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AC-coupled n-in-p pixel detectors on MCz silicon with atomic layer deposition (ALD) grown thin film

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Abstract

We report initial characterization of our novel sensor process solutions with AC-coupled n\textsuperscript{+}/p\textsuperscript{−}/p\textsuperscript{+} pixel detectors made on 150 mm diameter p-type Magnetic Czochralski silicon (MCz-Si) wafers. The pixels were segmented in a 52 × 80 dual column array and designed to be AC capacitive coupled. The resistive coupling between pixels, allowing quality assurance probing prior the flip chip bonding, was realized with thin film metal-nitride resistors fabricated by sputtering deposition. This approach allows us to omit punch-through resistor structures, which reduces the overall process complexity. Moreover, our previous studies have emphasized that applying ALD Aluminum Oxide (Al\textsubscript{2}O\textsubscript{3}) field insulator and passivation layer results in negative net oxide charge and thus additional p-spray or p-stop surface current termination structures are not necessary. Our focused application is a radiation-hard ALD AC-coupled pixel detector to be used in future particle physics experiments, such as the High-Luminosity Large Hadron Collider (HL-LHC), as well as photon counting applications. The pixel detectors were tested at Helsinki Institute of Physics (HIP) Detector laboratory and Ruđer Bošković Institute (RBI). We show measurement
data of pixel detectors and other test structures. For the TiN resistors surrounding pixels, the resistance values were measured to be about 15 kΩ. Data of electrical properties, full depletion voltage and leakage current are shown as well. Our Transient Current Technique (TCT) measurements indicated clear pixel segmentation with excellent homogeneity. For further study, AC-coupled sensors were hybridized to PSI46dig read out chips (ROC) by flip-chip interconnection technique and tested with a radioactive source.

**Keywords:** Pixel Detector, Magnetic Czochralski Silicon, Atomic Layer Deposition, Flip Chip Bonding

1. Introduction

Pixel detectors made of silicon are frequently used for particle tracking applications at different high-energy physics experiments. In CERN HL-LHC, an extensive amount of Si modules will be installed [1–3]. Thus, the use of Si material is pertinent from both the material as well as the production cost perspective. Amongst the Si materials, Magnetic Czochralski silicon (MCz-Si) is a potentially radiation hard detector material due to its higher oxygen concentration with respect to traditionally used Float Zone silicon (Fz-Si) materials. The concentration of oxygen in MCz-Si is about 5-10×10^{17} \text{cm}^{-3} and it can be adjusted by magnetic field in the growing process [4]. The higher oxygen concentrations in high-resistivity MCz-Si wafer enables the formation of shallow-level defects known as thermal donors (TDs). These can also be introduced on purpose, in order to compensate the acceptor doping and thus tailor the full depletion voltage ($V_{fd}$) of the Si sensor. Also proton radiation deterioration has been shown to have less impact on sensors processed on this material [5].

There is a strong motivation for the use of p-type Si. Conventionally, n-type Si wafers are used as the detector material. A disadvantage of n-type detectors is the Space Charge Sign Inversion (SCSI) effect that tends to appear after irradiation with high fluences. This causes a decreased sensitivity of the detector. On the other hand, a detector made of p-type Si is not only expected to eliminate the SCSI problem, but also can reach higher charge collection efficiency (CCE). However, in this configuration the positive oxide charge in SiO$_2$ becomes an issue. The accumulation of surface electron charges results in poor spatial resolution. Thus, a p-spray or p-stop implantation step has to be added in the Si detector process. In this paper, we applied thin film oxidation insulator instead of conventional SiO$_2$. Especially, the thin film grown by atomic layer deposition (ALD) is recognized as an attractive approach as semiconductor processing technique. Coating with ALD aluminium oxide (Al$_2$O$_3$)
provides a field insulator providing natural surface current termination due to its
well-known negative oxide charge property \([4, 6–9]\). By replacing \(\text{SiO}_2\) with ALD
\(\text{Al}_2\text{O}_3\), we can omit the p-spray or p-stop process steps.

An AC-coupled detector design has the benefit that a signal can be separated from
leakage current. The signal current from an incident particle can be propagated to
the readout electronics through a pixel capacitor, while the leakage current flows to
the common bias line via a bias resistor fabricated in the peripheral area of the pixel
areas. Our AC-coupled structure consists of an \(\text{Al}_2\text{O}_3\) insulation layer between the
implanted silicon substrate and the pixel electrode. In this study we applied a thin
film ALD aluminum oxide as the insulation material and sputtered Titanium Nitride
(TiN) as the biasing resistor material.

