ess

EUROPEAN
SPALLATION
SOURCE

ESS Detector Coatings Workshop
Linköping Sweden

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2020-02-13

Agenda

1 Background: ESS and B_4C

2 Detector Coatings Workshop in Linköping

3 Production Development

4 Research Development

5 DLC Related Works

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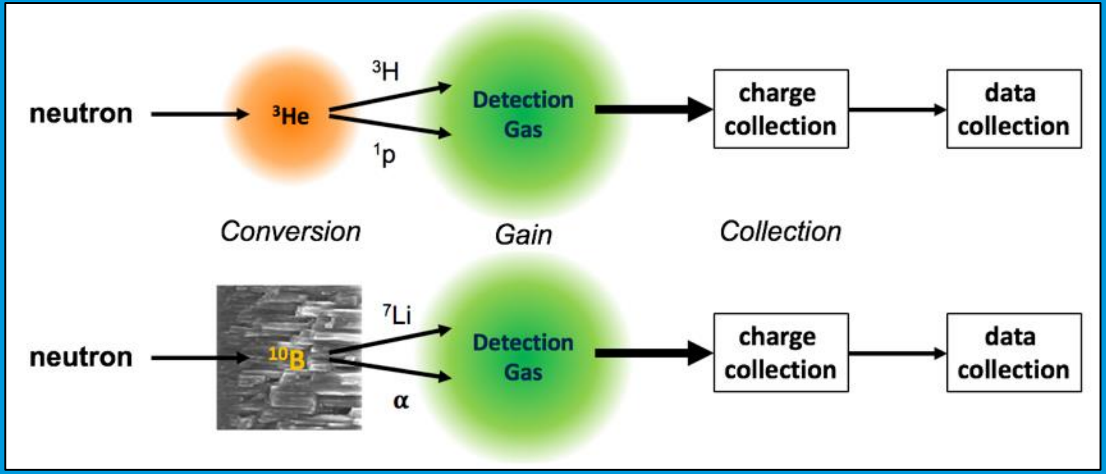
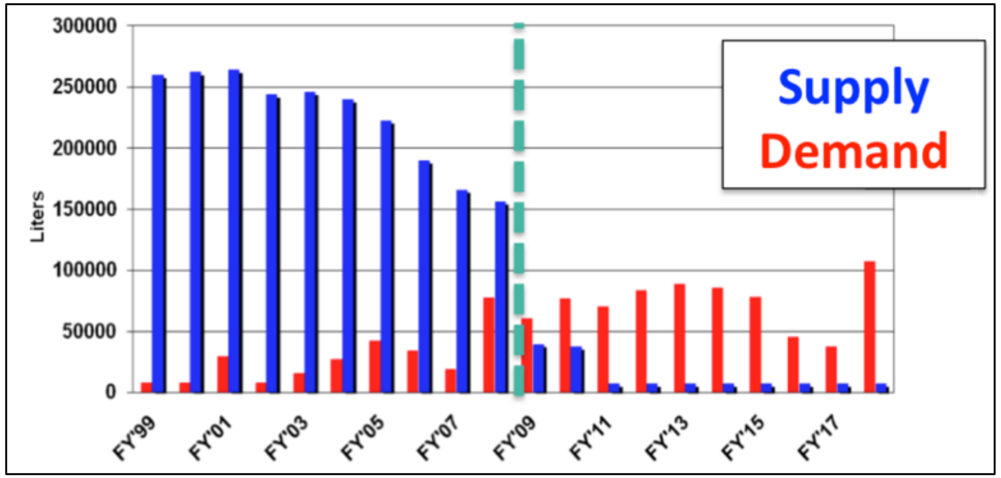
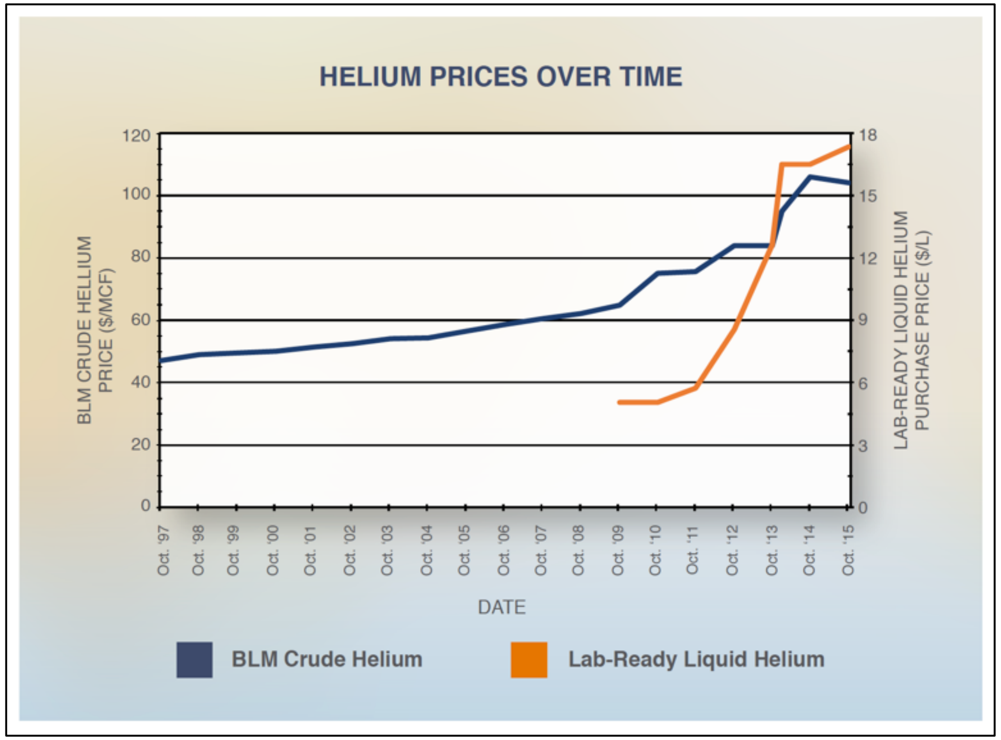
Background: ESS and B_4C



