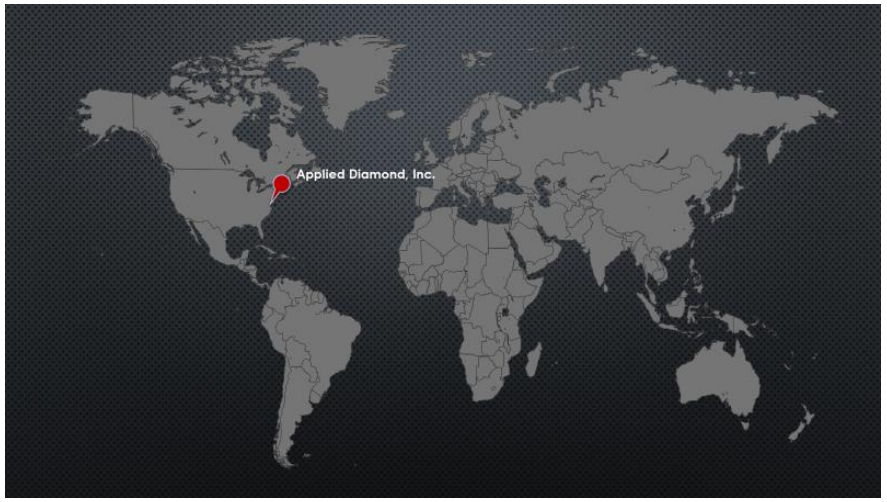
Applied Diamond, Inc.

OVERVIEW

Applied Diamond, Inc
3825 Lancaster Pike, Wilmington, DE 19805, Phone: (302) 999-1132, FAX: (303) 999-8320
www.usappliedddiamond.com, Email: services@usappliedddiamond.com

About Us



Incorporated in 1986

- Headquartered in Wilmington, DE USA
- DDK founded in 1986 by Ray Tabeling.
  - Originally Spinoff 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

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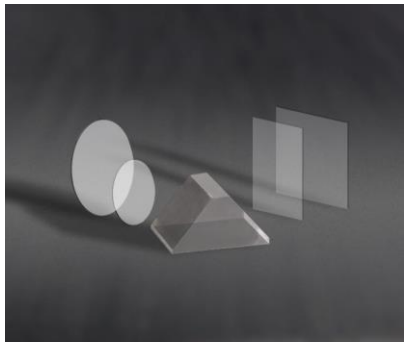
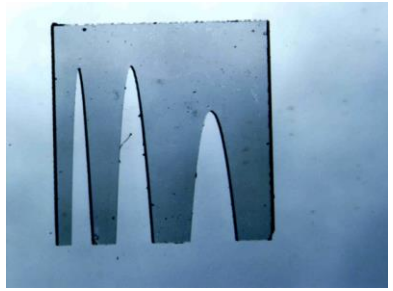
Manufacturing Capabilities

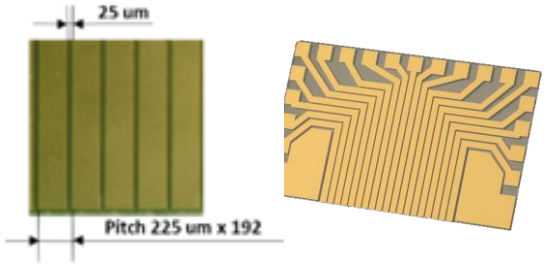


Diamond processing

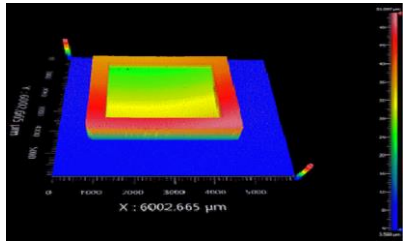
- Laser cutting (5 µm stepping resolution) – custom sizes/shapes
- Grinding, polishing and Super Polishing (< 1 nm rms)
- Dry etching (ICP-RIE) – for producing diamond topography patterns and ultra-thin parts
- Metallization – for electrical contacts/heat spreaders
- Bonding – vacuum brazing assemblies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



