R&D on CaLIPSO Project in the context of P2IO LabEx.

Project goals

We foresee that these studies will be patented, once the technology will be mastered. Thus, we request that the LabEx P2IO keeps this information confidential.

The CaLIPSO research program funded by the LabEx P2IO focused on achieving a proof of concept of a liquid ionization chamber heavily pixelated, using a liquid the trimethyl bismuth (TMBi). This meant to achieve the detection of free charges through capacitive coupling in an ultraclean environment (ultra-high vacuum technology) free of electronegative contaminants. In addition, the detection medium is chemically aggressive. If the surfaces implemented would be white (perfect light diffusive surface), that would be a bonus.

We explored and optimized the resistive ceramic technology and prepared small test devices in order to demonstrate the technology.
We proposed to use the beam equipment available at CSNSM (SEMIRAMIS) to implant metals at the surface of commercial, but pure alumina ceramics. This material is used industrially to produce electronic circuits and muti-layers assemblies are considered.

The proposed work included:

• Development of resistive ceramic technology.

Criteria for success:

Mastering the process for producing 1-10 GOhms.square resistive ceramics stable over time and versus cleaning processes.

• Prepare a prototype of a single pixel ionization chamber.

Thus prepare a comprehensive, but minimum detector to validate our technologies using resistive ceramic (Charge drift, Frisch grid, signal extraction and readout electronics).

• Multilayer "detectors" electronic-board technology.

This technology must allow the use of resistive ceramic and densely multipixelised detectors.
We foresee to work step by step:
- First, Construction, mechanical and hermiticity tests, chemical stability tests on a prototype bard, called " Zero pads Board."

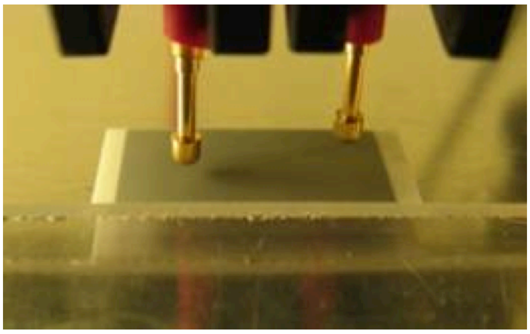
Success Criteria: Hermiticity, Resistance to aggressive chemicals, uniform resistive layer of the final surface.
- Then realization of a low density detection prototype called "sensor pads 256", in order to test readout electronics
- Finally realization of the densely pixelated board, “detector 1600 pads.”

Description of the work achieved

Resistive ceramics:

This is what we spent most of our effort. Our first idea was to achieve a deposition of a thin layer of titanium on the surface of an alumina, followed by implantion of Nickel ions. Nickel ions implanted should allow one to turn the material’s surface resistive. The Titanium layer

maintains the implanted surface to the electrical ground. The surface charges flow and this titanium layer prevent destructive discharge to happen at the ceramic surface.
It appeared that the literature on this topic, used to design this process is simply false: the authors had not noticed that high-dose implantation of metal ions destroyed the surface of the alumina thus consuming materials during implantation. The published values of dose (ions / cm2) were because of this very overestimated.



Surface resistivity measurement of a small ceramic sample.

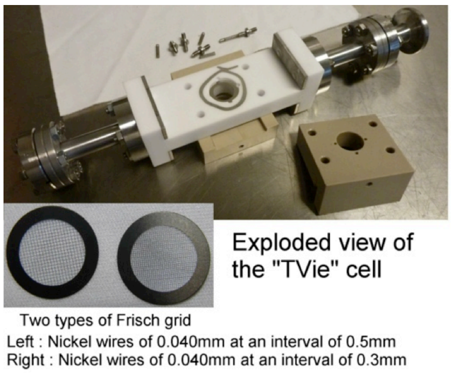
• We observed that titanium deposit, proposed to minimize degradation of ceramics under irradiation fulfills its role very well. But at the highest implantation doses, needed to achieve the desired values of resistivity, ceramics was nevertheless very fragile.
• This titanium deposit proved to participate in a complex manner to the surface conduction of the resistive ceramic. We measured that during Nickel ions implantation, the titanium atoms of the surface layer where also implanted at very shallow depths in the ceramic, dominating the properties of electrical conduction.
• Upon implantation, the nickels ions also eject oxygen atoms from the ceramic, which bind to the titanium layer. The oxides formed make difficult to control the chemical attack, when we wanted to strip the titanium from the ceramic surface after implantation.

These results convinced us that this process would not allows us to make reliable long lasting detectors.

We decided to deposit a very thin layer of noble metal (Ir) <10 nm on the surface of the ceramic. The resistivity was measured : few kOhm.carré. Low intensity and low energy ion beam was then used to insert this metal in the ceramic surface, and obtain the desired resistivity without much degrade the surface of the ceramic. We conducted several sets of samples. We finally prepared few samples with a resistivity of a few GOhm.carré, after cleaning with deionized water, corresponding to our need. Remaining is the study of the technology repeatability, in order to consider a production of the larger surface detector.

