EUROPEAN SPALLATION SOURCE
ESS Detector Coatings Workshop
Linköping Sweden

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2020-02-13
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Background: ESS and B$_4$C

Down: APS POPA Report (2016)
Towards $^{10}$B-based $n$-detectors

**ESS baseline detector technologies for initial suite**

- Imaging: 1 instrument
  - Various
- NMX: 1 instrument
  - Gd-GEM
- Indirect Spectroscopy: 3 instruments
  - He-3 PSD Tubes
- SANS: 1 instrument
  - SoNDe

**ESS active detection area from initial suite**

- Direct Spectroscopy: 3 instruments
  - Multi-Grid
- Diffraction: 4 instruments
  - Jalousie (3)
  - Am-CLD (1): B-10 MWPC
- Reflectometry: 2 instruments
  - Multi-Blade
- SANS: 1 instrument
  - Boron-Coated Straws

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
- Low temperature process possible
Detector Coatings Workshop in Linköping
Process development for B₄C coatings

- 2010: First tests in Samson
- 2011: 2m long demonstrator (1200 blades)
- 2014: IN5 segment (27000 blades)

Agrees with simulations
First prototype (100 blades)
Process shown to be mature enough for upscaling

Prof. Jens Birch
Thin Film Physics
LiU

Dr. Carina Höglund
Former Colleague
ESS

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ESS DETECTOR COATINGS WORKSHOP
Process development for B₄C coatings

Requirements to B₄C 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

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

TEM

SEM

ERDA

Near fully dense film
Good adhesion on Ti, Al, Si
Uniform thickness
Robust with increased thickn. and temperature
No clear difference between natB₄C and ¹⁰B₄C

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

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

LiU-ESS-FRM II collaboration

C.Höglund et al., Rad. Phys. Chem. 113 (2015) 14
Process development for $\text{B}_4\text{C}$ coatings

2014: Setup ESS Detector Coatings Workshop in Linköping and 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
ESS Lead Partners for instrument construction

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!

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

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

F. Piscitelli et al., J. Inst. 12, P03013 (2017)
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Research Development
Large area coatings

Center (< 670 mm)

< 380 mm

< 180 mm

Thickness heat map

Wrapping around the table (~ 2100 mm)

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

Multiblade
Temperature sensitive substrates

- Max. 4.5 µm done.
- Scratch proof.
- B₄C and Cu done.
- (soft) PCBs, Si-diodes...
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

  0.5 mm Al5754

  50 µm Al-foil

  200 µm Al1050
Our material ‘tool box’

**Substrate materials**
- Metals: Al, Ti, Ni, Nb, Au, Cu, Gd
- Ceramics: Si, Si$_3$N$_4$, SiO$_x$ (thermal silica), object glass, SiC-Si$_3$N$_4$ composite, AlN, DLC
- Others: PCBs (G10 and FR4), Pyralux

**Coating materials**
- Non-metal: B$_4$C
- Metals: Al, Ti, Cu
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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.
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

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?

- Highest resistivity achieved \(~8 - 10 \text{k} \Omega / \text{sq.}\).
- Other characterization, including thickness measurements, has not been done.
Get in touch

Thanks for your attention!

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