Metallization

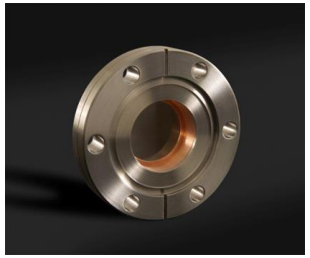


Dry Etching (ICP-RIE)



25 µm thick diamond membrane

Brazing



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Manufacturing Capabilities

CVD Diamond growth

- Multiple microwave plasma CVD reactors for diamond growth (ASTeX and home-made systems, 5-15 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

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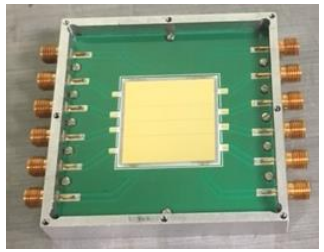
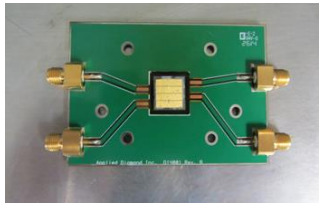
Manufacturing Capabilities

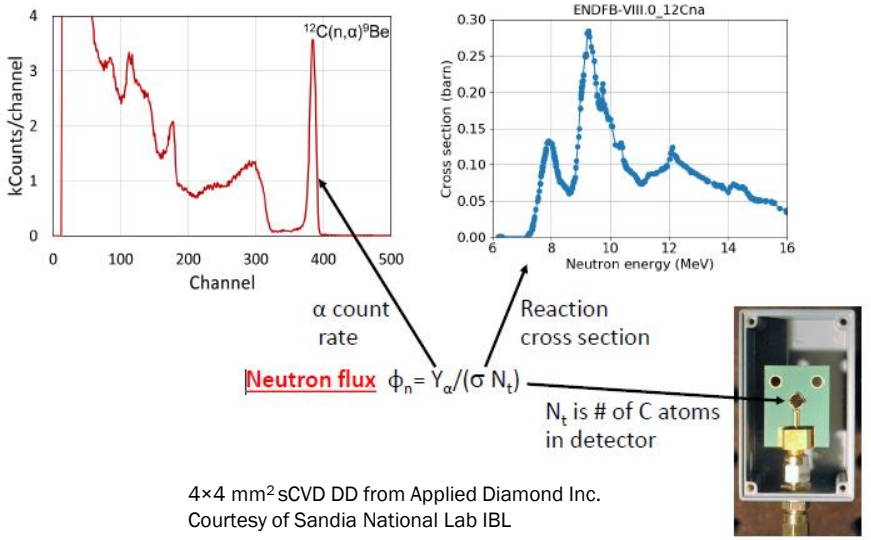
Diamond radiation detectors

- 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.



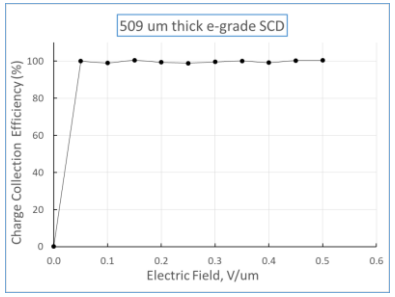


Spectroscopy of 14 MeV neutron source

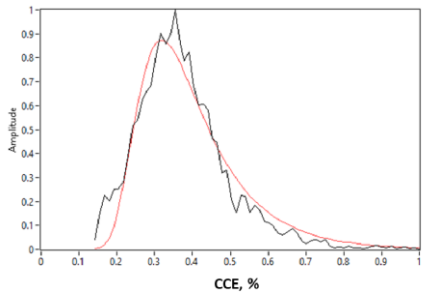


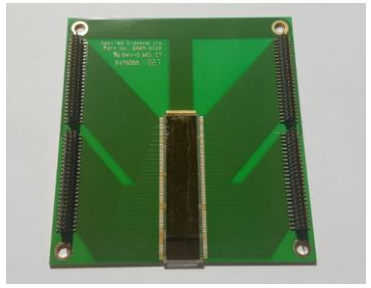
4×4 mm^2 sCVD DD from Applied Diamond Inc.
Courtesy of Sandia National Lab IBL

