ESS Detector Coatings Workshop in Linköping

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150317

With a big thank you to all our collaborators, and in particular the ILL, with whom this collaboration started!

Outline

- What were the conclusions and outlook in October 2013 (last CERN meeting) presented by Carina Höglund
- Todays status of ESS Detector Coatings Workshop in Linköping and $^{10}B_4C$ thin film production
- Depositions on different substrates
- Mulitigrid prototypes for ILL
- Many collaborations

Conclusions and outlook

- **Alternatives to ^{3}He detectors** are urgently needed
- The **next generation of neutron detectors** will contain **^{10}B thin films**

- $^{10}B_4C$ thin films can be **PVD** deposited with **~80 at.%** of **^{10}B**
- Elevated temperatures and high deposition rates yield **good adhesion**
- The $^{10}B_4C$ coatings are **not damaged** by **neutrons**
- The process can **easily** be **up-scaled** for large area neutron detectors
- Thermal **CVD** and **PACVD** for complex structures is under development

- **Technology demonstration**
  → 4 multi-grid prototypes, incl. IN6 segment ➔ **50% detection efficiency!**
  → Grooved cathodes can raise the efficiency by 30%
  → Several collaborations show promising prototype results
- **Up-scale** the process
  New deposition system (dedicated for $^{10}B_4C$) is available in ~6 months!
  2019 → First 7 instruments at ESS need **>3000 m^2 coatings**
  2025 → 15 more instruments at ESS need **>5000 m^2 coatings**

ESS Detector Coatings Workshop in Linköping – Milestones

- Sep 2013 a CC800/9 Sputtering machine from CemeCon was ordered
- Feb 2014 a contract was signed for the ESS Detector Coatings Workshop, situated in Linköping 5 min by bike from the University and 2,5 h by train from Lund. The area of the workshop is 225 m^2 +140 m^2 office
- The sputter machine, “Chewbacca”, was installed and tested last summer
- Linda was hired and started working 1 of Sep as “Coatings Production Engineer”
- The Workshop, that was almost empty by Sep, was gradually filled up with office furniture and supplies but more importantly with lab equipment and furniture
- 7th of Nov we had our big inauguration event and the sputtering machine got his name – Chewbacca!


CC800/9
Custom


Chewbacca
7 Nov 2014


CC800/9
Custom

ESS Detector Coatings Workshop in Linköping – Production

- Staff at ESS in Linköping:
  - Carina, Susann and Linda
  - add out right now for a production technician
  - temporary manpower as appropriate
- Linda role is to focus on Production and Carina/Susann on R&D
- Spring 2015, focus will be on R&D, small scale production and getting the WS up and running for full production
- Approximately 1000m^2 per year in full production
- Raw Production cost below 1000€ per m^2 at full machine capacity
- For example up to 500 IN5 blades per run and 3 runs per day

