising results as possible replacement for SU-8, including a reduced susceptibility to humidity.

Further studies are ongoing into alternative materials choices and possible sealing during transport and storage.

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