Applied Diamond, Inc.

OVERVIEW

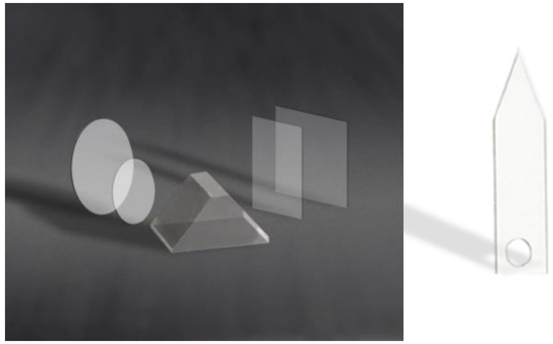
About Us

Incorporated in 1986

- Headquartered in Wilmington, DE USA
- DDK founded in 1986 by Ray Tabeling.
  - Originally spin-off from DuPont
  - 2001 – First CVD reactors, established Applied Diamond, Inc
  - 2022 – DDK and Applied Diamond, Inc merged into single commercial entity, Applied Diamond, Inc
- Fabricate and sell diamond parts/assemblies/devices for a variety of uses
- Primarily self funded, small percentage from SBIR programs.

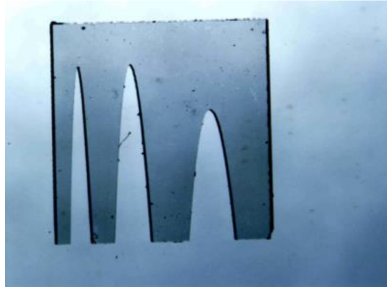
Diamond window

Manufacturing Capabilities

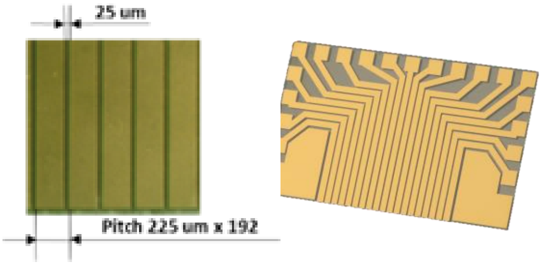


Diamond processing

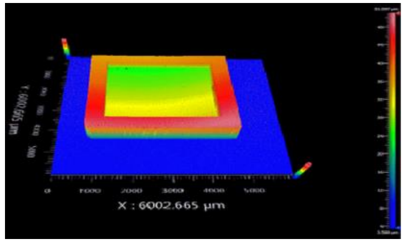
- Laser cutting (5 µm stepping resolution) – custom sizes/shapes
- Grinding, polishing and Super Polishing (< 1 nm rms)
- Dry etching (ICP-RIE) – for producing diamond topography patterns and ultra-thin parts (down to 5 µm)
- Metallization – for electrical contacts/heat spreaders
- Bonding – vacuum brazing assemblies and compression bonding
- Fiber optic diamond cleaving tools
- Tungsten carbide and sapphire knives for light microscopy



Metallization

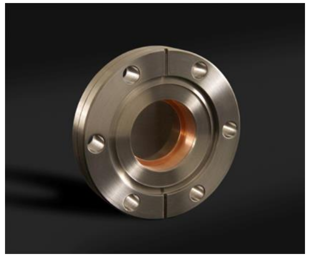


Dry Etching (ICP-RIE)



25 µm thick diamond membrane

Brazing



∅ 25 mm diamond window

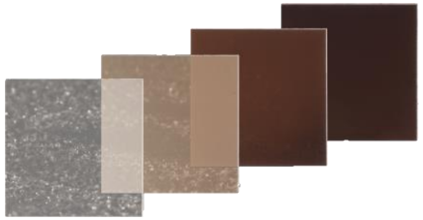
Manufacturing Capabilities

CVD Diamond growth

- Multiple microwave plasma CVD reactors for diamond growth (ASTeX and home-made systems, 5-10 kW)
- Polycrystalline and single crystal diamond (PCD and SCD)
- Tooling, optical and electronic grade CVD diamond
- Isotopically pure diamond (C-12, C-13)
- Thin films and ultra-thin membranes (down to 50 nm thick)
- Doped diamond films (boron, nitrogen)