Single Pixel Prototype

We designed and developed the technologies needed for the assembly of an ionization single pixel detector model, TMBi compatible. These technologies include hermetic feedthrough, (ultra-high vacuum quality), the realization of Frisch grids of controlled mesh size, mechanical hermetic technologies: collaboration with a company skilled in low-temperature hermetic feedthroughs, used in high-end electronic has not provided conclusive results. We then had to find "mechanical" solutions to these issues.
Then we worked on the IDeF-X ASIC, developed for spacecraft experiments. We showed that, thanks to the flexibility of the ASIC design, this device fulfills our needs for the readout of CaLIPSO charge signals.
All these technologies now seem under control,

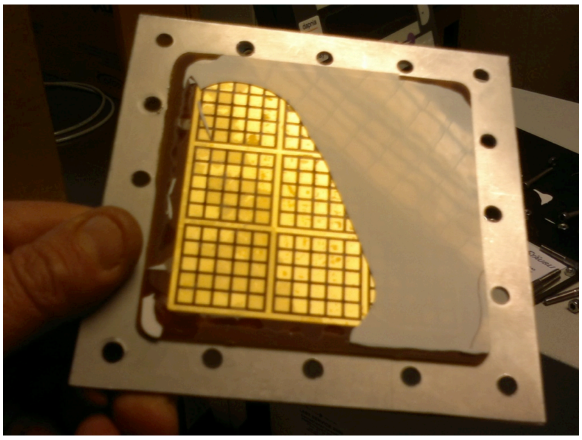


Single pixel detector prototype.

and we wait for the liquid TMBi to be sufficiently purified to test them on the assembled test device.

Multipixel detector board technologies

We looked for subcontractors and ordered a detector board prototype called "sensor pads 0" according to our difficult set of constraints.
We observed that:
• The assembly looks of good quality and allows "routing" of densely pixelleted bord (PCB Class 6).
• Tests boards have withstood the strongest chemical attack (hot concentrated sulfuric acid). A slight infiltration has only been found on one of them. This will be discussed with the manufacturer.
• The mechanical strength test was not conclusive: the polymer selected to carry out the assembly (polyimide) proved to be too "soft". Deformations induced by pressure on the detector when pumping on a close volume stressed the thin ceramic layer and caused it to break.



Premier prototype de circuit - détecteur multicouche après le test de résistance mécanique et de tenue au vide. Un second prototype sera nécessaire.

Technological progress on the single pixel prototype allowed us to relax constraints on our specifications. So we decided to change the hardware implementation of the routing circuits circuit in the board. A second prototype will be necessary to check these options. It is likely that we'll make the second test board directly into the 256 pads configuration.

Publications

Since we foresee to patent some of these technologies, we did not published any of the above results.

Expenses

The allocated funds (40 k€, 40% of the requested amount) did not allow us to the build the charge detector. We focused on the implementation and optimization a resistive ceramic technology needed to prepare the detector. (Our group is still looking for the complementary funds).
• The realization of samples "test" resistive ceramic surface. This includes the purchase of ceramic samples, UHV samples cleaning procedure before and after implantation, the evaporation of thin film materials, the implantation of ceramic samples to adjust their surface resistivity, chemical attacks. Over fifty wafers were prepared.
• Tests and measures related to the resistive ceramics: a dedicated test bench was mounted at the IRFU to measure and monitor the samples stability. We used surface analysis techniques available at CSNSM (Scanning Electron Microscope, Fluorimetry, Rutherford backscattering), in order to understand our sample behaviors.
• Subcontracting Multilayer detectors boards.
• Single-pixel prototype technologies, hermetic feedthroughs.

• The beam tools needed to implement the full surface of the detector is evaluated by the CSNSM to cost 12 k€. This item was deferred until we have settled the preliminary technologies.

Possible valorization of the project

This work is a part of a larger project, whose goal is to build a new positron annihilation detector. The proposed CaLIPSO detector should allow to assemble high resolution, efficient PET scan apparatus, optimized for the imaging of brain (or multi-rodent) biochemical activity. This apparatus would facilitate research on neurodegenerative diseases, as well as tests on drugs for cancer therapies.

Future of the project after P2IO funding.

We will go on our fund raising work (23 applications up to now), in parallel with our lab work, in order to build the first full-features CaLIPSO detector.

Relevance of the project within P2IO and specific added value for P2IO

This work was designed and was run in close collaboration between three P2IO labs, CEA-IRFU, CNRS-CSNSM and CNRS-IPNO. The goal is one of the topic priorities of P2IO: detector development for health sciences. P2IO funding allowed us to initiate this work and was decisive in bringing the CNRS-CSNSM lab in the CaLIPSO collaboration and in the Health Science business.
This work has spent more than the budget allocated by the P2IO R&D program. It did allowed us to demonstrate two of the key technologies needed for the realization of the CaLIPSO charge demonstrator.

Sincerely
The CaLIPSO team.