100% Charge Collection in SCD



Improved Charge Collection of PCD





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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.**

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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.
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**

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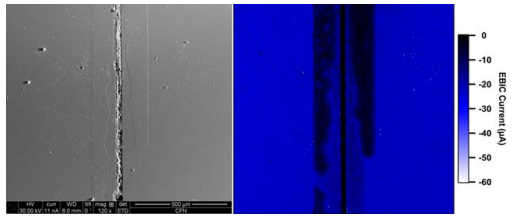
Development of mosaic bonding methods

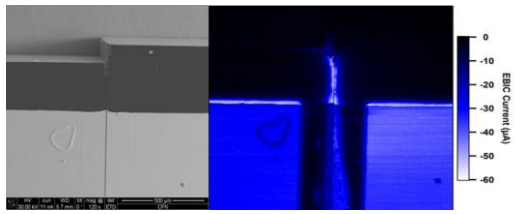
1st Approach (use foreign bonding materials)

- 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 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.
  c) Brazing – plates were side polished, vacuum brazed, gap ~50 µm, uncontrolled brazing alloy spreading to the plates.
  d) Soldering – plates were metallized and back-side soldered on PCB. Gap ~100 µm.

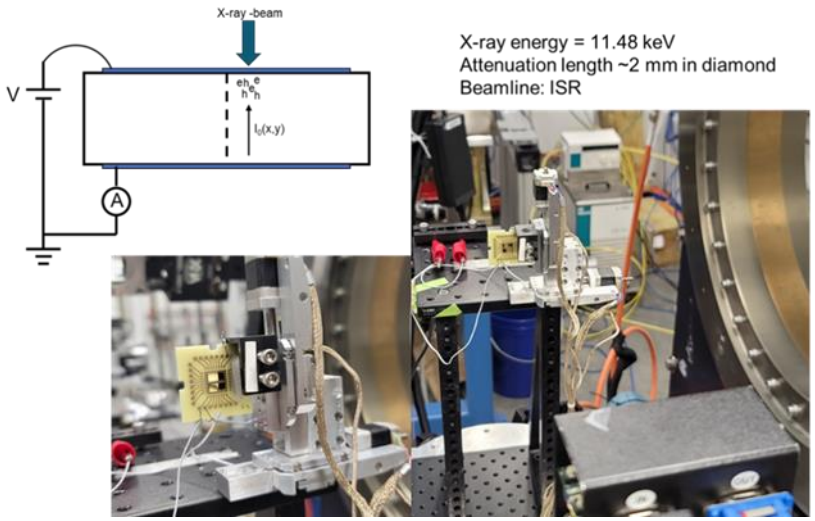
  Bonded plates were metalized and tested at BNL (EBIC- electron beam induced current and XBIC- X-ray induced current). The real "dead zones" at the junction were measured to be up to 200 µm.



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

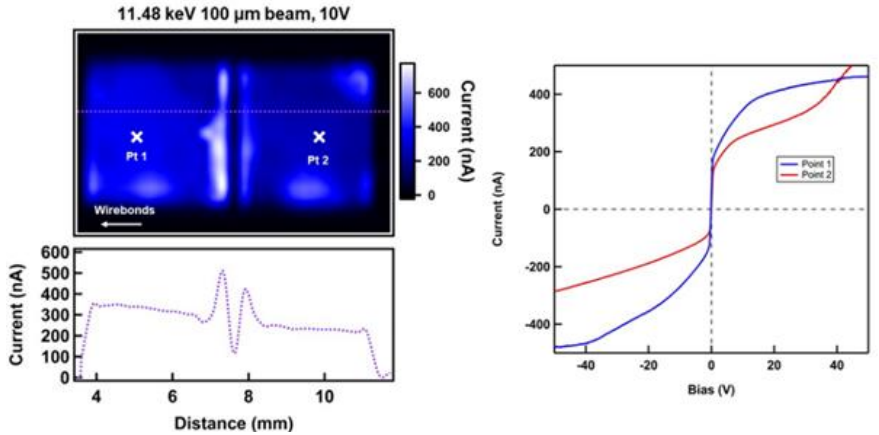


SEM and EBIC images of the **compression** bonded sample at 50V of bias.