ASTeX PDS-17 microwave plasma enhanced chemical vapor deposition system

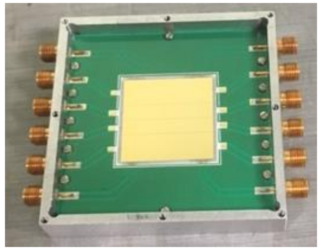
B- and N-Doped Diamond

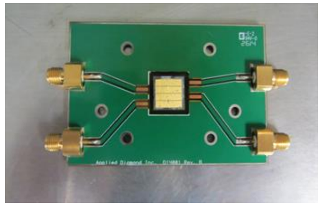


Manufacturing Capabilities

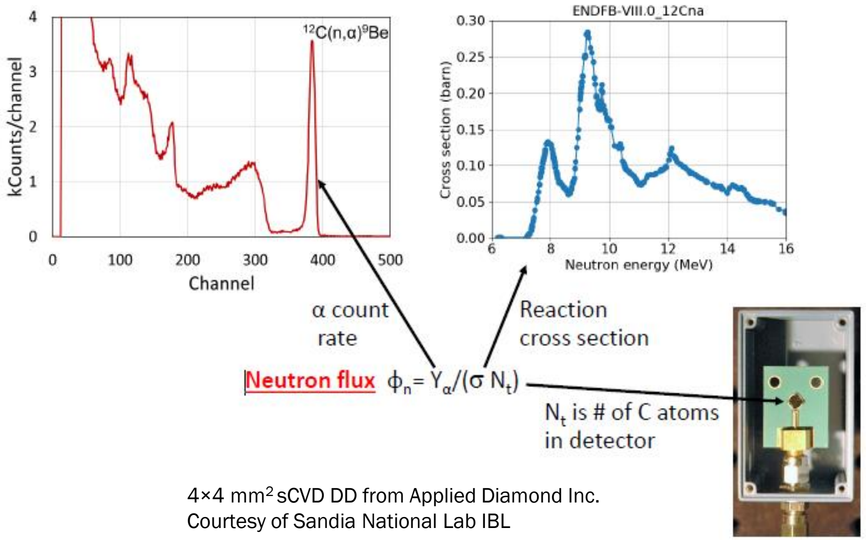
Diamond radiation detectors

- High quality electronic grade (< 1 ppb nitrogen) single crystal diamond (sizes up to 4.5×4.5 mm^2) and large size polycrystalline diamond (sizes up to 2.5”).
- Standard thickness of 50, 100, 250 and 500 µm. Custom sizes and thicknesses are possible.
- The diamond detectors are available in a range of packages, using either FR4 or ceramic PCB.





Spectroscopy of 14 MeV neutron source



α count rate

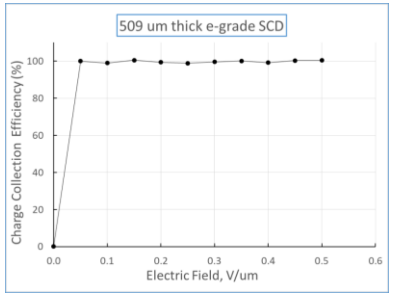
Reaction cross section

Neutron flux $\phi_n = Y_\alpha/(\sigma N_t)$

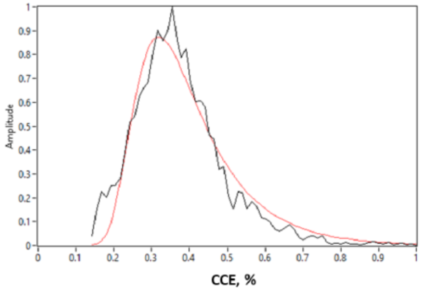
N_t is # of C atoms in detector

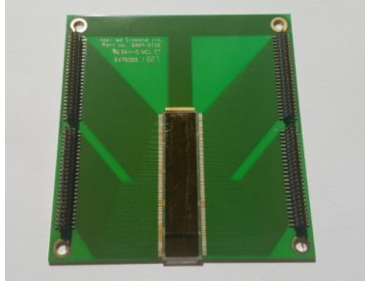
4×4 mm^2 sCVD DD from Applied Diamond Inc.
Courtesy of Sandia National Lab IBL

100% Charge Collection in SCD



Improved Charge Collection of PCD





DOE SBIR Phase II Project

Fast, Large-Area Detector for Position and Energy Determination

Grant Supported by Department of Energy, Office of Nuclear Physics
Nuclear Physics Instrumentation, Detection Systems and Techniques
Topic No. DOE 2021-38b
Grant: DE-SC00201452
Award Date: 02/03/2020

Principal Investigator: Valeriy Konovalov, Ph.D.

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Introduction

- Radiation detectors used in experiments for energy, timing and position measurements of heavy ion beams should be fast and very radiation resistant.
- However, heavy ion beams pose challenges for today’s common radiation detectors, like scintillators and Si-detectors, which are sensitive to radiation damage by heavy ions. The experiments with much higher intensity ion beams such as at the FRIB facilities at Michigan State University will require even more radiation-tolerant detectors than currently exist.
- Diamond detectors have an excellent radiation tolerance and have been found to withstand irradiation doses many times exceeding the Si-detectors. Diamond detectors are inherently faster than Si-detectors, can produce high quality spectra, and can operate at high temperature without cooling.
- One of the main problems preventing the wide application of diamond detectors is a rather small size of available electronic grade single crystal diamond (SCD), up to 4.5 mm. Available large area polycrystalline diamond (PCD) detectors are not suitable for energy spectroscopy.
- Our long-term objective for this project is to develop and make large area mosaic single crystal diamond (SCD) detectors allowing simultaneous position and energy determination and having a fast time response.