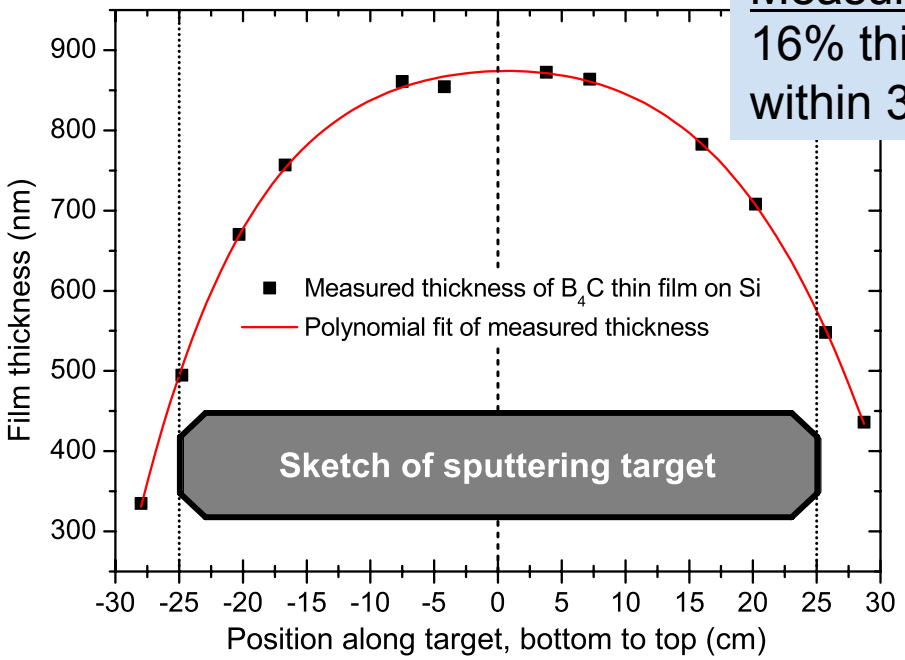
Carina Höglund

Susann Schmidt

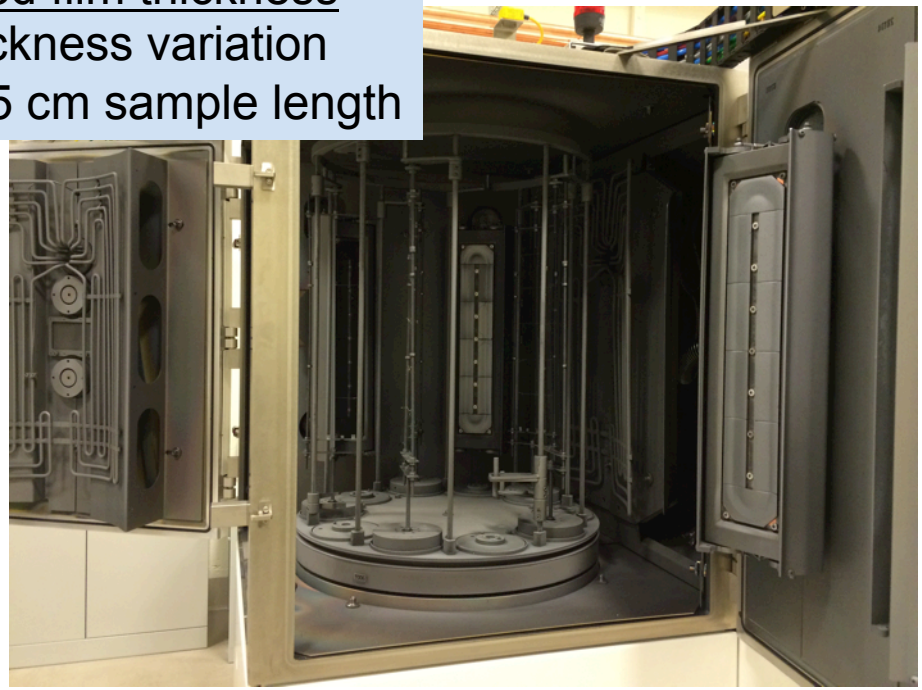
Linda Robinson

ESS Detector Coatings Workshop in Linköping – Chewbacca, CC800/9 Sputtering machine

- Batch loading system
- 4 magnetrons for 8x50 cm^2 sputter targets with shutters
- Targets with B_4C, $^{10}B_4C$
- 40-50 cm sample height depending on uniformity, double coated maximum 13 cm wide, diameter of substrate table in chamber is 60 cm
- 3-fold substrate rotation