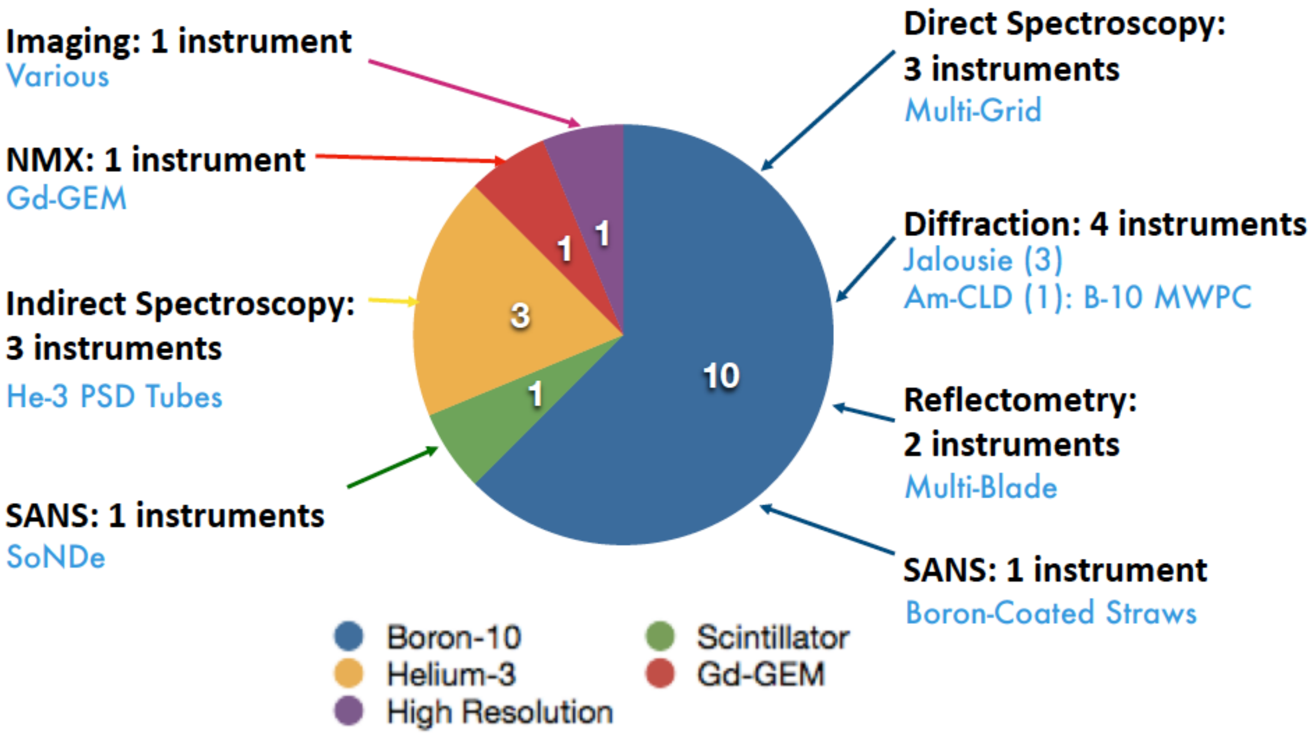




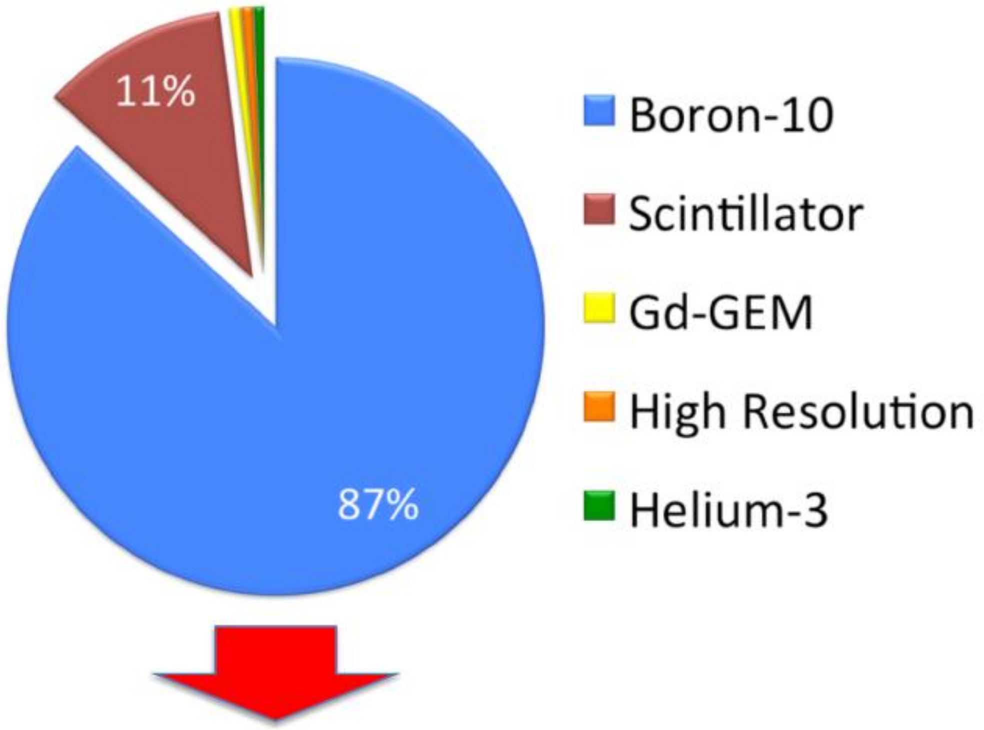
Up: ESS Technical Design Report (2013)
Down: APS POPA Report (2016)

Towards ^{10}B-based *n*-detectors

ESS baseline detector technologies for initial suite



ESS active detection area from initial suite



Focus on large areas!

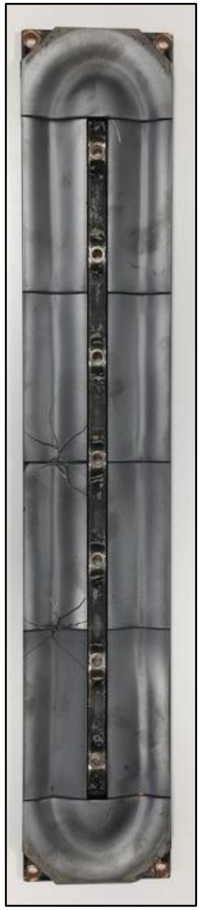
Towards ^{10}B-based *n*-detectors

Chemical compound: boron-carbide (B_4C)

- Non-toxic
- Chemically stable
- Relatively high B-stoichiometry (20 at.%)
- Commercially available

Deposition technique: magnetron sputtering

- Established process
- Commercially available
- <u>Low temperature process possible</u>



B_4C powder and target

Magnetron sputtering machine from CemeCon

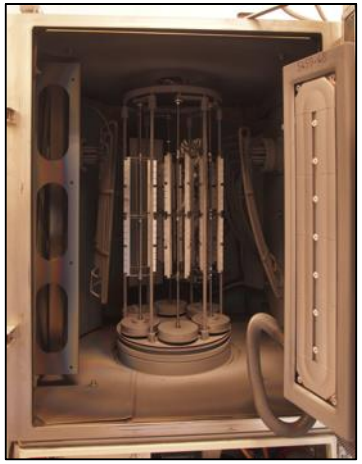
2

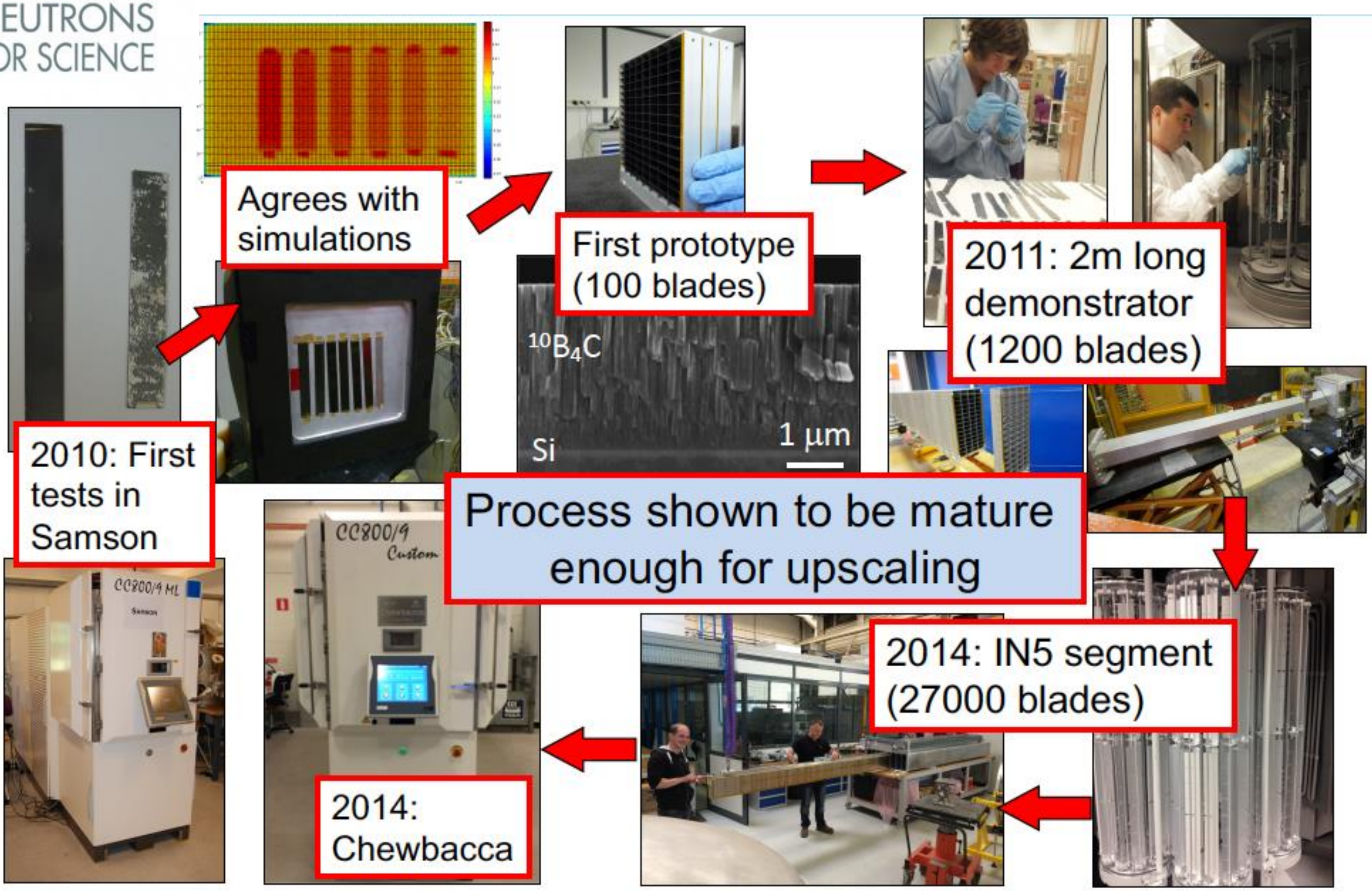
Detector Coatings Workshop in Linköping

Process development for B_4C coatings

Prof. Jens Birch
Thin Film Physics
LiU

Dr. Carina Höglund
Former-Colleague
ESS



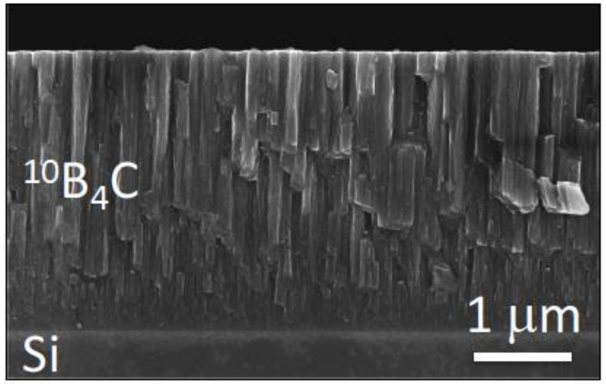


Process development for B_4C coatings

Requirements to B_4C thin films as neutron converter:

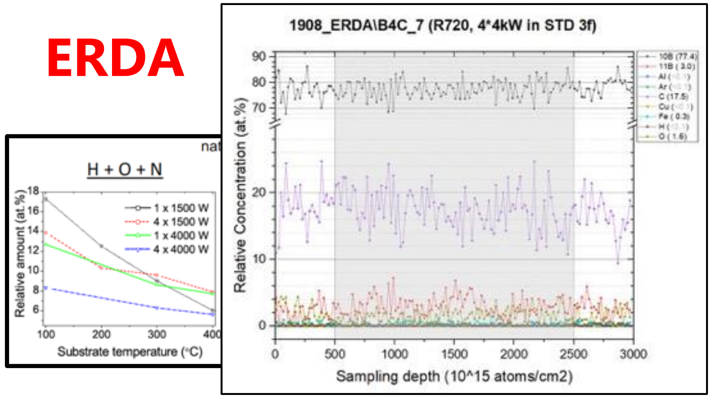
- Good adhesion to substrates
- 1- or 2-side coating possible
- Large area possible
- High density
- Low impurity level
- Controllable thickness and uniformity
- Neutron radiation hard
- Cheaper or at least similar to pre-2009 ^3He price

SEM ~3 µm $^{10}B_4C$



- ✓ Near fully dense film
- ✓ Good adhesion on Ti, Al, Si
- ✓ Uniform thickness
- ✓ Robust with increased thickn. and temperature
- ✓ No clear difference between $^{nat}B_4C$ and $^{10}B_4C$

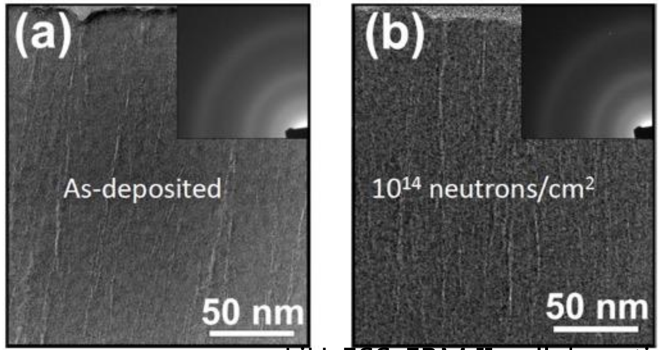
ERDA



- ✓ <2 at.% of impurity (H, O)
- ✓ 96-97 % ^{10}B enrichment
- ✓ Results are consistent

- ✓ Special thanks to LiU and the Tandem Lab in Uppsala University for setting up measurements.