Technical Objectives

Two major approaches:

1) Use the highest quality small e-grade SCD commercial plates to tile them together into a large area mosaic SCD plate using foreign bonding materials.
2) Make “All-Diamond” mosaic SCD. Overgrow CVD diamond between the adjacent small lower quality SCD plates to make a single large SCD overgrown layer. Use it as a substrate to grow an e-grade SCD layer on its top. Laser cut, polish and use a new e-grade large SCD plate. Reuse the remaining SCD substrate to make new e-grade large SCD plates.

1st Approach

- **Develop mosaic bonding methods**
    a) Epoxy – use a suitable epoxy to “glue” two plates together.
    b) Compression Bond – use a thin gold layer on the diamonds’ edges and, with pressure, temperature and time, form a bond between the surfaces.
    c) Brazing – form a thin metal joint using a reactive copper/silver braze.
- **Reduce the width of “dead zones” between adjacent SCD tiles**

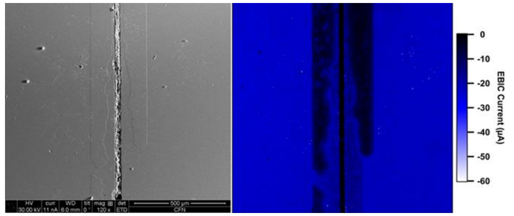
2nd Approach

- **Develop CVD diamond overgrowth technique to form large mosaic wafer**
- **Develop the CVD growth process for high quality e-grade SCD.**
- **Grow and test a e-grade large SCD on mosaic SCD substrate**
- **Construct a new CVD reactor dedicated to the growth of e-grade SCD**

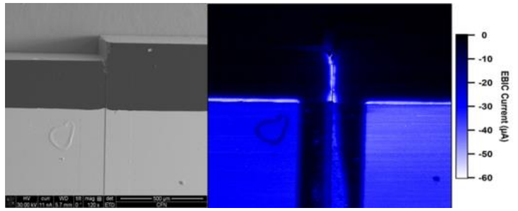
Development of mosaic bonding methods

1st Approach (use foreign bonding materials)

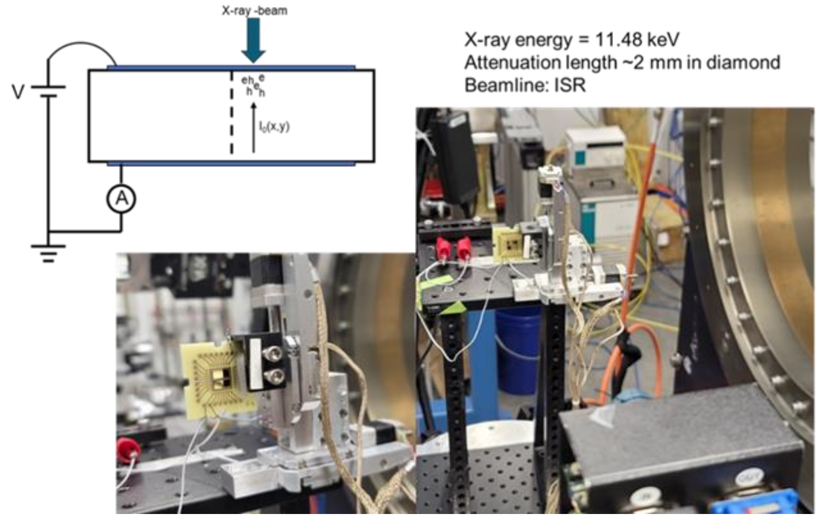
- A variety of methods were attempted to create a robust butt joint between two SCD plates
  a) Epoxy – two plates were side polished, bonded by NASA approved epoxy, and tested, gap ~ 50um.
  b) Compression Bond – plates were side polished, Au/Cr was sputtered on sides, compressed and vacuum heated. Gap ~50 µm, plates shifted against each other
  c) Brazing – plates were side polished, vacuum brazed, gap ~50 µm, uncontrolled brazing alloy spreading to the plates. Needed repolishing.
  d) Soldering – plates were metallized and back-side soldered on PCB. Gap ~100 µm. Good for thin diamond (≤ 50 µm, low bias voltages).
- Bonded plates were metalized and tested at BNL (EBIC- electron beam induced current and XBIC- X-ray induced current). The real “dead zones” at the junction were measured to be up to 200 µm.



SEM and EBIC images of the **epoxy** bonded sample at 50V of bias.