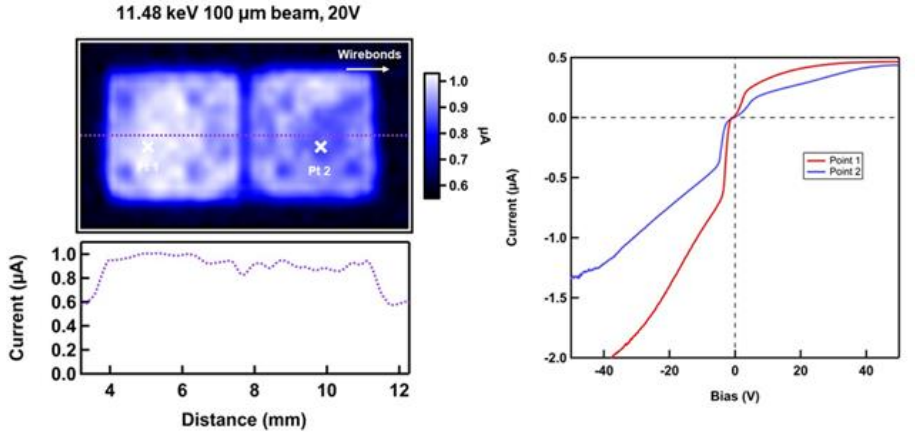


Setup for XBIC at ISR.

XBIC on Compression-bonded Sample



XBIC on Epoxy-bonded Sample



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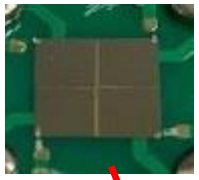
Large area mosaic SCD detector prototype

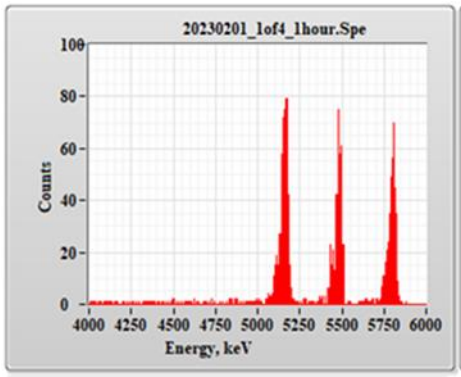
Mosaic diamond sensor (configured for alpha spectroscopy):

- 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

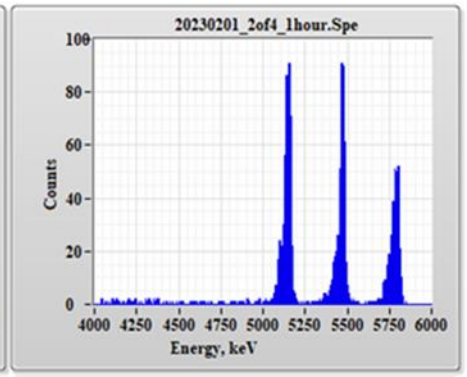


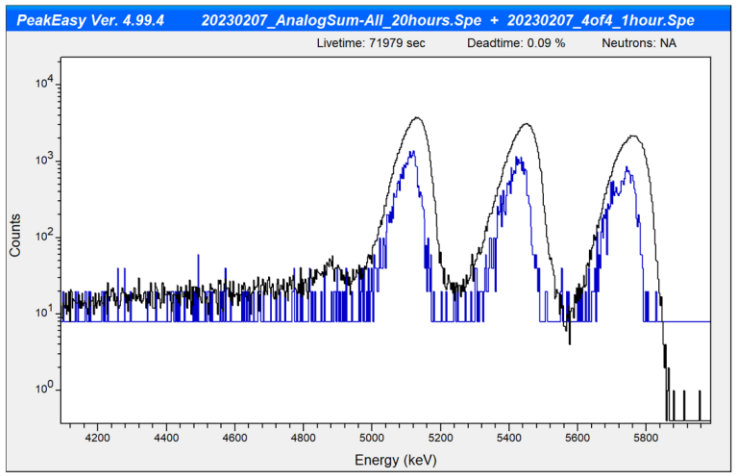
Multi-alpha check spectra from different tiles