TEM



LiU-ESS-FRM II collaboration

- ✓ No significant influence on physical properties or compositions.
- ✓ Low ^{10}B atoms consumption within a flux of 10^8 n/cm^2/s

C.Höglund et al., Rad. Phys. Chem. 113 (2015) 14

C.Höglund et al., J. Appl. Phys. 111 (2012) 104908

S.Schmidt et al., J. Mat. Sci. 51 (2016) 10418

UPPSALA UNIVERSITET

Process development for B_4C coatings

2014: Setup ESS Detector Coatings Workshop in Linköping and Chewbacca

Chewbacca

2017 Apr.: Move to current site

2017 Aug.: Lai joined in ESS

2019 Feb.: Carina Höglund left ESS

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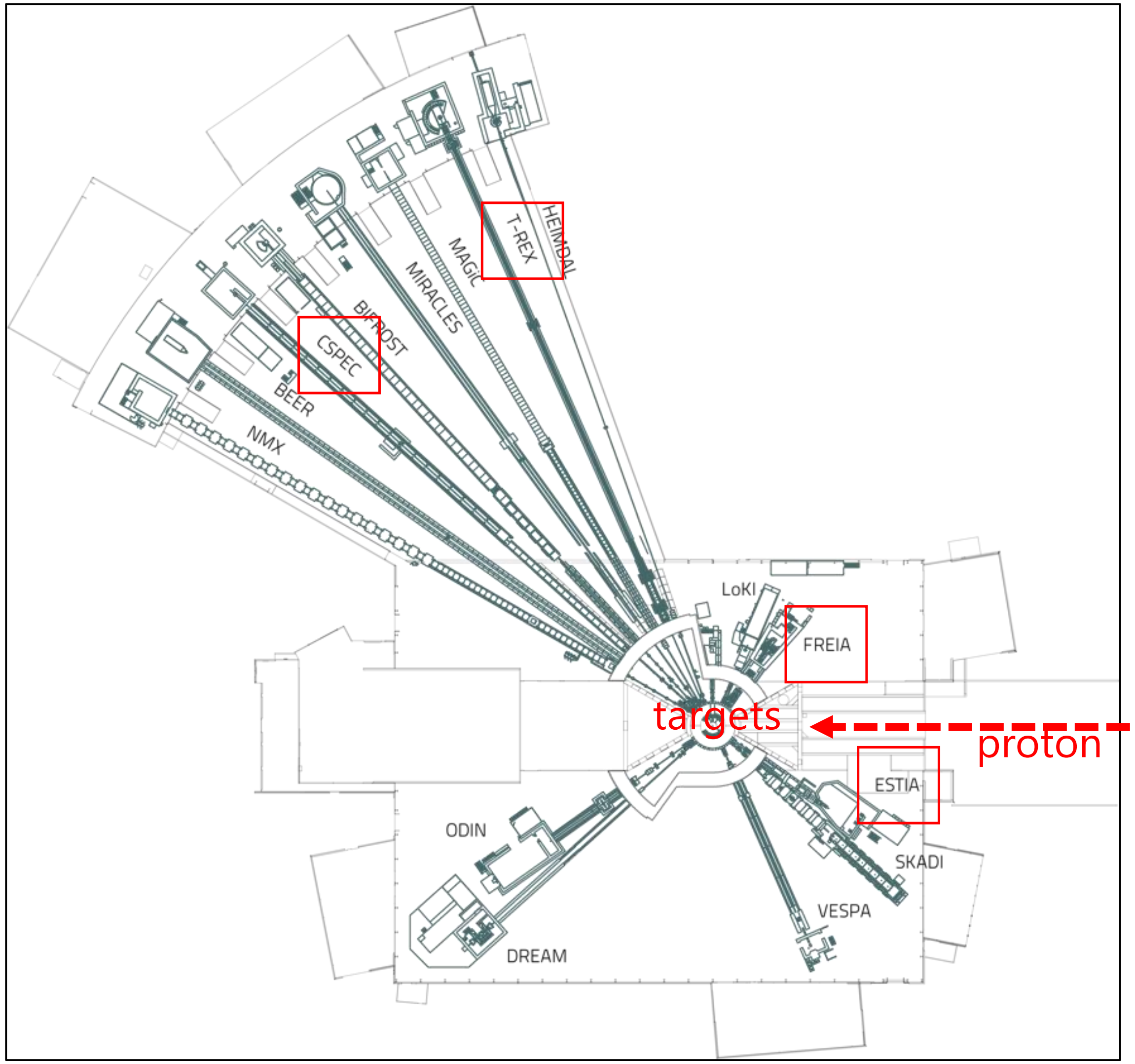
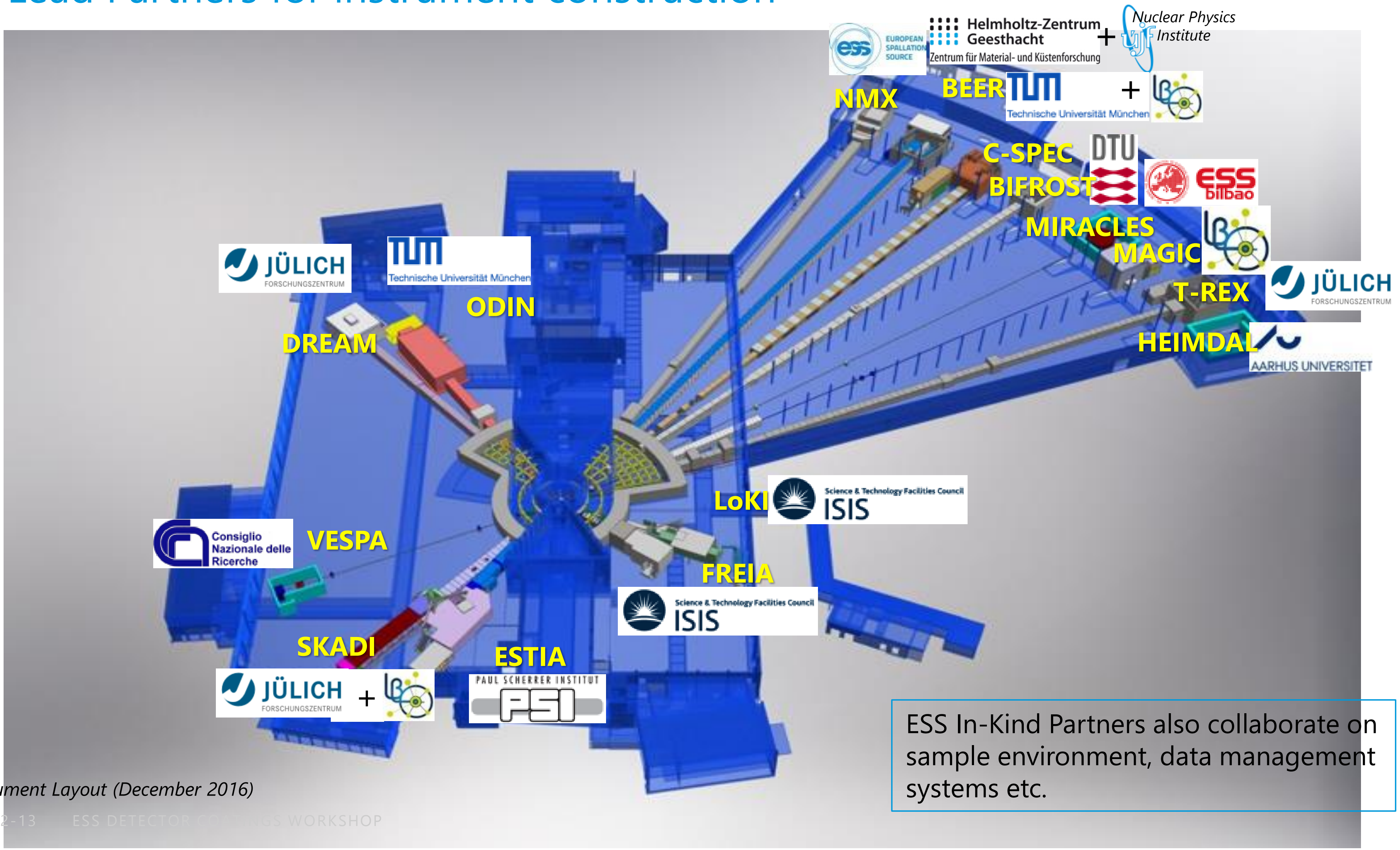
Production Development



NSS Neutron Instrument positions

ESS Lead Partners for instrument construction



ESS Instrument Layout (December 2016)

ESS In-Kind Partners also collaborate on sample environment, data management systems etc.