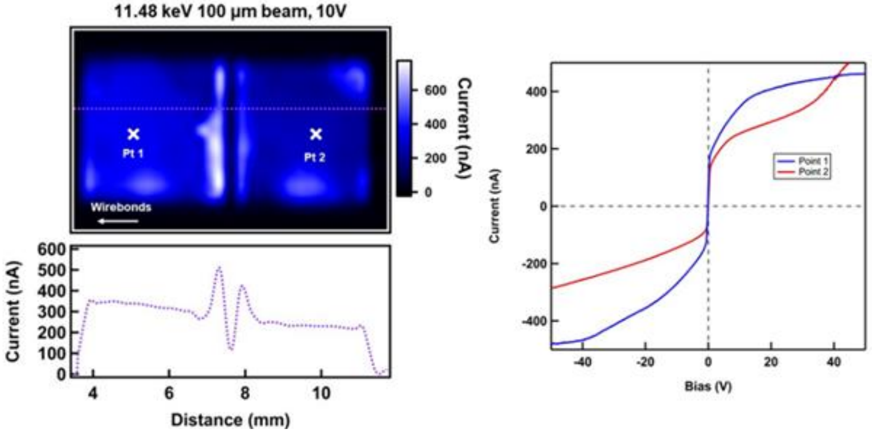


SEM and EBIC images of the **compression** bonded sample at 50V of bias.

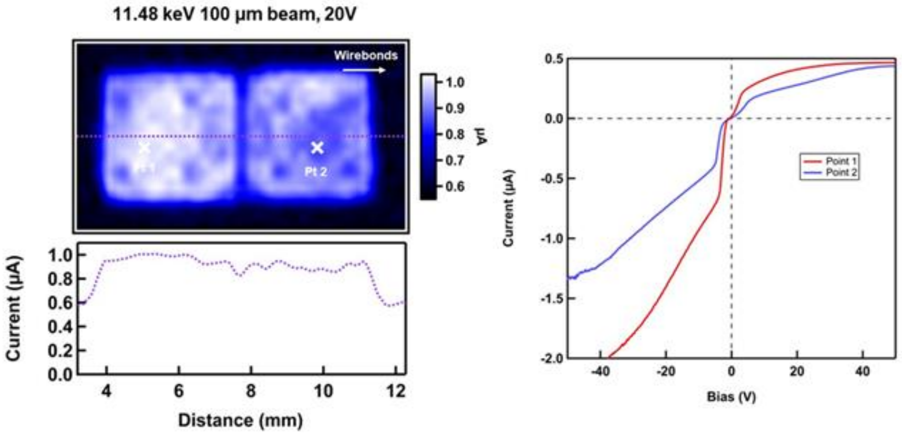


X-ray energy = 11.48 keV
Attenuation length ~2 mm in diamond
Beamline: ISR

Setup for XBIC at ISR.



XBIC on Compression-bonded Sample

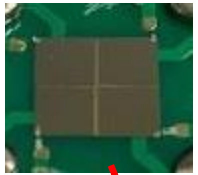


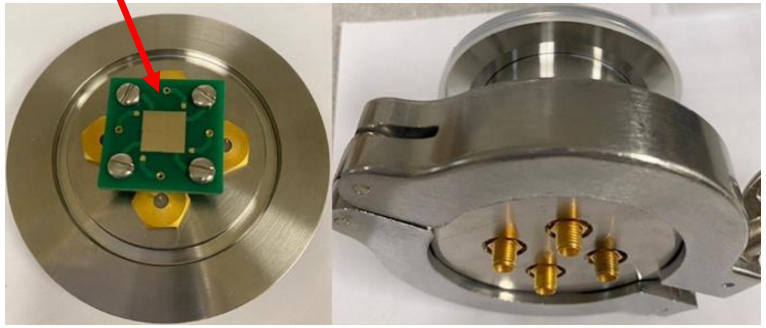
XBIC on Epoxy-bonded Sample

Large area mosaic SCD detector prototype

Mosaic diamond sensor (configured for alpha spectroscopy):

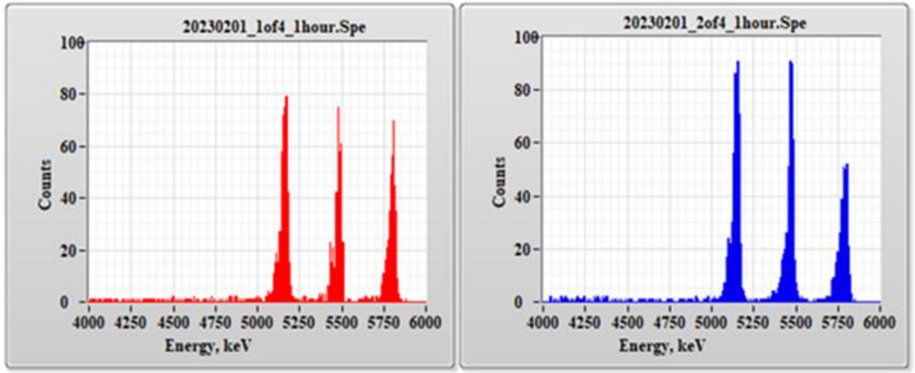
- 2 x 2 = 4 tiles, 9 x 9 mm total area, 52 ± 1 µm thick, 100 µm “dead area” between tiles
- Vacuum flange assembly
- Independent signal feedthrough (SMA) for each sensor
- Spectral resolution – 50 keV across the area