Measured film thickness
16% thickness variation
within 35 cm sample length

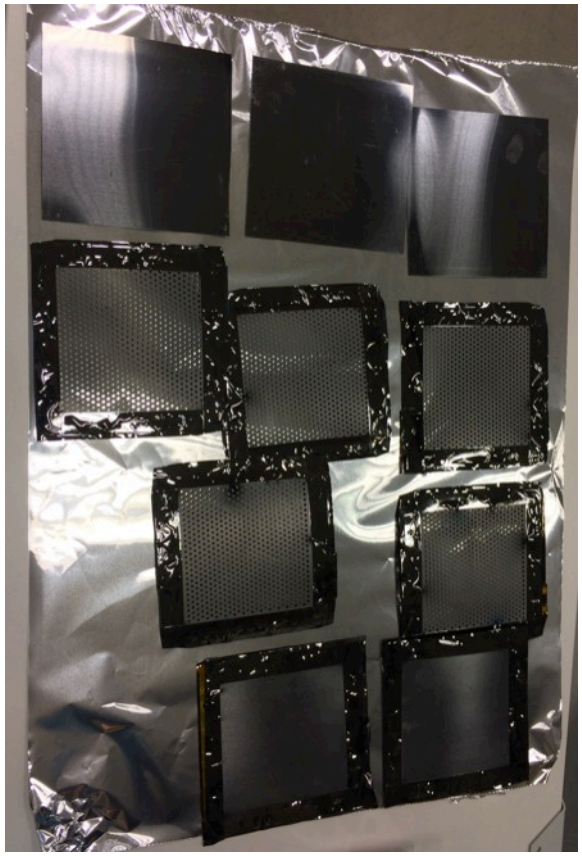


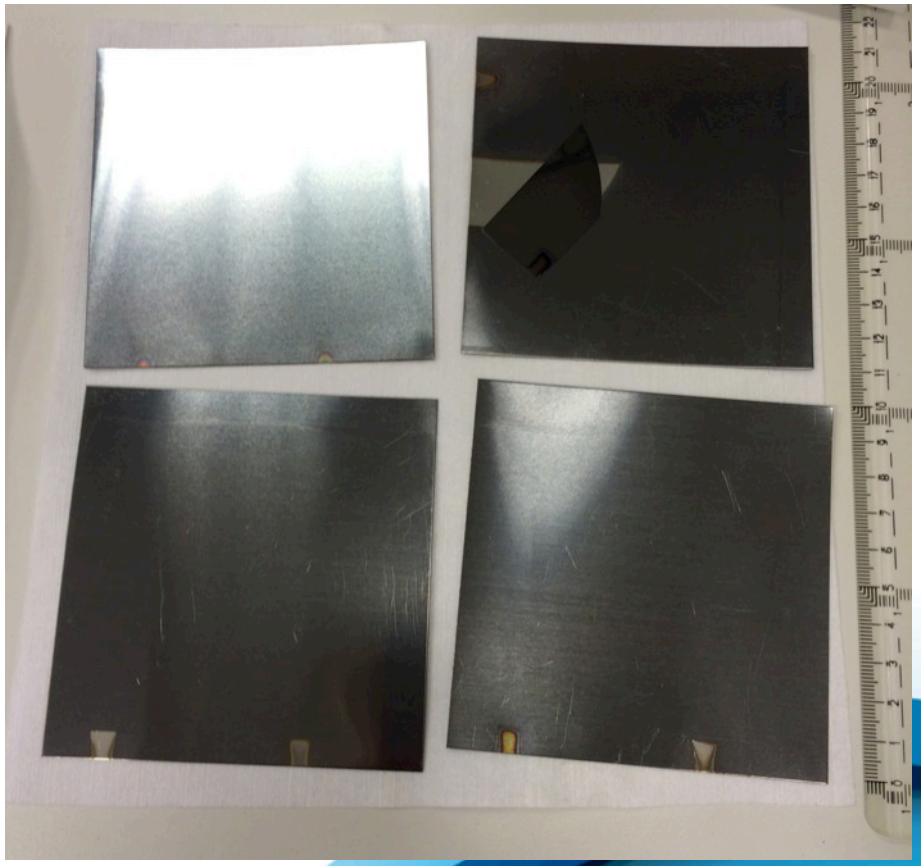
ESS Detector Coatings Workshop in Linköping – at the moment

- Depositions - Chewbacca is up & running
- Cleaning - Ultrasonic bath (with dimensions 50cm x 30cm x 15cm) at the WS
- Characterization – we have an optical microscope (Olympus BX51) at the WS and easy access to characterization equipment available at the University such as SEM, TEM, SIMS, XPS, XRD, (ERDA and RBS at different location) etc.
- Template – if you want depositions to be done please fill in our template explaining what kind of deposition that you need and on what kind of substrate and send it to us.
- The depositions will be prioritized and ESS/ILL collaborative needs will be at the top of the list
- Lead time for a big order is about 4 months because of the targets, less for smaller orders

Coating on different substrates -Al

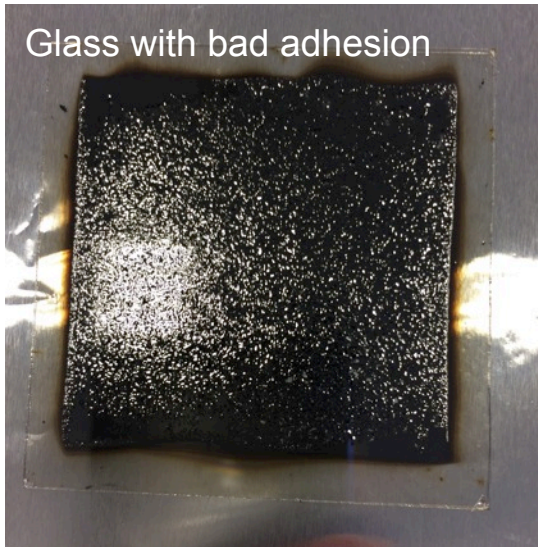
- Coating B_4C on Al in different shapes is no problem