For characterization of the fabricated sensors we used Transient Current Tech-
nique (TCT), which is a commonly adopted method \([10–12]\). Especially, observation
of the charge collection behavior with the good spatial resolution provided by mod-
ern laser systems can confirm the success in sensor fabrication \([13]\), if for example
a dedicated readout ASIC is not available. Both electrons and holes contribute to
the induced current. Furthermore, short wavelength laser TCT gives us information
about the efficiency of our sensors for short range particles such as alpha parti-
cles or other ions possibly emitted from nuclear materials. In addition, 1060 nm IR
laser TCT characterizes the effectiveness of our pixel sensors for Minimum Ioniz-
ing Particles (MIP) which are present in scientific High Energy Physics (HEP) and
astrophysics experiments.

To validate the functionality of our AC-coupled pixel detector, we hybridized the
sensor to the CMS PSI46 dig read-out chip (ROC) \([14]\) by flip-chip bonding (FCB).
For this purpose, under-bump metalization (UBM) based on a TiW/Au metal stack
was prepared on the sensors. The hybridized detector assemblies were then tested
with radioactive sources, most importantly Am-241.

2. Detector processing

A 320 \(\mu\)m thick boron doped p-type 150 mm MCz-Si wafer with resistivity 4–8 k\(\Omega\)cm
from Okmetic Oyj was processed at Micronova Nanofabrication Center in Espoo, Fin-
land. First, a 300 nm thick dry oxide (\(\text{SiO}_2\)) layer was grown in oxidation furnace
(PEO Centrotherm) at peak temperature of 1100 °C. The first photolithography was
for alignment marks on the Si surface. The standard alignment marks were exposed
using a stepper (FPA3000-i4 Canon). The oxide layer and Si were etched using an
oxide etcher (LAM Oxford). The use of helium cooling gas was reduced to avoid risk
of wafer breakage. The depth of the alignment marks on Si was 100 nm. After the Si
dry etching, the photo resist was removed, and a second photolithography step was carried out. The implantation opening pattern was transferred to the existing thermal oxide layer by using buffered oxide etcher (BOE) as known as buffered hydrogen fluoride (BHF) wet etching. After the wet etching, the existing photo resist was removed. Then ion implantation step was carried out with the patterned thermal oxide hard mask. On the front side, phosphorus was implanted by an ion implanter (Eaton 8200) with the voltage of 60 kV and total dose of $10^{15}$ /cm$^3$. Similarly, boron field implantation was conducted using the voltage of 20 kV on the backside of the wafer. A yield in junction depth of approximately 3 µm was expected.

Figure 1: Process flow for AC-coupled pixel detectors on MCz silicon with ALD grown thin film.

After removing the hard mask by BOE, the wafers were cleaned using the Radio Corporation of America (RCA) cleaning process. Consequently, the implants were activated by thermal diffusion into the bulk Si, called drive-in step in the oxidation furnace. The activation step again deposits a thin layer of the oxide of approximately 100 nm, this was removed using the BOE bath. An insulation layer was deposited using ALD. The detailed steps of the ALD deposition are described in [15, 16]. The Al$_2$O$_3$ insulation layer was patterned by wet etching in an aluminum etching bath. Then a TiN bias resistor was deposited with a physical vapor deposition (PVD) RF sputtering (Von Ardenne). The TiN film was patterned using hydrogen peroxide.
After the thin film deposition processes, aluminum metal was deposited both front and backside using PVD DC sputtering (Von Ardenne). The backside metal was protected with a photo resist while patterning the pixel contact metal on the front side in the aluminum etching bath. The pixel detector configuration requires hybridization of the sensor and ROC by FCB technique. The ROC has SnPb solder bumps deposited by Advacam Oy. The solder wetting metal is required on the surface of a sensor side. Thus the UBM was prepared using PVD sputtering (MRC Mark-II). Tungsten-titanium (TiW) of 20 nm thickness as an adhesion metal layer followed by gold (Au) of about 200 nm thickness as a solder wetting metal were deposited. The UBM patterning was done by the metal lift-off technique. In this report, we diced the wafer before the UBM metallization step, because our design includes multiple other test structures, MOS capacitors and diodes, which do not require UBM. In other words, any further steps which may interfere with the surface structure and measurement of these other devices at this phase were avoided.

During the FCB step, the PSI46dig ROC and sensor were pre-aligned by the optics equipped in the FCB tool (FC150 SET). Figure 2 shows the cross sectional drawing of the AC-coupled Si pixel detector. The provided value of the post bonding accuracy form the tool manufactures is higher than 3 µm. In this study, we used a flux-less thermo-compression bonding which is controlling temperature and force while the pixel solder bumps establish the electrical connections. An eutectic point of a SnPb solder is about 184°C, applying the temperature with appropriate force, imposed range of 50-100 mg/bump, can establish the bump bond connections together with a reflow step. Also, another parameter leveling is an important sequence during the alignment procedure. We used an autocollimator installed in the optical unit adjusting the parallelism between the ROC and sensor surfaces.