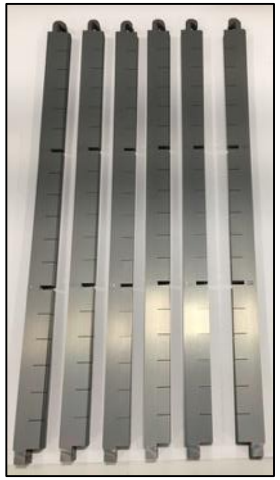
Multigrid detectors

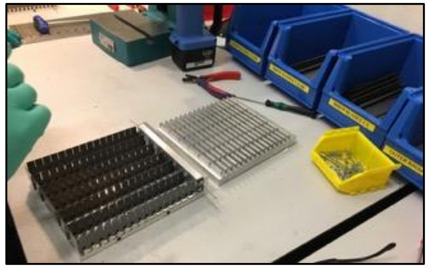
Candidate technology for CSPEC and TREX in ESS.

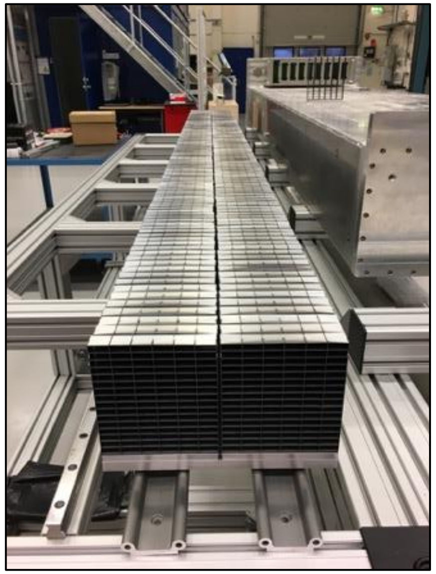
- Prototype 1 (2010)
- Prototype 2 (2011) – 2 m segment detector
- IN6 Segment (2012) – side-by-side comparison with ^{3}He
- IN5 Segment (2014) – full size production

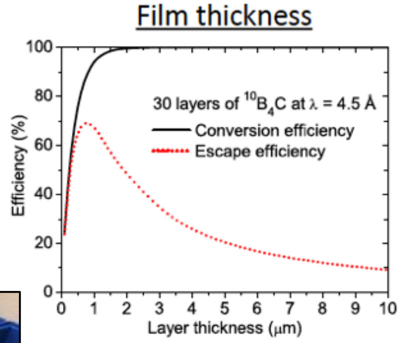
  (0.8 * 3 m^2 detection area, >100 m^2 coated area)
- MG.CNCS (2016) – side-by-side comparison with ^{3}He
- MG.SEQUOIA (2017-18) – thermal neutron detection
- MG.SEQUOIA 2 (2018-19)
- MG.300 (2019) – real CSPEC size, 1 vessel (2 detectors)

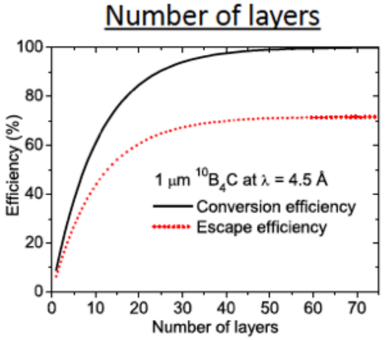
- CSPEC = 54 detectors = 7560 grids = **180,000+ blades**

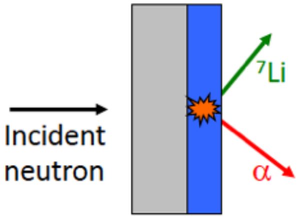


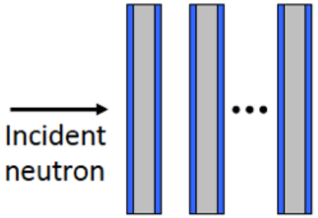


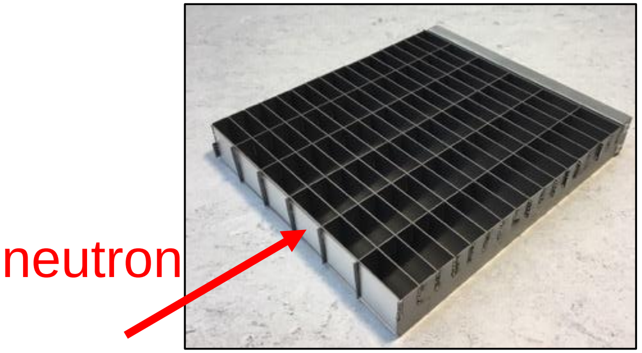












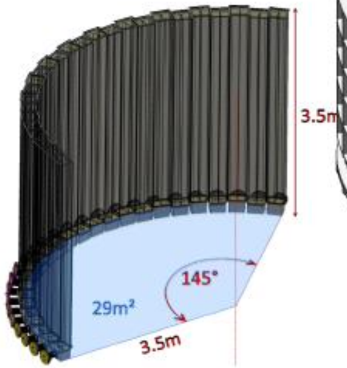
Multigrid detectors

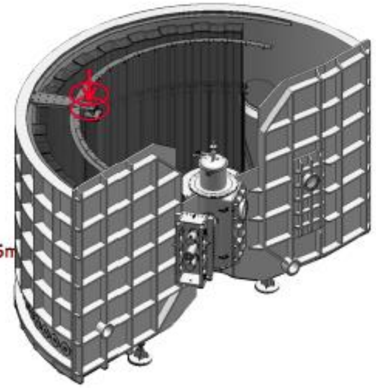
Mass production + engineering challenges!

FRM II
Forschungs-Neutronenquelle
Heinz Maier-Leibnitz

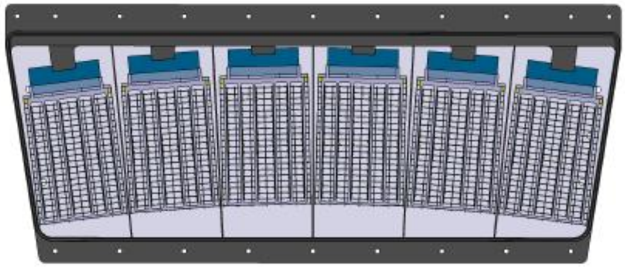
JÜLICH
FORSCHUNGSZENTRUM

CSPEC

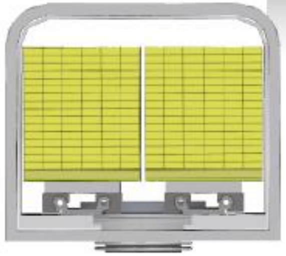


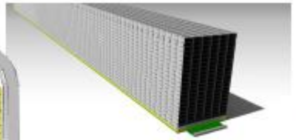


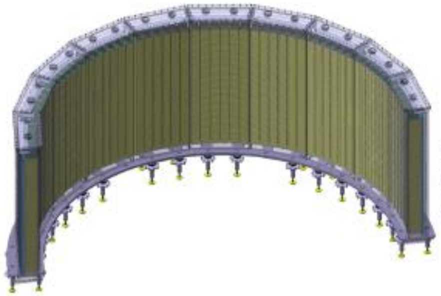
T-REX

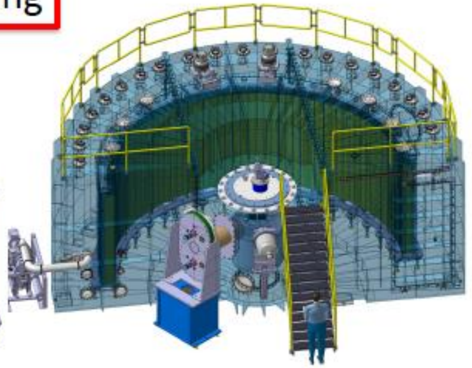


Detailed engineering design is ongoing









Multiblade detectors

Technology for ESTIA and FREIA in ESS:

The Multi-Blade project

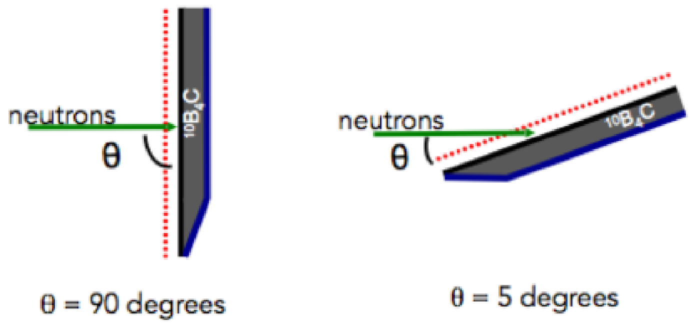
High counting rate capability

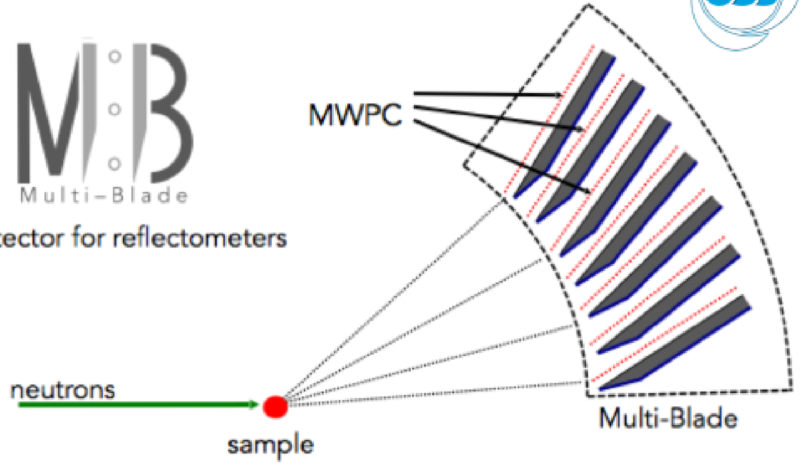
High spatial resolution

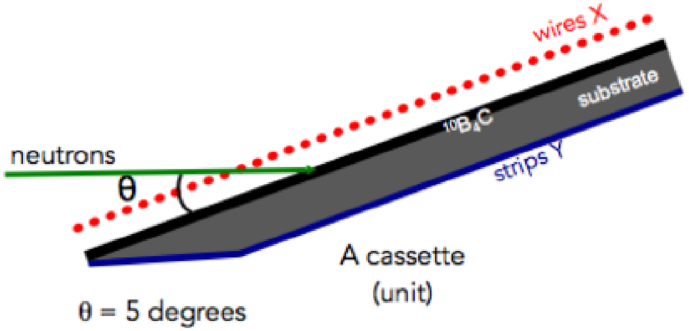
A single Boron layer inclined at 5 degrees