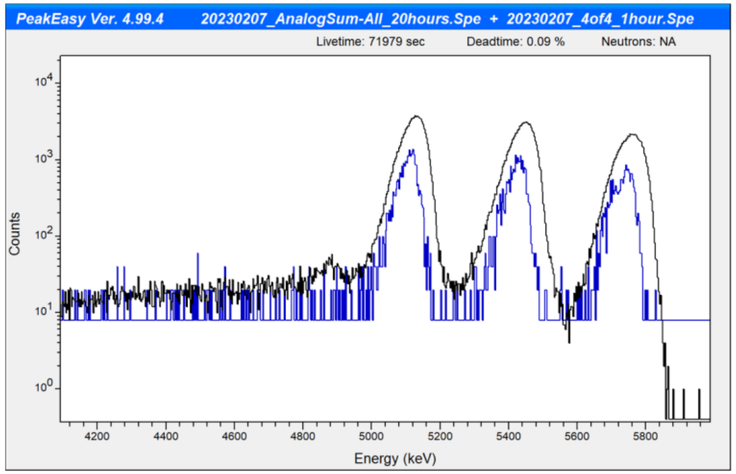
- Based on the developed large area mosaic SCD detector we designed a detector prototype configured for low energy particle spectroscopy (e.g. - 2-7 MeV alpha particles).
- Testing demonstrated an excellent energy resolution of 50 keV. This is a product which is ready for market. Potential market for that product is rather broad. It can be used for any type of high energy radiation measurements where a large area is needed (e.g. large beam sizes) and a good spectral resolution is required. In addition to be used as a fast detector for position and spectral measurements with high energy beams it can be used, particularly, for alpha spectroscopy.

Multi-alpha check spectra from different tiles



Tile - 1

Tile - 2



Spectra from #4 tile (blue) and the analog sum of all 4 tiles (black)

Direct Alpha Spectrometry of Nuclear Fallout

David Chichester - team leader, INL

- Development of instruments and methods for fieldable alpha spectrometry that are faster and easier than lab-based work is necessary. Fieldable methods for nuclear fallout pose new challenges.
- The beta dose rate from fresh nuclear explosive debris is extremely high, **>10^5 times** the alpha rate. High beta rates challenge traditional Si detectors. Operating in a very high beta radiation field, standard COTS Si-based spectrometers are overwhelmed with pile-up.
- High-speed, large area SCD radiation detectors provide a solution. SCD is inherently faster than Si and can produce higher-quality alpha spectra, even using COTS data acquisition electronics. SCD shows much less pile up.
- A new FASTPAC spectrometer was developed based on the large area SCD detector developed by Applied Diamond during the Phase II. Realistic NED materials have been tested with new spectrometer. Good market potential for the large area SCD detector have been demonstrated.

This work is a part of the Rapid Response Research Post-Detonation Forensics Venture and is supported by the National Nuclear Security Administration's Office of Defense Nuclear Nonproliferation Research and Development.

INL CON 23 71391
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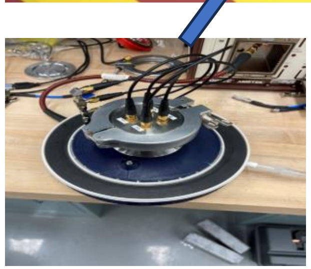
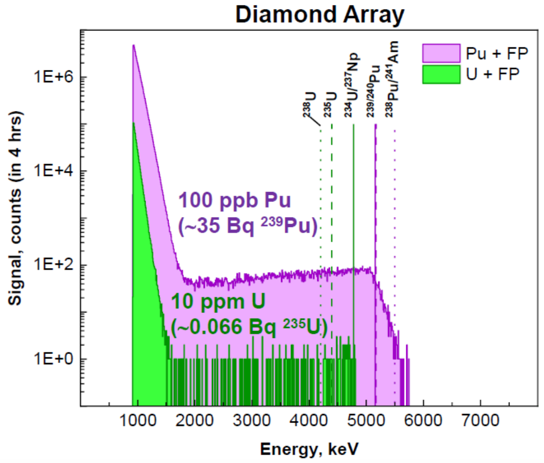
Idaho National Laboratory

Improved FASTPAC System

In this implementation the FASTPAC array is in an external Bell jar for vacuum; the signal is sent through the electronic chain of a COTS system which also held a Si diode spectrometer