The processed thin film TiN bias resistor thickness was measured under the optical profilometer (Vision 64 Burker). The measured bias thickness was 37 nm (see Figure 3). The average sheet resistance of the TiN thin film was measured to be 54 Ω/sq, with a variation of up to 15% over the wafer. This variation might be caused by a variation in thickness at the position of the wafer, as is not unusual in sputter deposition. Non-stoichiometric nitride film resistivity may also be affected by the deposition tool. Although this study is focused on research providing a proof of concept, a tool that was not available due to the technical reasons during this study, such as ALD, may endorse a better film and uniformity.

The resistance values for an individual pixel resistor was around 15 kΩ. This is lower than presented in one of our previous reports, where resistances of 0.1 MΩ and 0.08 MΩ for 12 nm and 23 nm thick TiN resistor structures grown by ALD were determined, respectively [4]. As expected, the resistance of the TiN film is decreased...
as film thickness increases, and resistance remains low when the sample is annealed in non-oxidizing conditions. To bias the detector even at higher leakage currents without significant voltage drop, a slightly lower value of the bias resistor is in fact favorable.

![Figure 2: A schematic drawing of the AC-coupled Si pixel detector: ROC having solder bumps (above) and sensor (bottom).](image)

3. Transient Current Technique Measurement

Different types of pixel sensors on our wafer were characterized with the TCT setup (Particulars d.o.o [17]) at the Detector Laboratory in Helsinki Institute of Physics (HIP) and at the Laboratory in Ruder Bošković Institute (RBI). Both facilities have identical basic setups that consist of a pulsed laser source, optics with an adjustable diaphragm collimator, and a sample holder with an XYZ-stage [18, 19]. The diced n⁺/p⁻/p⁺ sensor was attached to the measurement setup, and provided
with bias voltage from a Keithley 2410 SourceMeter to the back plane of the sensor chip. The signals were read from front side of the chip on a corner bias contact pad with the help of a contact needle.

Figure 4: Two types of pixel sensor designs: (a) PSI46dig-geometry AC-coupled pixel sensor with 100 µm × 150 µm pitch (b) RD53A-geometry DC-coupled pixel sensor with 50 µm × 50 µm pitch.

Measurements were performed at room temperature and the wavelength of $\lambda = 670$ nm for red laser and $\lambda = 1064$ nm for infrared laser were set with a repetition rate of 50 Hz. The two types of the sensors shown in Figure 4 were measured. In the Figure, the left hand side (a) shows a mask design of PSI46dig sensor (52 × 80 pixel matrix) and the right hand side (b) shows a design of RD53A sensor (400 × 192 pixel matrix). For (a) sensor, the applied bias voltage was 80 V. An area of 500 × 500 µm$^2$ was selected to produce a spatial homogeneity scan with a resolution of 5 µm. The CCE is defined as an integral over the TCT signal with a 25 ns time window. The transient signal is normalized by using the highest value of collected charged due to the measured signal in the scanned area. For the (b) sensor, the applied bias voltage was 100 V and an area of 1000 × 1000 µm$^2$ was scanned. The pitch between two bump openings is 50 µm, i.e. the size of the metallization on n$^+$ pixel is about 38 µm × 38 µm. The gap between the metals was 12 µm.
During the TCT study, we obtained signals from 20 to 110 V scans (Figure 5 (a)). In Figure 5 (b) the saturated CCE is indicating a full depletion voltage $V_{fd}$ of about 40 V. The leakage current was about 10 $\mu$A under the IR laser illumination condition. The TCT spatial scanning results are shown in Figure 6. The two scans show pixel sensors with $150 \times 100 \mu$m pitch compatible with PSI46dig readout chip, and sensors with $50 \times 50 \mu$m pitch designed for the RD53A ROC. Since the laser does not penetrate metal areas, the lowest amplitude in the scanned results is at the center of the metal. The bias lines and individual pixels are clearly visible which indicates that the TCT scan achieved a satisfying spatial resolution in the preliminary pixel scans.
4. Detector tests with radioactive sources