10B-detector for reflectometers

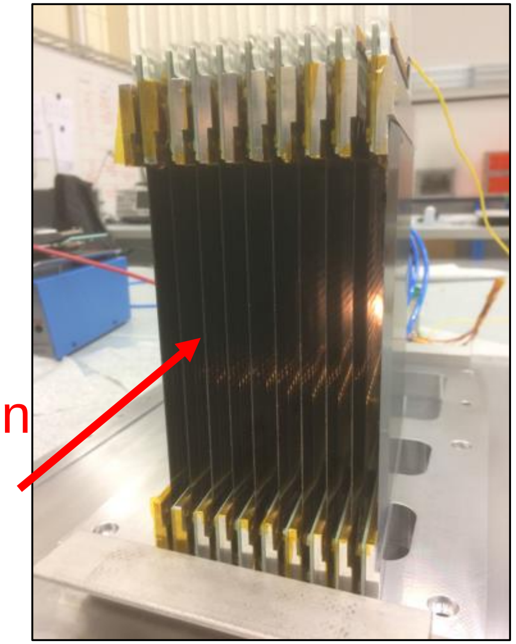
Efficiency <5% at 2.5Å   Efficiency 45% at 2.5Å







neutron



F. Piscitelli et al., J. Inst. 12, P03013 (2017)
F. Piscitelli et al., Proc. R. Soc. A 472 (2016)

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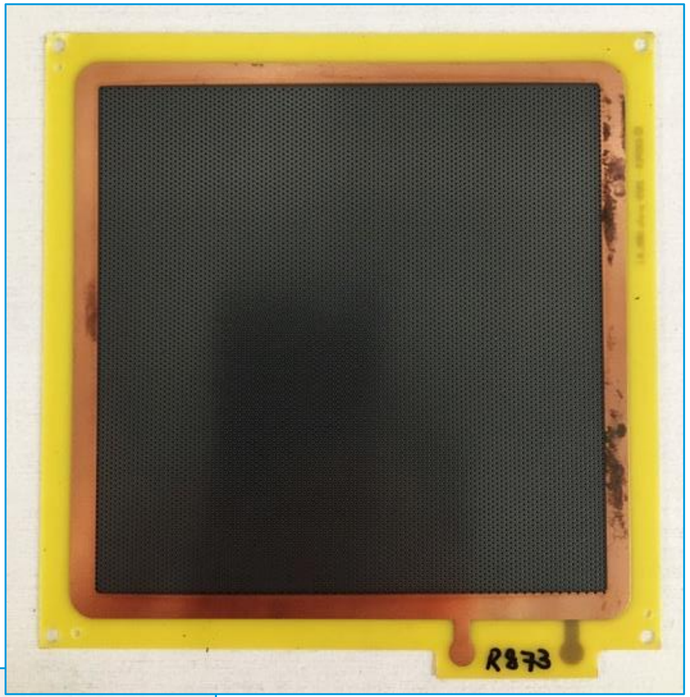
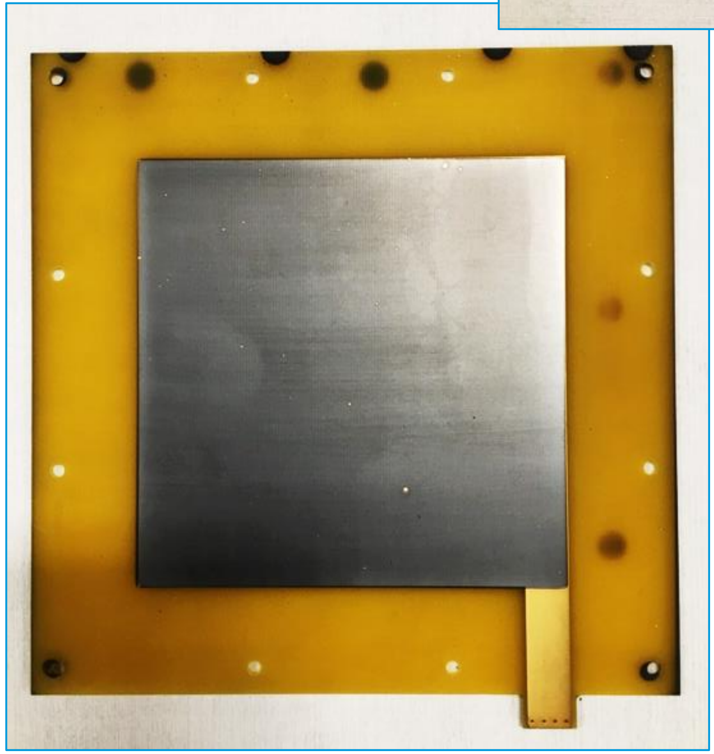
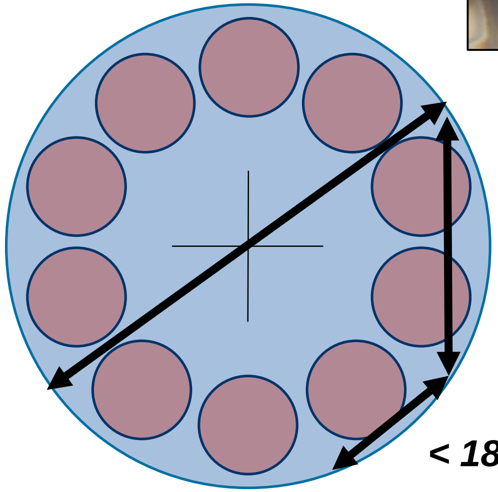
Research Development





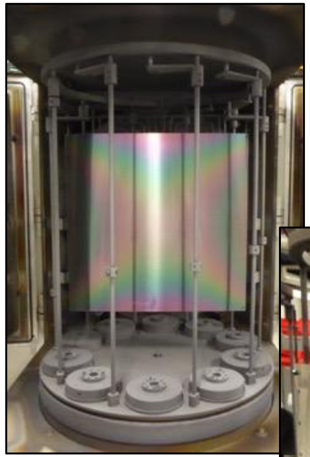
Large area coatings

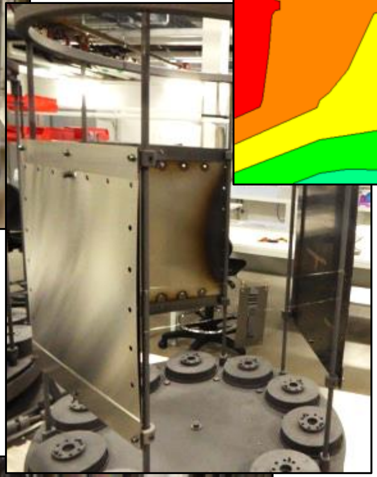
Center (< 670 mm)

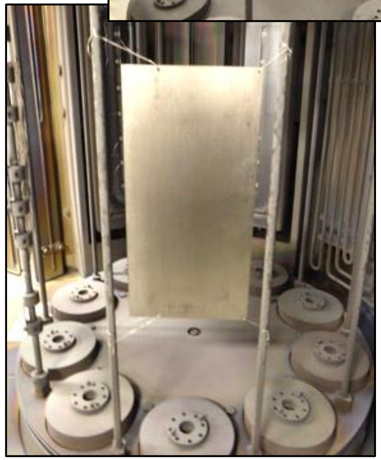


_< 380 mm_

_< 180 mm_

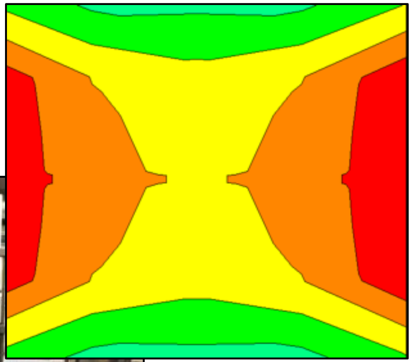






Multiblade

Thickness heat map

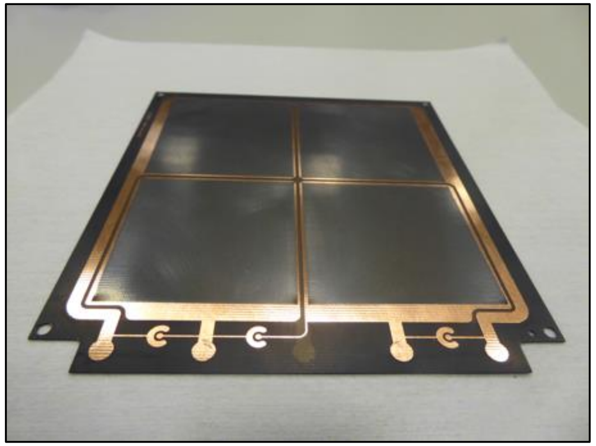


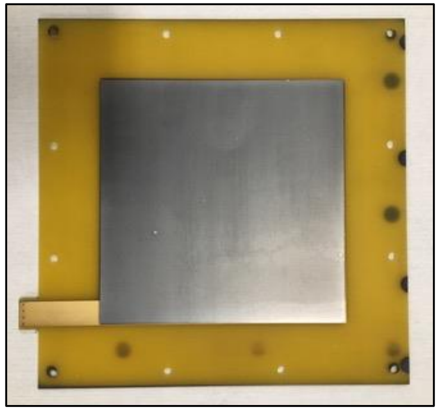
Wrapping around the table (~ 2100 mm)

