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# Ion beam evaluation of silicon carbide membrane structures intended for particle detectors

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ABSTRACT

Thin ion transmission detectors can be used as a part of a telescope detector for mass and energy identification but also as a pre-cell detector in a microbeam system for studies of biological effects from single ion hits on individual living cells. We investigated a structure of graphene on silicon carbide (SiC) with the purpose to explore a thin transmission detector with a very low noise level and having mechanical strength to act as a vacuum window. In order to reach very deep cavities in the SiC wafers for the preparation of the membrane in the detector, we have studied the Inductive Coupled Plasma technique to etch deep circular cavities in 325 μm prototype samples. By a special high temperature process the outermost layers of the etched SiC wafers were converted into a highly conductive graphitic layer. The produced cavities were characterized by electron microscopy, optical microscopy and proton energy loss measurements. The average membrane thickness was found to be less than 40 μm, however, with a slightly curved profile. Small spots representing much thinner membrane were also observed and might have an origin in crystal defects or impurities. Proton energy loss measurement (also called Scanning Transmission Ion Microscopy, STIM) is a well suited technique for this thickness range. This work presents the first steps of fabricating a membrane structure of SiC and graphene which may be an attractive approach as a detector due to the combined properties of SiC and graphene in a monolithic materials structure.



## 1. Introduction

Semiconductor detectors for ionizing radiation (X and gamma rays, electrons, protons, alpha particles, heavy ions) are extensively employed for spectrometry, dosimetry and imaging in many fields: from fundamental scientific research to medical applications, material analysis, security systems and industrial applications [1]. Both the physical and electrical properties of the material as the specific growing and device manufacturing technology play a fundamental role in determining the final performance of the detector. For example, the high energy resolution and the room temperature operation is possible if the device has a low current under reverse bias. This depends on the bandgap energy, the purity and the defect concentration of the semiconductor and on the quality of the junctions.

In the work presented here, the first steps to fabricate a membrane proton detector in silicon carbide starting from commercially available 4H–SiC substrates is described. As contact material, we apply graphene grown on semi-insulating silicon carbide.

### 1.1. Thin (membrane) detectors in MeV ion systems

Normally semiconductor detectors are designed to have enough thickness that charged particles (MeV) ions are completely stopped within the sensitive volume of the detector (the depletion area where no free charge carriers are present). As an example, 300 μm of silicon is sufficient for 1–3 MeV protons while more energetic ions have a larger range and need a thicker material.

In contrast, thin (membrane) particle detectors are designed to just pick off a part of the kinetic energy from the ion, called a delta $E$ ($\Delta E$) detector. Especially the combination of a thin $\Delta E$ and a thick detector (telescope) allows determination of both the ion energy

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and its mass, and is a standard technique in high energy physics and also in ion beam analysis e.g. Elastic Recoil Detection Analysis (ERDA).

A special application is to use only the $\Delta E$-detector with the purpose of being a particle counter. It is applied in accelerator based systems to study the influence of low-dose particle radiation on living cells. In such a setup, a micrometer sized focused proton (or alpha particle) beam with an intensity of typically 1000 ions/s is aimed at living cells through a very thin vacuum window. The $\Delta E$ detector is then used as a transmission detector to register each single particle before it hits the cell. Combined with a beam deflector it is possible to deliver doses from 1 ion and higher to a specified part of a cell, and very detailed dose response relationships can be obtained. This emerging technique has during the past 15 years brought new insights into cell radiology. In such a setup it is of vital interest to minimize the thickness of the detector membrane as it is a source of ion scattering that makes the focused ion beam less sharp.

Such single ion thin detectors were previously fabricated by etching deep cavities in silicon that leaves a membrane thickness down to 5 μm [2]. The membranes are the active part of the detector where passing MeV ions deposit a small part of their kinetic energy.

Thinner membranes scatter less, however, in addition to technical limitations to fabricate such ultra-thin membranes, less kinetic energy is transferred to the detector and at some point the signal will be lost in detector noise. Cooling improves the situation as was shown for silicon detectors having membranes down to 5 μm [3]. An important contributor to the noise is the temperature dependent leakage current through the biased detector, thus cooling reduces the noise.