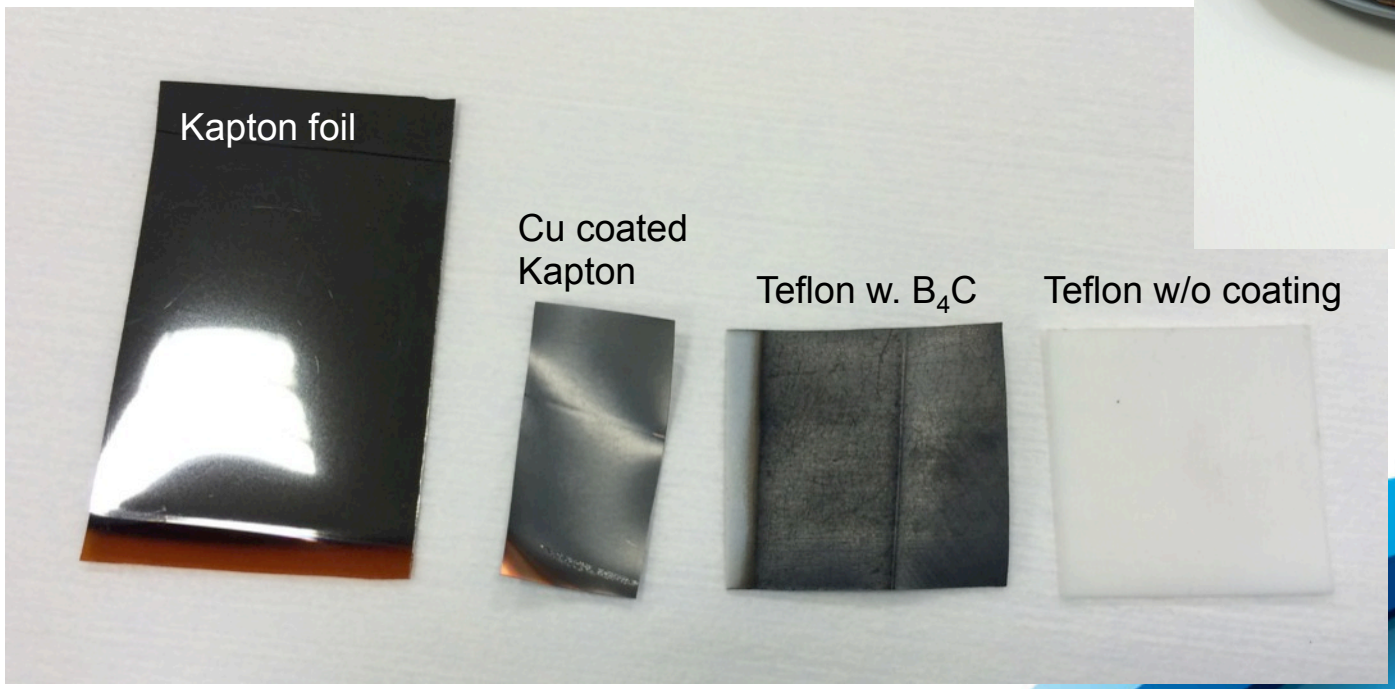
Coating on different substrates – Glass, Kapton tape and Cu

For glass it is important with a clean surface. You se different results on glass, the middle picture has a coating that sticks well to the sliding glass. On top of the sliding glass is Kapton tape with B_4C coating and afterwards the tape was removed with the coating still attached.



Coating on different substrates – Teflon, Kapton foil, Cu coated Kapton foil

- First attempt with B_4C coating seems to stick well to Kapton foil and Cu coated Kapton foil. But the Cu coated foil got a bit bend.
- The first attempted of coating on Teflon also seems to stick, but is cracked.



Summary - Substrates used for $^{10}B_4C/natB_4C$

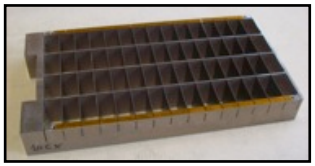
Easy	Ongoing	Not tried
Al	Glass - ok solution on clean samples	Which substrates do you think you will need?
Al-foil	Ni - ok solution	
Stainless steel	Some LT materials	
Al_2O_3	MgO	
Si	Teflon	
G10	Cu coat. Kapton	
Kapton tape	Kapton foil	
Cu		

- Testing various substrates = Ongoing work
- Higher temperatures always give better films!