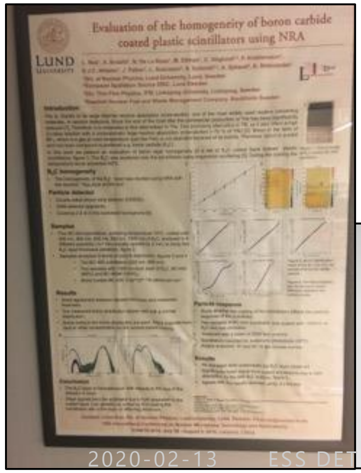
Wrapping around the towers (~ 330 mm x 10 piece)

Temperature sensitive substrates

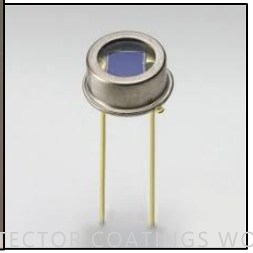
- Max. 4.5 µm done.
- Scratch proof.
- B_4C and Cu done.
- (soft) PCBs, Si-diodes…

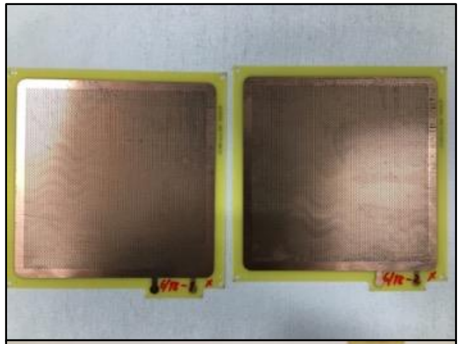


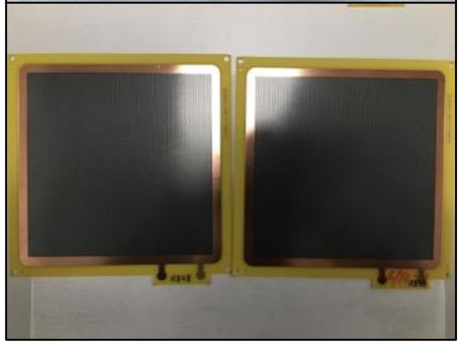


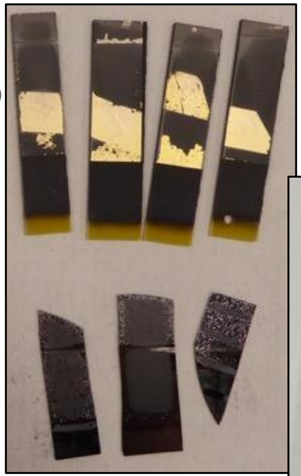


↑ **Cu**/DLC/Pyralux

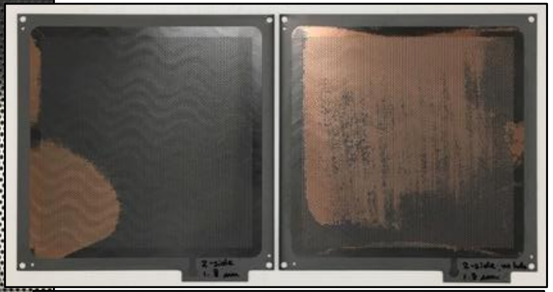








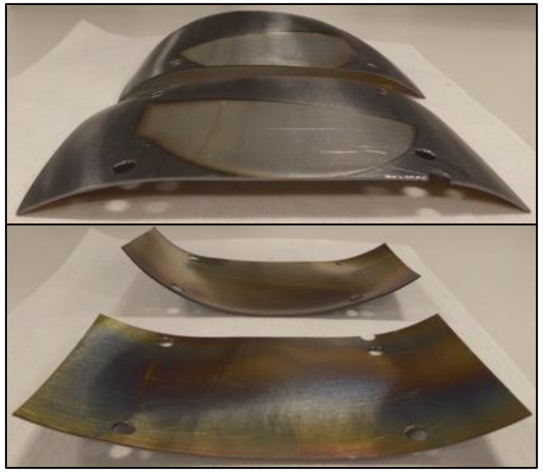
Very challenging



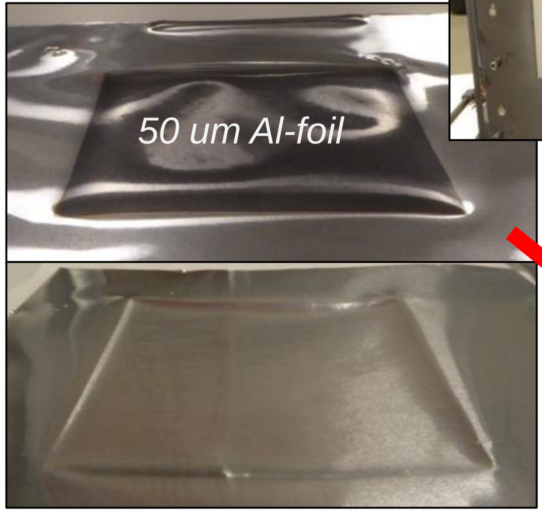
Mechanically soft substrates

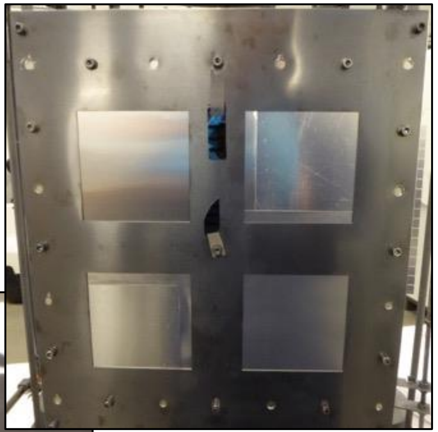
- **Bending**: partly coated soft substrate, e.g. 1-side coated Al

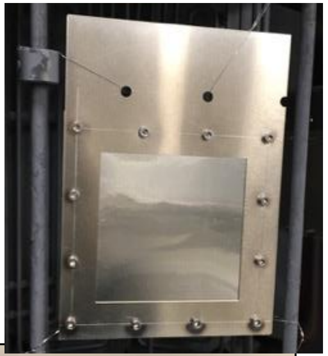
5 μm B_4C on 1 mm Al

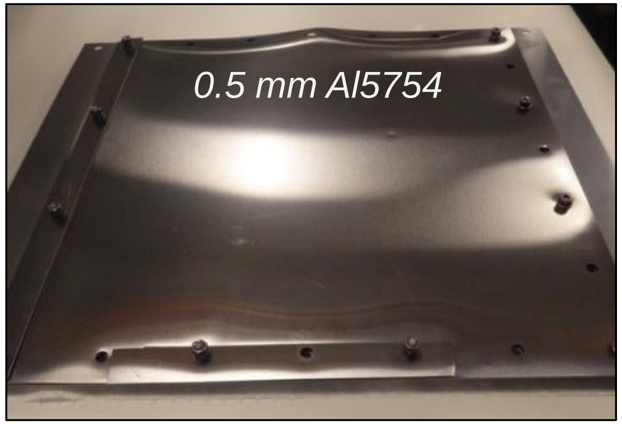


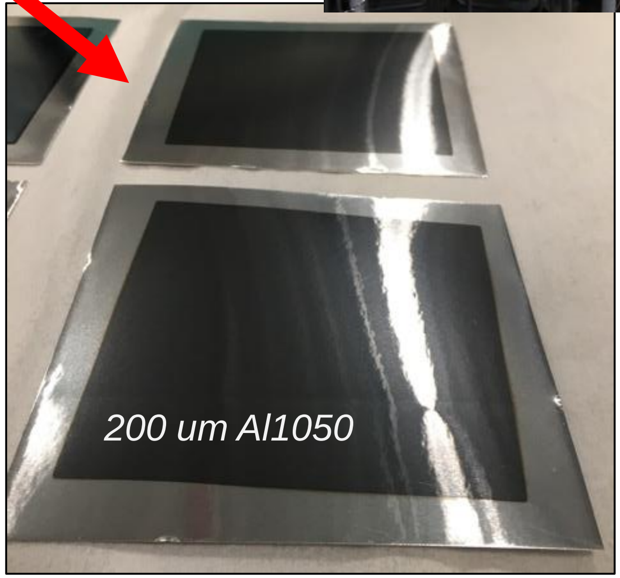
- **Buckling**: physical constrain to thermal expansion, e.g. masks
    - ✓ Lower temperature preferred











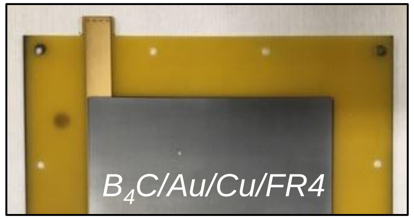
Our material 'tool box'