Tile - 1



Tile - 2





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

- 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. 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.

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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.

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.

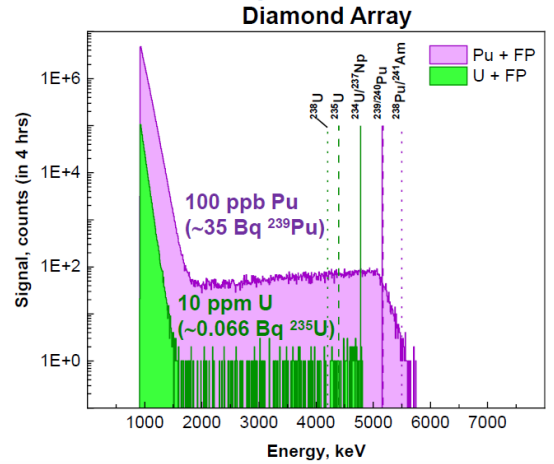
INL CON 23 71391
Approved for public release; distribution is unlimited.

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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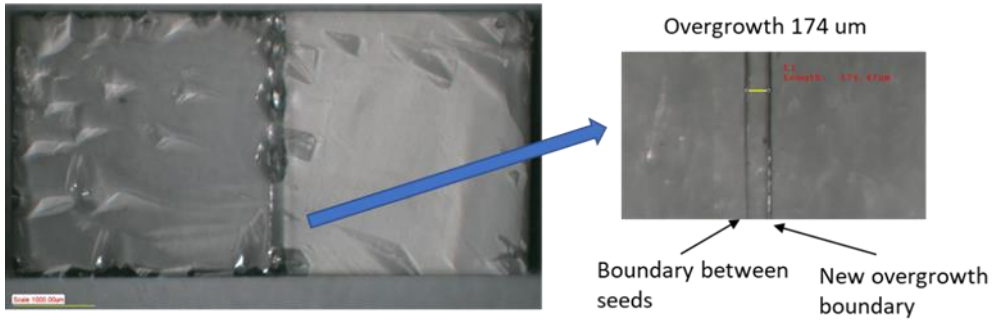
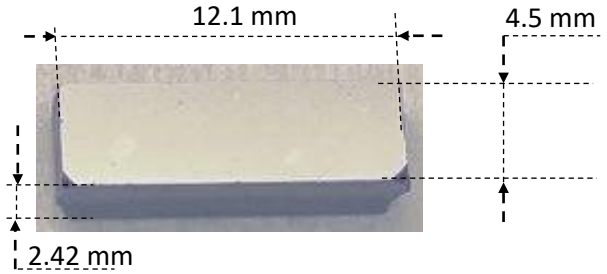
Fabrication of Large Area “All-Diamond” Mosaic SCD

- 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.
- CVD process (temperature, pressure, methane %) was adjusted to provide better growth quality.
- A single SCD layer was grown over the 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



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.
- 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 to 32%.

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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.).**
- **“All-diamond” bonding approach can potentially provide a single piece of large area electronic grade SCD. The advantage – it will be grown at Applied Diamond (in US not in UK). The progress here includes:**
  - Successful growth of a single 4.5×12 mm piece of e-grade SCD from lower grade SCD seeds. BNL testing is pending.
  - The growth of electronic grade SCD was significantly improved. Charge collection efficiency of 32% was achieved with old type CVD reactor.
  - Construction of new CVD reactor, tailored for e-grade SCD growth was completed. Currently the reactor is in the installation/testing mode.

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