Multi-grid prototypes at ILL

Collaboration ESS – LiU – ILL
Based upon ILL Multigrid design

Prototype 1 (2010) – **Proof of principle**



Prototype 2 (2011) – **Long detector segment**

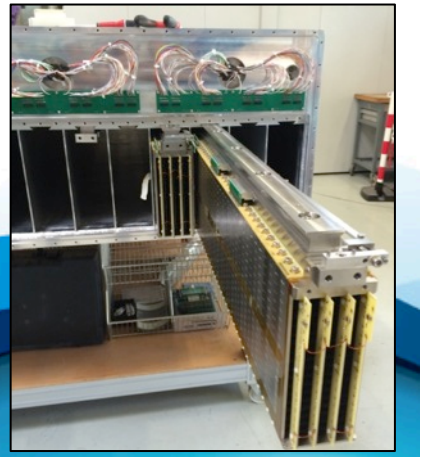
- ➔ 96 frames, 2 m long
- ➔ 1200 blades coated with $^{10}B_4C$

IN6 segment (2012) – **Side-by-side comparison with ^{3}He**

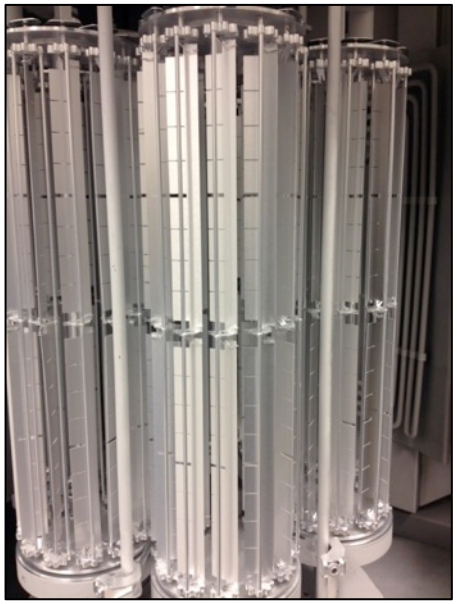
- ➔ Replace 25 st ^{3}He-tubes
- ➔ 2 weeks “normal” beam time

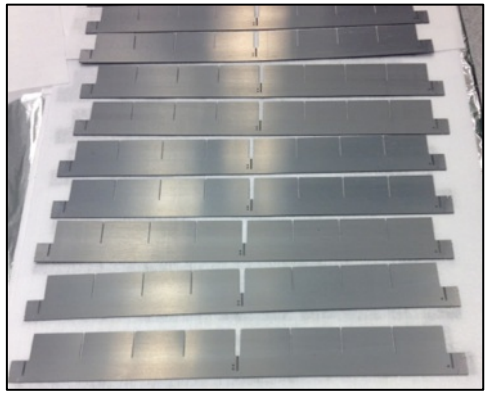
IN5 segment (2014) – **Full scale segment,**

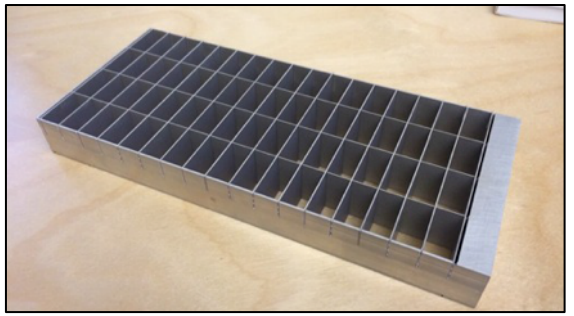
- ➔ Mass production test



IN5 segment

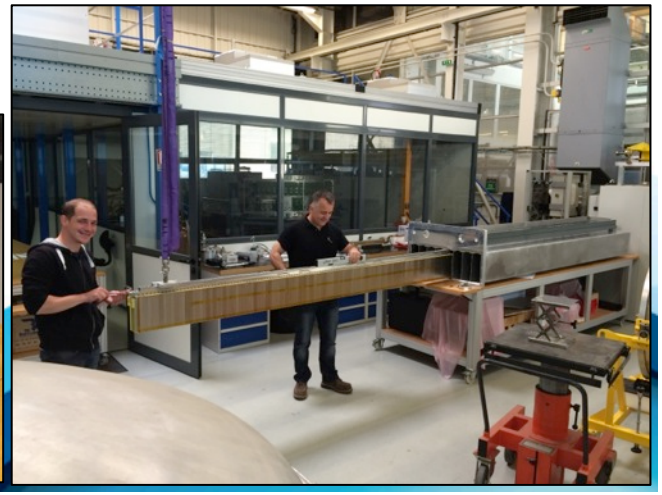






- Collaboration ESS – LiU – ILL
- 0.8 x 3 m^2 detector area
- 27 000 blades with $^{10}B_4C$ (>100 m^2) in 45 days
- factor 10 higher volume than produced before

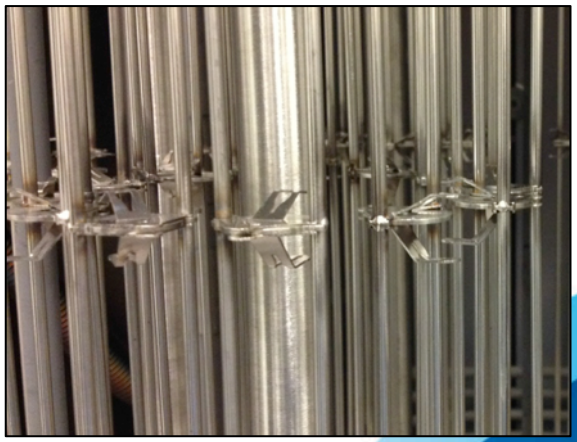
➔ Mass production test!