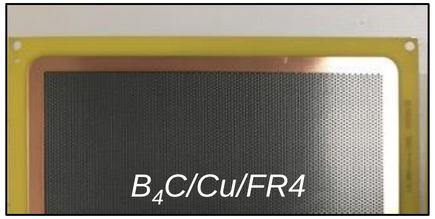
Substrate materials

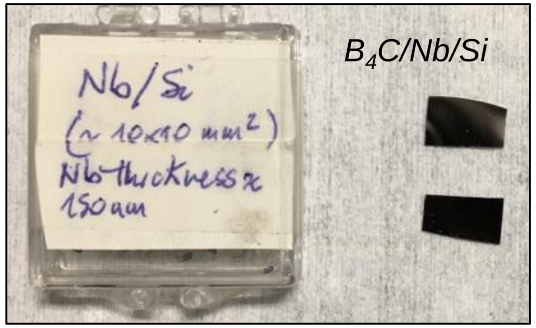
- Metals: Al, Ti, Ni, Nb, Au, Cu, Gd
- Ceramics: Si, Si_3N_4, SiO_x (thermal silica), object glass, SiC-Si_3N_4 composite, AlN, DLC
- Others: PCBs (G10 and FR4), Pyralux

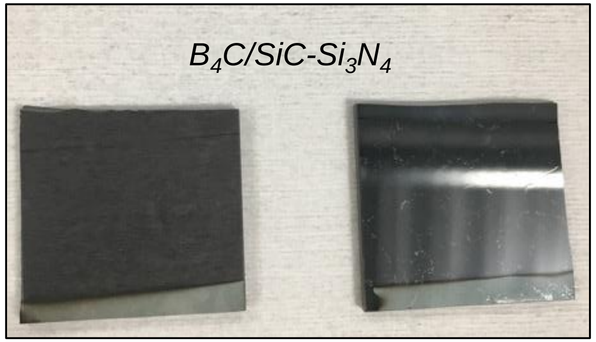
Coating materials

- Non-metal: B_4C
- Metals: Al, Ti, Cu

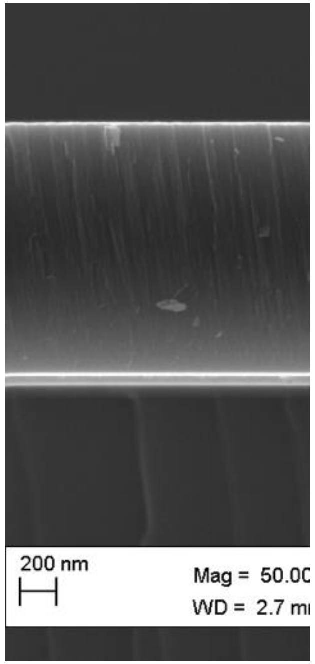




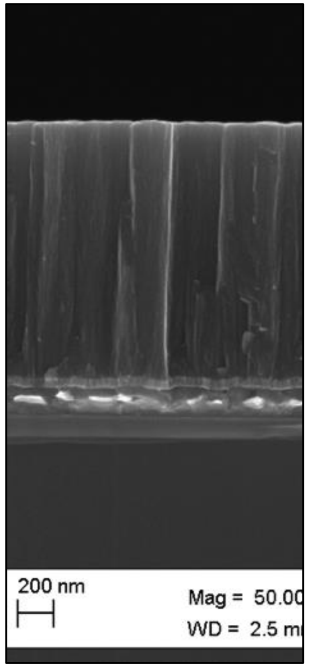




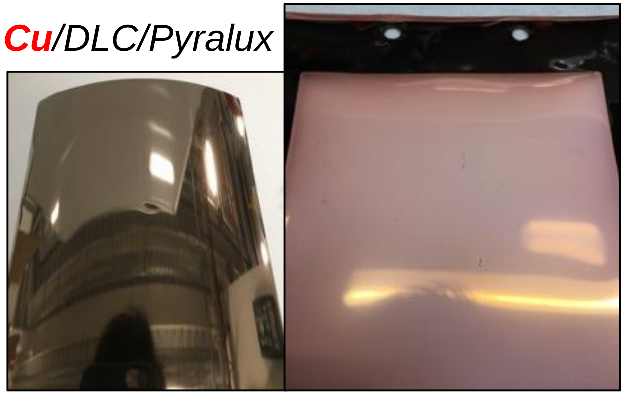
$B_4C/Si_3N_4/Si$



$B_4C/AlN/Si_3N_4/Si$



Cu/DLC/Pyralux



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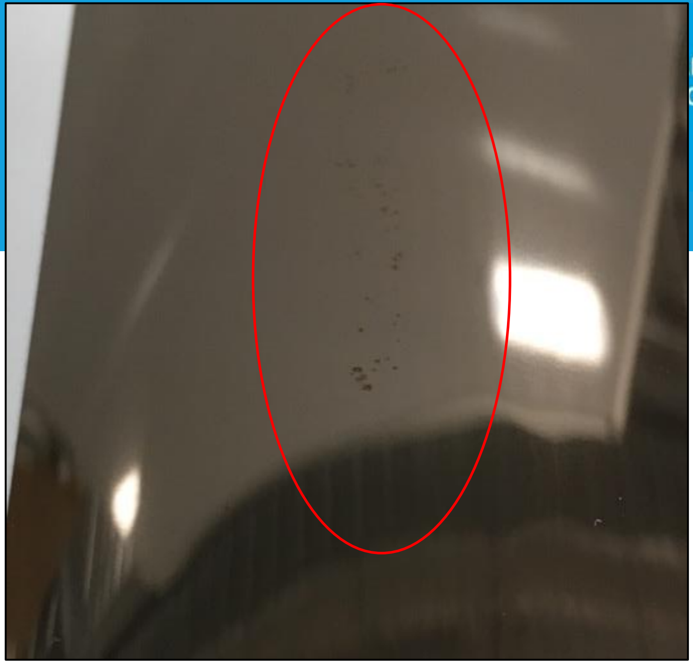
DLC Related Works

Cu deposition on DLC



As received samples

- 4 DLC/Cu-Kapton foils received.
- Packed and separated by paper – we like that!
- Stains and scratches found on the surfaces when received. See red circles to the right.

Cu deposition on DLC

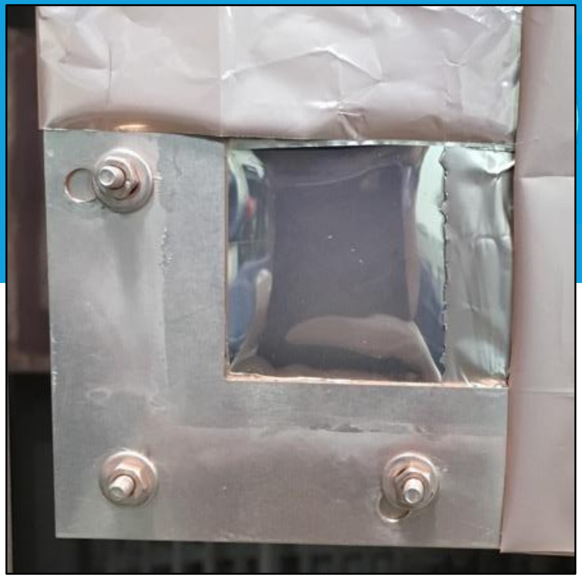
R1013

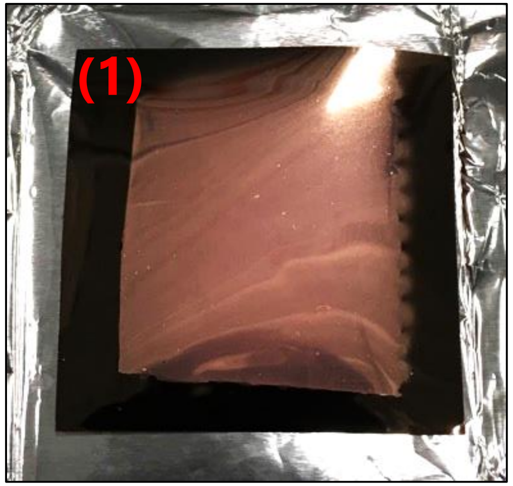
- Sample cut into 1/8 size and masked with Al.

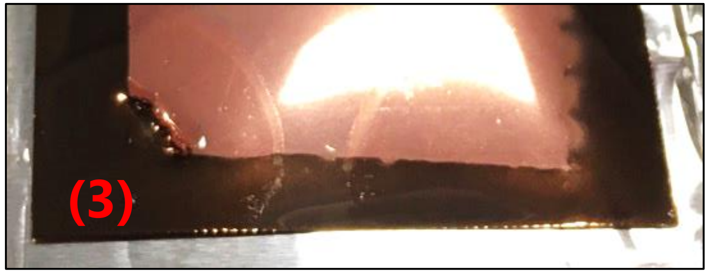
- (1) Cu coated onto the sample without problem. No spontaneous delamination observed.

- (2) Scratch test with tweezers can peel off the Cu layer. However the delamination did not propagate to a larger area → *OK adhesion*.

- (3) After isopropanol spray to the surface, the Cu layer stayed without apparent change.







Cu deposition on DLC

EUROPEAN SPALLATION SOURCE

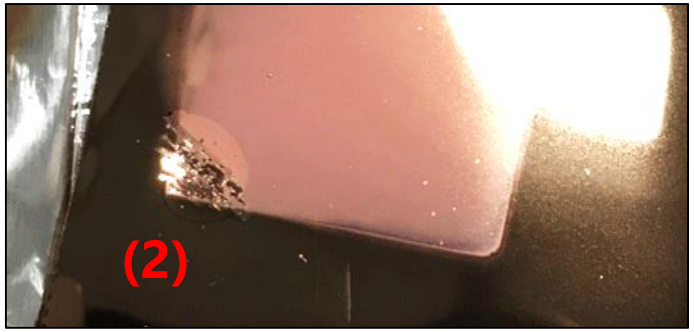
R1014

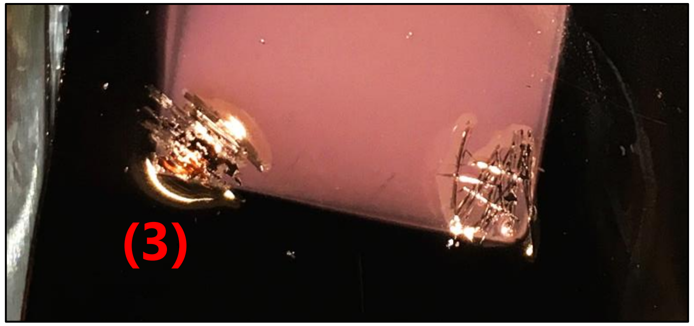
- Sample cut into 1/8 size and masked with Al.