For evaluating the functionality of our AC-coupled pixel detectors, we followed the testing protocol of the standard CMS pixel detector quality assurance tests. Two fully assembled detectors, one with an implanted bias line and one with a metal bias line, were then exposed to radiation for testing the detector response. We used a measurement setup that is located in the Detector Laboratory at HIP. The calibration isotope Am-241 with its characteristic gamma emissions at 59.5 and 26.4 keV was used. The detectors were each connected to a printed circuit board (PCB) via wire bonds from the contact pads on the ROC. The PCB holding the detector was then connected through an adapter card via a 68 pin ribbon cable to the CMS detector test board (DTB). We used the pXar readout software for quality assurance and data collection [20]. The concept of the CMS based pixel sensor readout and related terminology are described in detail in [21]. The distance between source and sample was 3 cm. During the measurement, a 40 V negative bias was applied to the back plane of the detector for full depletion. Typically, testing of the pixel unit cells (PUCs) requires a signal calibration, mainly for the timing of the trigger. The working point with optimal values are chosen from the center value of the tornado-plot in a VthComp vs CalDel mapping [21]. In pXar and the PSI46dig ROC, units of calibration charges (Vcal) are used, where 1 Vcal corresponds to 50 electrons.

During the spectra measurements, a Vcal trimming value was chosen between 100 to 120 in the pXar software. Although at trimming values of 65 - 80 we started to...
see the detector responding, at higher values noisy pixels are reduced significantly.

In Figure 7 the effect of the trimming values is clearly seen. Figure 7 (a) shows a recorded Am-241 spectrum of the detector with the metal bias line at trimming value of 65 Vcal and Figure 7 (b) at trimming value of 100. It has to be noted that for the AC-coupled pixel detectors higher trimming values are needed compared to standard DC-coupled CMS pixel detectors, that show good results with values around 35 that correspond to 1750 electrons as lower threshold for signal processing of the PUCs [22].

![Figure 7](image)

Figure 7: (a) Pixel detector trimmed to a threshold of 65 Vcal, (b) the same detector trimmed to 100 Vcal.

The reason for that could be the baseline noise of the AC-coupled. In our pixel detector, the AC-coupled pixels act as a high pass RC-filter, hence less noise from the Si bulk leakage current would be expected [23]. However, the noise is affected by the total input capacitance per pixel, which is is dominated by the comparatively high coupling capacitance. Regarding this, it should be noted that the PSI46dig ROC was not developed and optimized for capacitive coupled pixel sensors. We suspect that the observed noise might be caused by the specification limits of the PSI46dig read out chip [24, 25]. This issue will be addressed in further studies.

Interesting is the comparison of both detectors under testing shown Figure 8. On the top, one can see the Am-241 spectrum recorded by the detector with the implanted bias line, and on the bottom by the detector with the metal bias line. The measurement conditions were otherwise equivalent. The spectrum of the detector with the implanted bias line, in figure 8 on top, shows approximately 10% lower position of the 59.5 keV gamma peak in terms of Vcal. Also visible is a lower energy
resolution as the 59.5 and the 26.4 keV peaks are not separable. This may indicate that we have a drain of charge into the bias ring through charge collection to the bias line implant, which leads to lower collected charge as well as poorer energy resolution.

Figure 8: Left: Layouts of different bias line configurations in the mask, right: Am-241 spectra of pixel sensors with different bias line configurations. A shift in the centroid channel of the main peak is visible.

In figure 9 (a) an Am-241 spectrum is shown, recorded with the detector with the implanted bias line. The fitting of the plot uses the crystal ball function for the 59.5 keV gamma emission. On the left side of the photopeak, we observed the trend of a Gaussian function with a polynomial tail. One reason is that we do not have full absorption of the gamma-ray energy in the 320 µm thick sensor. The absorption efficiency of for example a 40 keV photon in Si is less than 5%.
However, for particle tracking applications the performance of our pixel detector in terms of energy resolution and signal-to-noise ratio is sufficient. The spectrum shows a 59.5 keV photopeak and a 26 keV k-line peak. The supplemental spectroscopic result qualitatively validates that our AC-coupled pixel detectors are functional. In Figure 9b the hit map for this measurement is shown. It shows that 99% of the individual pixels are responsive with a uniform behaviour, which is comparable to CMS pixel detectors.

5. Summary

We fabricated AC-coupled pixel sensors on 150 mm diameter MCz silicon wafers at Micronova Nanofabrication center in Finland. ALD grown thin film process was optimized and repeatedly applied in our process [26, 27]. We confirmed that individual pixels are effectively separated using scanning TCT. The TCT measurements showed full depletion at a voltage of 40 V. Therefore we can validate that our tailored material and processes are functional. Also, the fabricated detectors using hybrid technology on the PSI46dig ASIC with flip chip bump bonding technique was established. Fully assembled detectors were tested by measuring spectra of the radioactive isotope Am-241. According to our measurements, we have successfully demonstrated functional AC-coupled n+/p−/p+ pixel detectors with ALD grown thin insulator film.
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