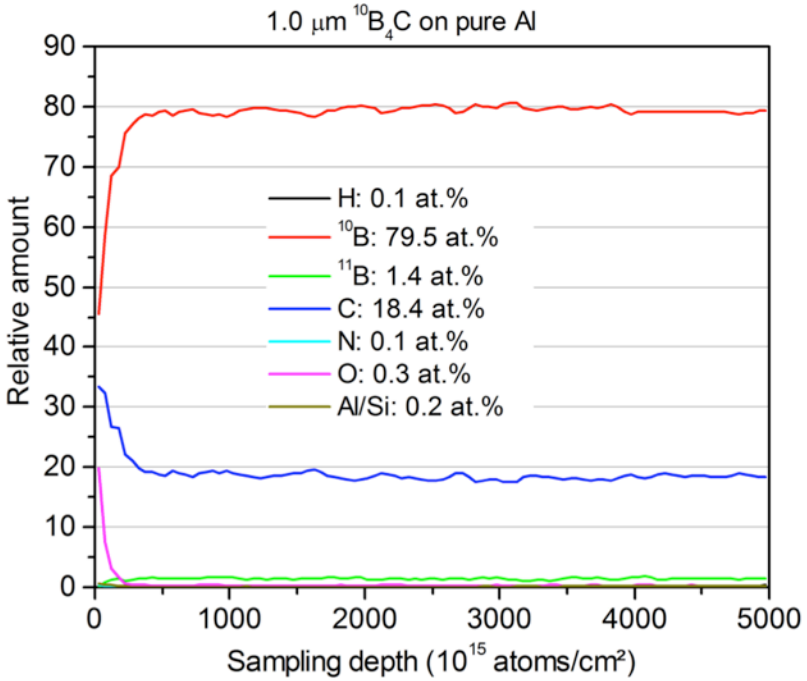
The deposition system – Samson at LiU

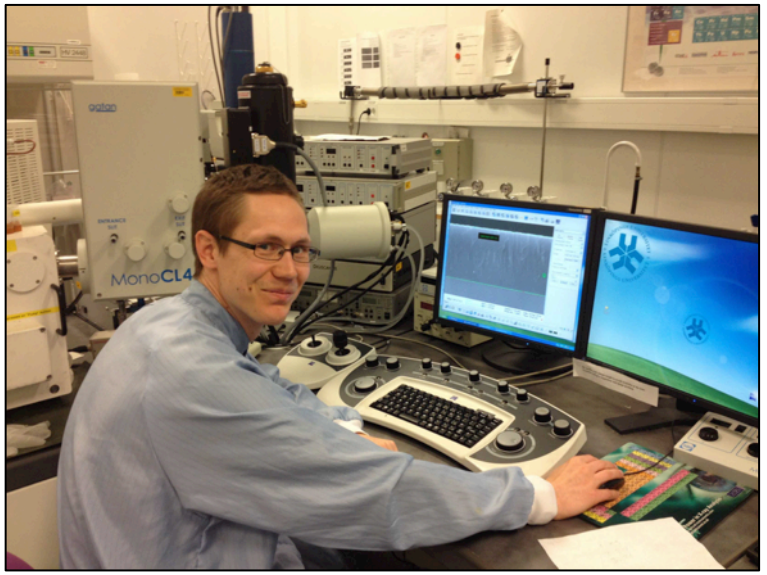
CC 800/9 deposition system, “Samson”

- 6 magnetrons with 8 x 50 cm^2 sputtering targets
- 4 x $^{10}B_4C$ and 1 x Al targets
- Specially designed sample holders for IN5-segment
- 3-fold substrate rotation
- 288 two-side coated blades per run ➔ >1 m^2 per run
- ~8 runs per segment
- 1 run of 1 μm $^{10}B_4C$ took 9 hours in total (~3h coating)