- (1) Cu coated onto the sample without problem. No spontaneous delamination observed.

- (2) Cu layer can only be scratched off with hard force on the tweezers, that is enough to damage the foil. The Cu layer was scratched off in spots instead of big flakes as in R1013, and no further propagation → *much better adhesion*.

- (3) After isopropanol spray to the surface, the

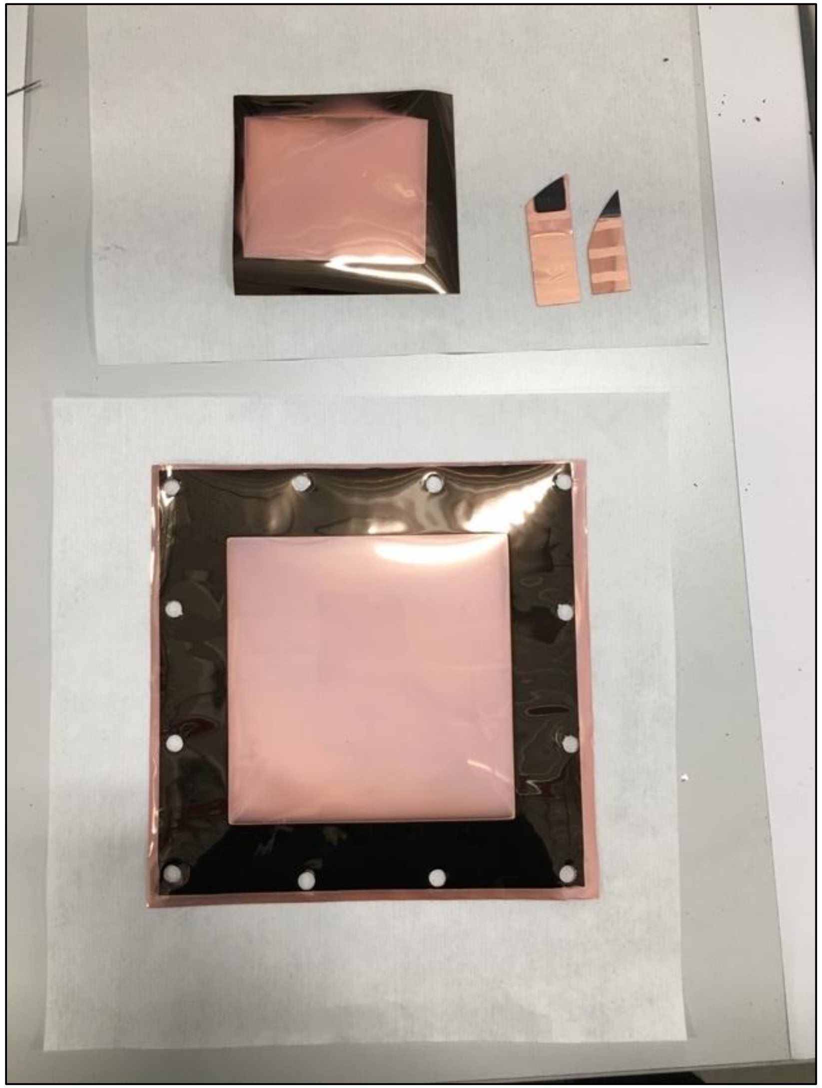




Cu deposition on DLC

R1015

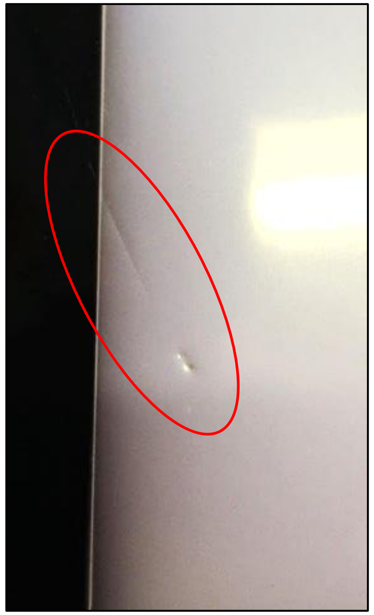
- Test with larger sample size: a 1/4-size and a full-size sample.
- Same process as in R1014.
- The full-size sample was drilled with holes to be mounted in the mask.
- Cu deposition was done with no problem.
- Scratch test done on 1/4-size sample and show no difference with R1014.



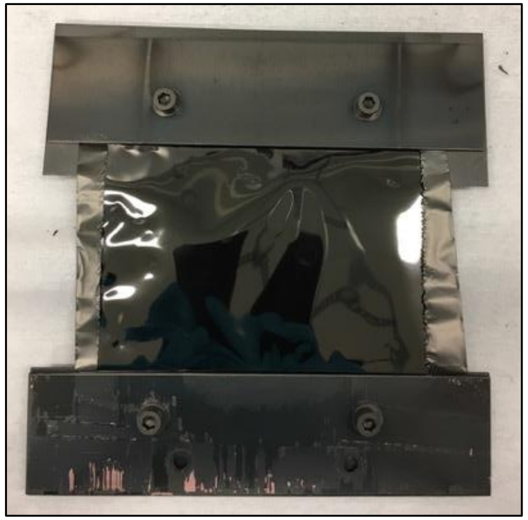
Cu deposition on DLC

R1016

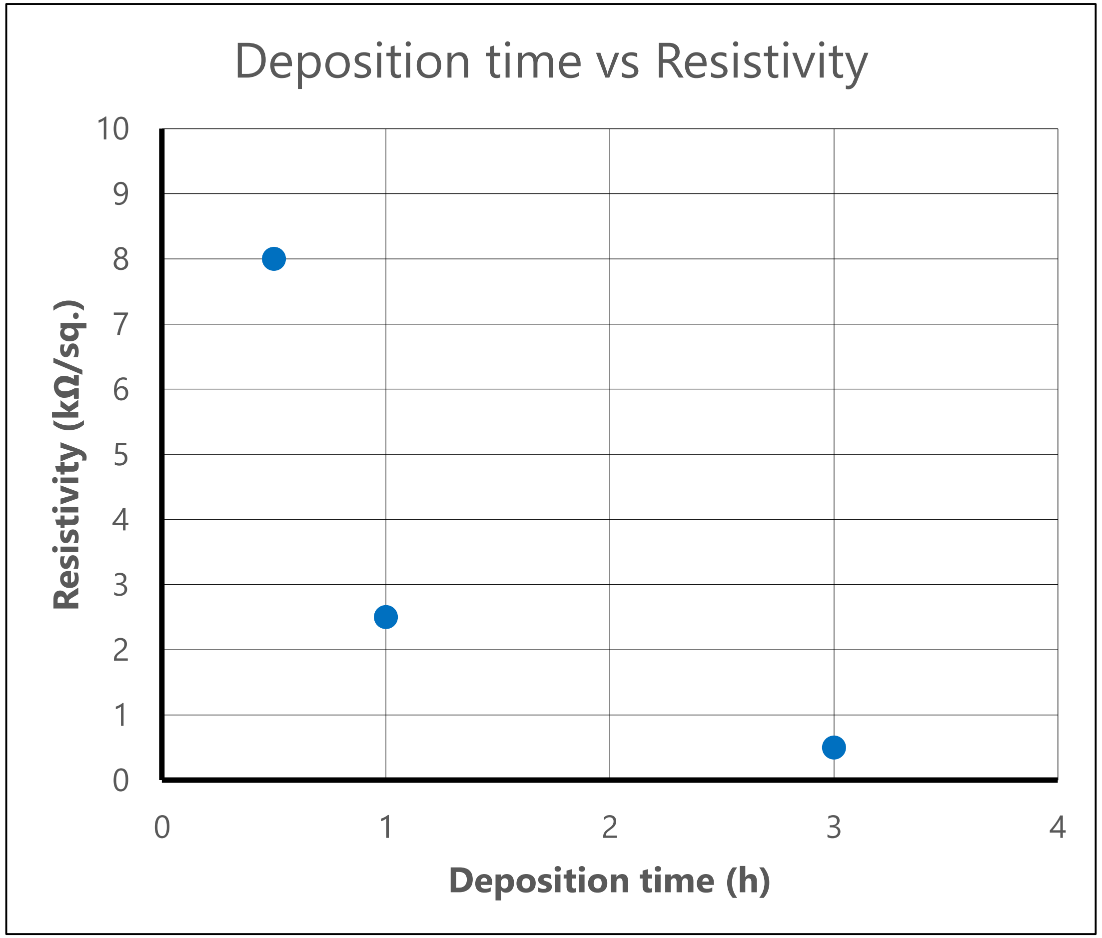
- 2 full-size sample mounted. Same process as in R1014 but aimed for higher thickness.
- Samples were drilled with holes to be mounted in the masks.
- Cu deposition was done with no problem.
- The stains and scratches found on as-received samples can still be seen on the surfaces. See red circles to the right.



DLC deposition?



- First try in Feb. 2020.
- Highest resistivity achieved ~8 – 10 kΩ/sq.
- Other characterization, including thickness measurements, has not been done.



Get in touch

Thanks for your attention!

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