Testing of short-lived fission products



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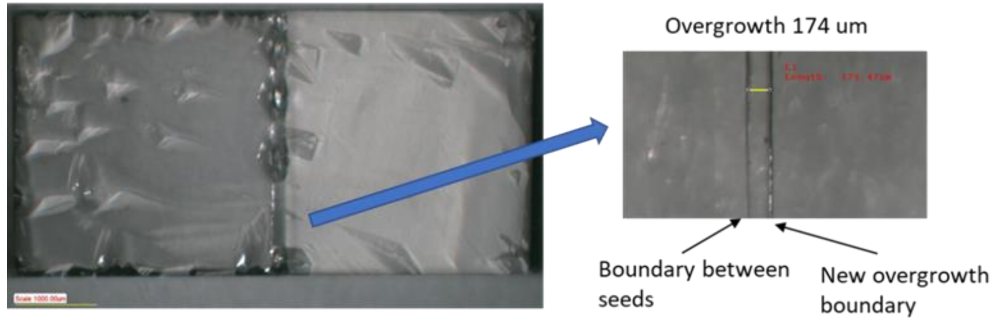
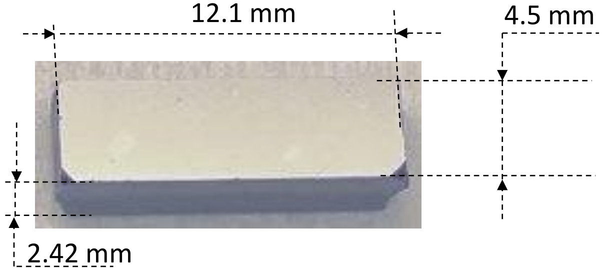
Fabrication of Large Area "All-Diamond" Mosaic SCD

- Two lower quality SCD plates were placed into the CVD reactor next to each other. CVD diamond layer was successfully overgrown from one plate to another completely closing the gap.
- Sample holder was improved to provide better growth quality
- CVD process (temperature, pressure, methane %) was adjusted to provide better growth quality reducing the growth of non-epitaxial defects and cracks.
- A single SCD layer was grown over the two 6×6 mm SCD HPHT plates forming a 4.5×12.1 mm substrate used to grow ~ 270 µm tick e-grade SCD layer on the top.
- Other e-grade 2×1 mosaic SCD substrates are in the production.
- **Main Problems**:
  - Non-epitaxial diamond (PCD) growth – Hillocks (from twins, dislocations). Hillocks were significantly reduced using the 3-5° off-cut (100) seeds. High quality SCD seeds (very expensive!) will be finally used.
  - Cracks. Caused by stress, likely from PCD hillocks and sudden reactor stops. More CVD growth process adjustment and seed preparation is necessary.



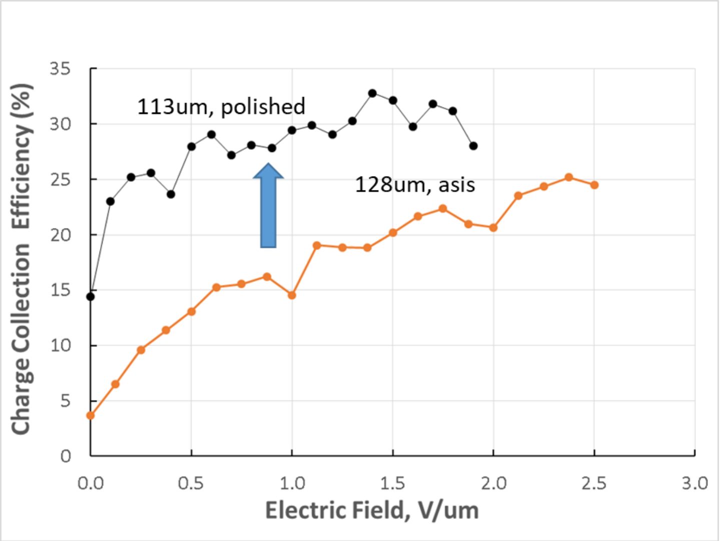
Two SCD seeds bonded by a single overgrown diamond layer. The overgrowth from the initial boundary between seeds and final boundary on the grown layer is about 174 um.



Development of electronic-grade SCD

- Previously, Applied Diamond successfully produced a good quality e-grade PCD but didn’t succeed in the growth of good quality e-grade SCD. Main problem was the growth of PCD defects on SCD. SCD and PCD – two different CVD processes.
- CVD process for the growth of e-grade SCD was improved. Old tube reactor similar to a new constructed reactor was used.
- Charge collection efficiency (CCE) for e-grade SCD was improved from initial 2-3% to about 35%. Post-treatment of SCD (polishing, etc.) demonstrated the positive effect.

<u>Conclusion</u>: Need further process development to reach 100% CCE.