As noise is related to the leakage current, a change of detector material can lead to much lower noise. One good material is diamond and several authors report on the production of thin diamond detectors, e.g. [4] starting from single crystal chemical vapor deposited (scCVD) material and creating cavities by $Ar/O_2$ plasma etching. Such devices were evaluated in [5] showing excellent detector capabilities and could in addition to the role of being detector also act as a vacuum window.

Another example is silicon carbide (4H–SiC being the most common) where the leakage current is several decades lower than for silicon due to the difference in bandgap (3.3 vs 1.1 eV). A change from silicon to silicon carbide suggests a reduction in leakage current of at least a factor of 100, possibly 1000 [6]. Like diamond, the physical strength of SiC also allows it to be used as a combined vacuum window and detector.

### 1.2. Graphene covered silicon carbide membrane detectors

We have used the commercially available SiC wafers which have a thickness of 325 μm and currently applied for transistor devices. Ideally a much thinner slab is better suitable for a particle detector but for the first development work we have focused on the commercially available material. It is expected that thinner substrates will be available in the future and can also be specially prepared for detector purposes once the full concept is demonstrated.

In our proposed structure, we will use graphene (a layer of carbon atoms only one or a few atomic layers thick with extremely high electrical conductivity) as a contact material that collects carriers generated by protons at the inner cavity of the membrane, see Fig. 1.

In comparison with scCVD diamond membrane, the graphene based membrane applies a combination of semi-insulating semiconductor (SiC) with a highly conductive well defined layer (graphene). In addition, the graphene is firmly bound to the SiC due to the sublimation process, and not deposited like metals. The materials combination is a monolithic layer that combines advantages of both silicon carbide and graphene. The graphene layer is also quite robust against mechanical damage.

The graphene acts as contact material since the graphene covers the inner cavity all the way to the backside of the SiC substrate. It was shown that graphene on SiC exhibits Schottky properties [7]. The electrons excited by the proton beam could be driven to the graphene layer by an ohmic contact on the front side together with an applied voltage (Fig. 1). This can be used to evaluate the number of protons similarly as for silicon based surface barrier detectors. A different suggestion is to measure the change in electrical conductivity of the graphene, however, this alternative needs to be modeled more in detail before it can be selected. The hardness of SiC is, however, a challenge in the fabrication process to create a thin membrane inside of a deep etched cavity. This also presents a challenge since the graphene is produced on an etched surface. A rough surface leads to an enhanced growth of the number of layers, and potentially less defined thickness and electrical characteristics. The graphene fabrication is also challenged by that the graphene formation differs between the on-axis surfaces of the substrate backside, the wall of cavity, and the bottom of cavity.

The smoothness of the etched membrane is thereby critical for the graphene forming process, and in turn depends on the etching procedure, cavity depth and the quality of the SiC material.

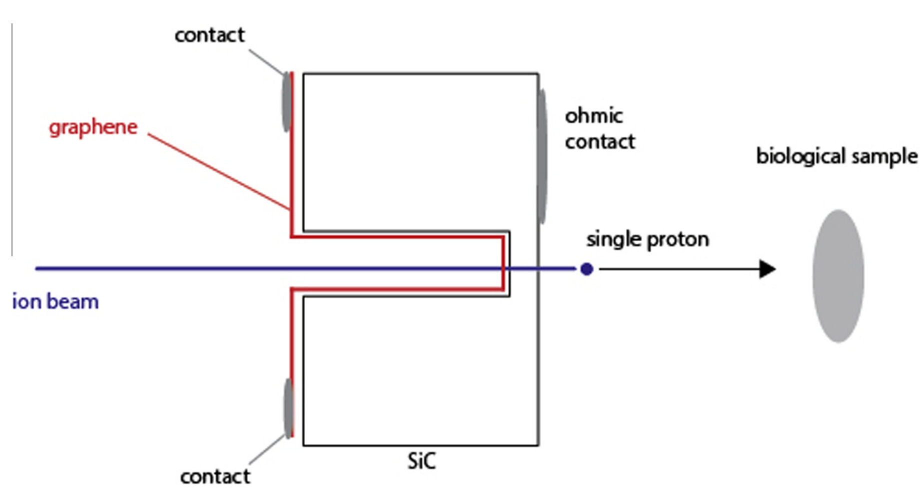


**Fig. 1.** Principal layout of the proposed silicon carbide/graphene membrane detector. The proportions are not to scale and graphene is in reality in direct contact with the SiC. The side with the cavity is referred to as the backside in the text, and the ohmic contact is on the front side.