Quality control





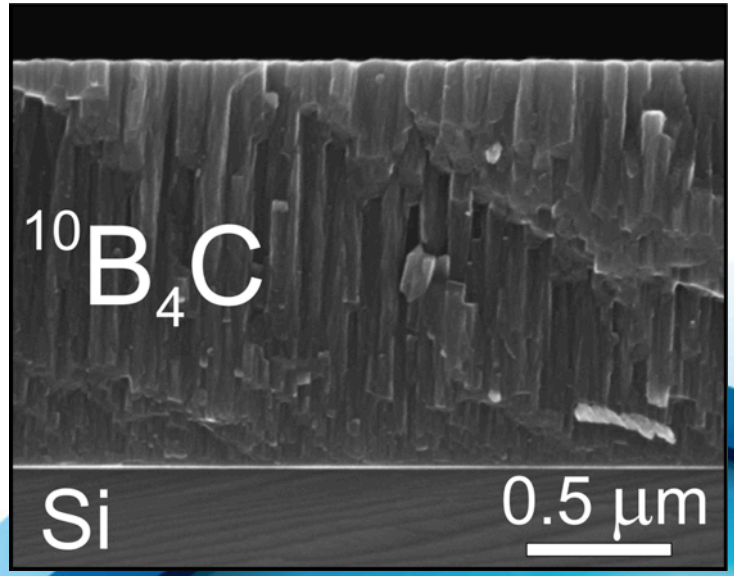
Si and pure Al blade from each run kept as reference

ERDA

- 79.5 at.% ^{10}B
- H + N + O = 0.5 at.%

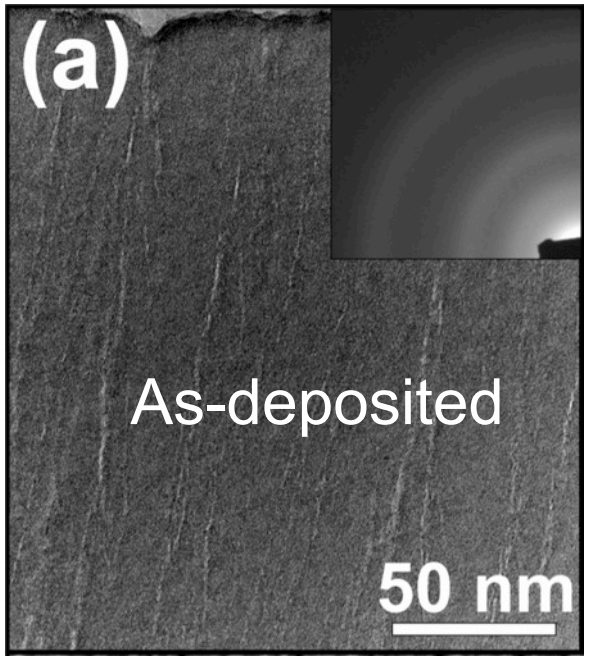
SEM

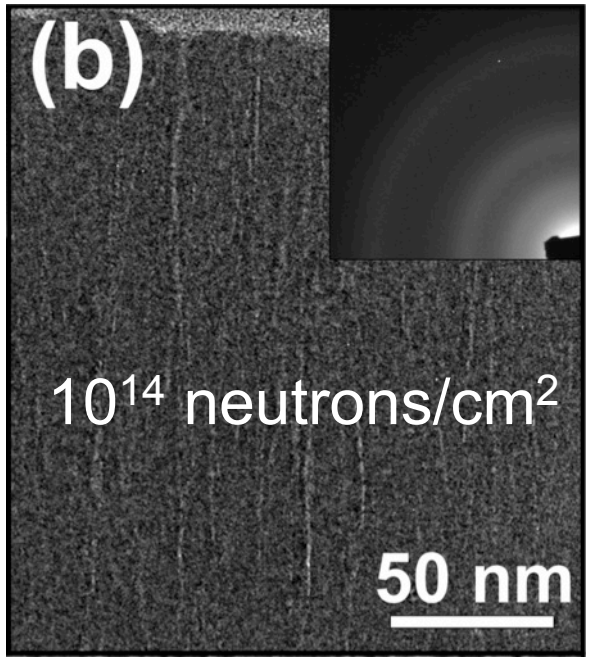
- Dense and columnar films
- Smooth surface
- Thickness variation ± 7 % (center to outer blade)



Radiation hardness

Radiation hardness (FRM II): **10^{14} neutrons/cm^2** on 1 μm $^{10}B_4C$ films
(800 years in a detector irradiated 24/7 with a flux of 10^8 n/cm^2/s)