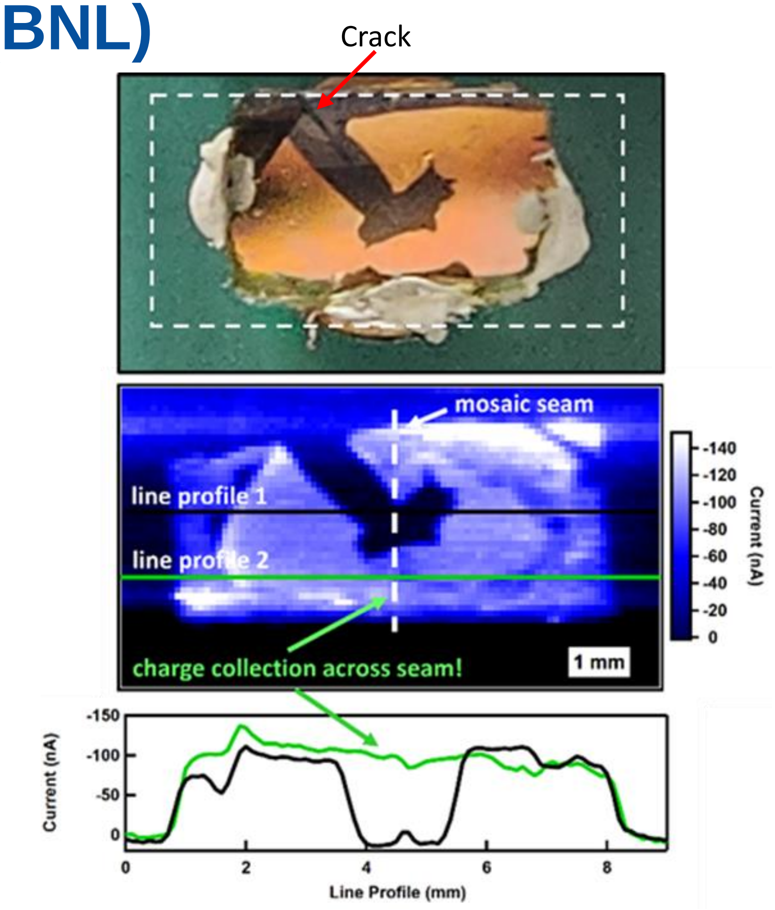


Electronic grade SCD grown on low quality cheap optical grade 3x3x0.3mm mm SCD seed. Additional polishing of the substrate side improved the CCE.

Testing the large size e-grade SCD detector at XBIC (BNL)

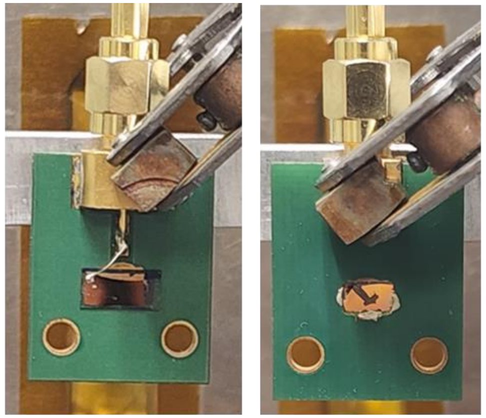
- A large electronic grade SCD layer was laser cut-off and polished, finally making a 4.3 mm×8.8 mm × 250 µm SCD plate. Unfortunately, a small crack in the diamond developed during the final polishing of its second side so the metal was patterned to avoid overlap with this crack. Diamond was mounted on PCB.
- The detector was installed on a XY-motor stage in the XFP beamline at NSLS-II for XBIC measurements (broad spectrum, 4.5÷16 keV, up to 1.6 x 10^{16} ph/s flux.
- In the area where the metallization is present the **response is uniform**. Thus, the charge collection across the junction area between two initial small diamond plates is continuous and no drop is observed.

This is the critical result for that project, demonstrating the feasibility of our approach to create large size mosaic SCD with continuous response across the whole area.

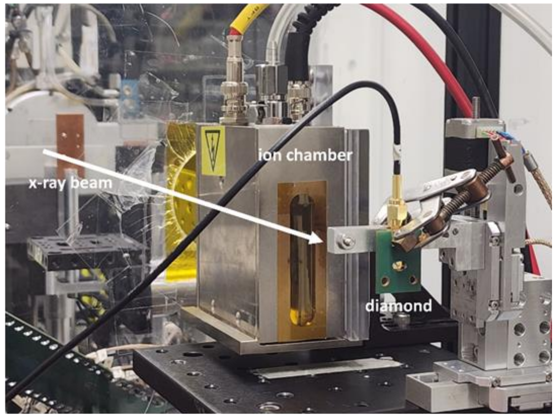


Picture of the detector from the beam exit side of the PCB.

- Top – optical image
- Middle - XBIC image showing the response of the diamond with bias applied (leakage current subtracted).
- Bottom - Line profiles showing that there is charge collection across the mosaic seam where the metallization is continuous.



Front and back of the mounted diamond detector

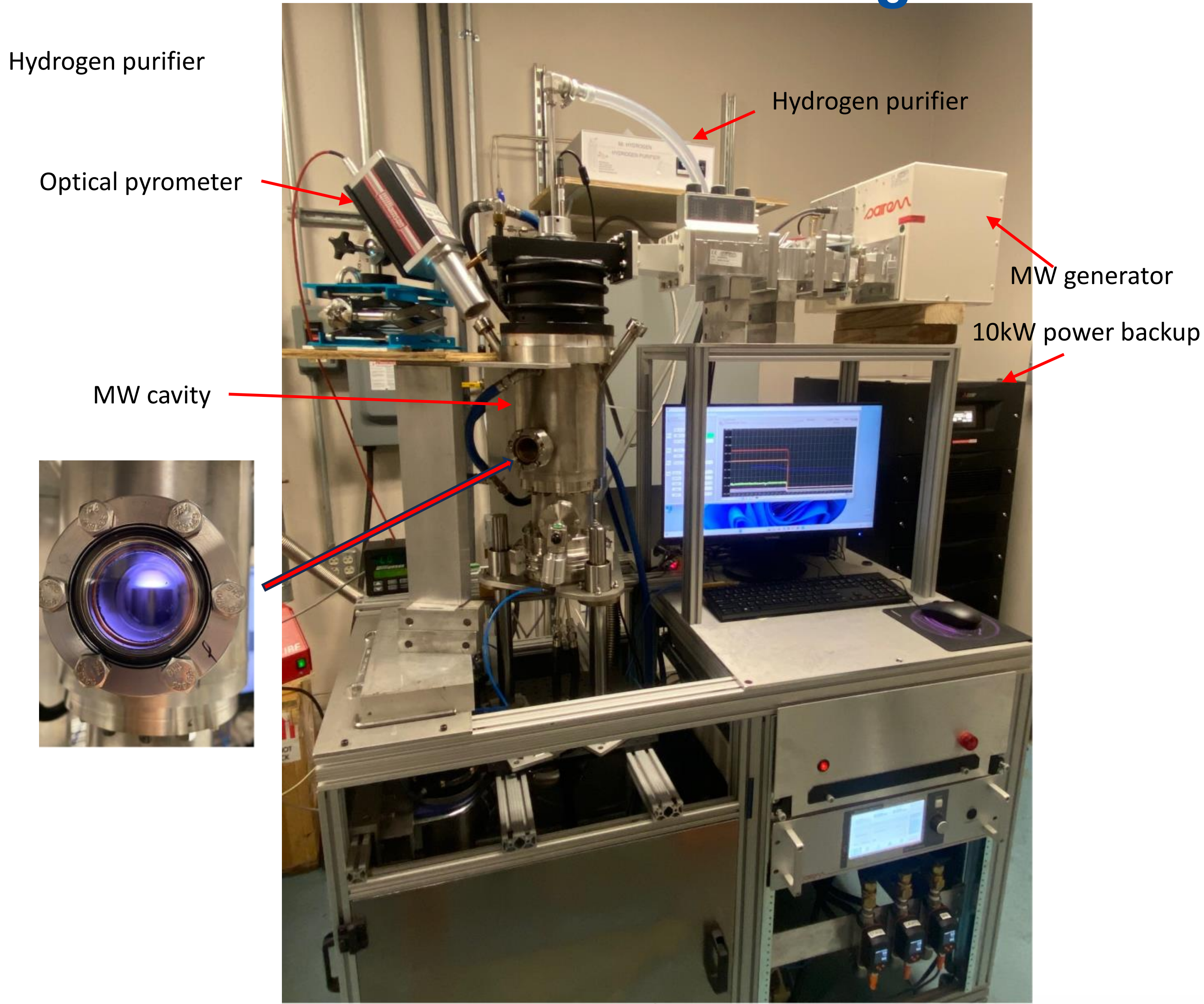


Beamline setup at XFP

Construction of dedicated CVD reactor for electronic SCD growth

New reactor main features:

- **Full power backup (10 kW) for preventing sudden short power interruptions (~90% of all stops, very common in our area)**
- **Robust UHV system minimizing air leaks to < 1ppb level**
- **Improved MW cavity geometry, designed using numerical simulations.**



Conclusions

- **Developed "primitive" mosaic bonding of small commercial electronic grade SCD plates by foreign material is suitable for fabrication of detectors where the 100-200 µm width of "dead zones" isn't important, e.g. - just the large area or 0.5 - 1 mm spatial resolution are required.**
- **Developed prototype of mosaic SCD detector (9×9 mm total area, 52±1 µm thick) demonstrated an excellent spectral resolution of 50 keV with alpha particles, which is similar to Si-detector. There is a current commercial interest in that product.**
- **The total area can be scaled to much large sizes (e.g. 36×36 mm, etc.).**
- **Developed "all-diamond" bonding of small SCD plates to make a large area mosaic SCD with continuous detector response across the whole area.**
- **Constructed a new MW CVD reactor, tailored for e-grade SCD growth (no short power failure stops, <1 ppb leaks).**
- **Remaining main problems:**
  - ✓ Improve the quality of the CVD diamond overgrowth process, eliminating growth defects and reducing stress/strain in diamond leading to cracks.
  - ✓ Improve the quality of electronic grade SCD material to reach 100% CCE.