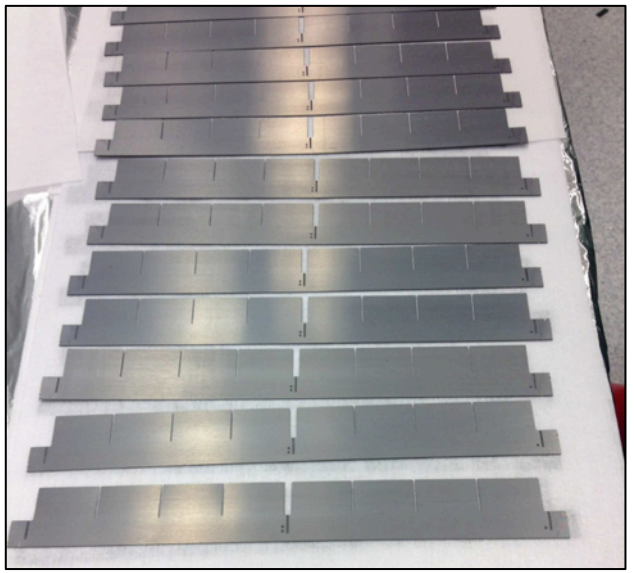
- **No influence** on adhesion, composition, morphology, structure, etc
- 0.000156 % of the ^{10}B atoms were consumed
  (Lifetime of ^{10}B atoms with 10^8 n/cm^2/s is >20 000 years ☺)
- First coatings made in 2010 show no sign of ageing after >4 years

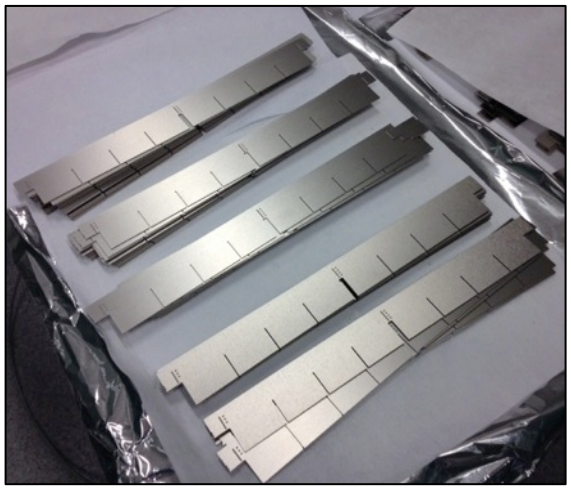
➔ Neutrons will not shorten the lifetime of the coatings!

ESS-LiU-FRM II collaboration
C. Höglund et al., submitted

Blades that were coated

- Coatings for 6 out of 8 segments:
  - 6 x 128 x 16 two-side coated blades
  - 6 x 128 x 2 one-side coated blades
- 2 different substrate types to lower α-background:
  - Al with 25 μm Ni-plating, “Ni-plated Al”
  - Al with ppb level of U and Th, “pure Al”
- Blades were numbered 1 - 4 to identify chamber position
- Coated blades are enough for in total 9 segments





Blade material	Coating thickness	Good blades
Al + Ni	0.75 μm	2356
Al + Ni	1.0 μm	2758
Al + Ni	1.5 μm	2508
Pure Al	0.75 μm	4690
Pure Al	1.0 μm	4752
Pure Al	1.5 μm	5030

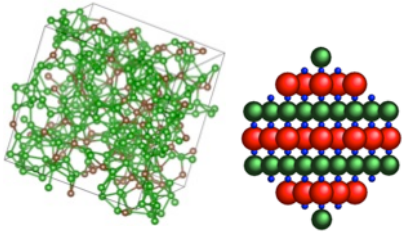
Packing and shipping

- Specially designed boxes
- In total 50 boxes!
- Shipped to Lund

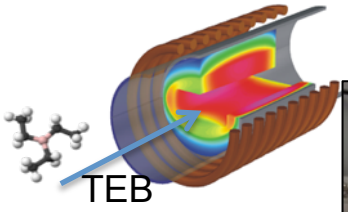
People who helped out with the Multi-grid prototypes in Linköping

Many collaborations

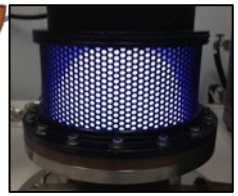
EUROPEAN SPALLATION SOURCE

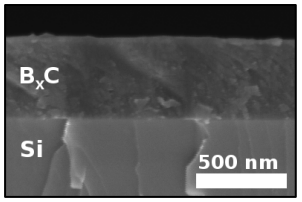


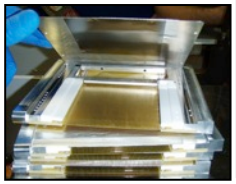
DFT-calculations

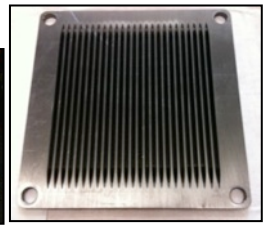


CVD

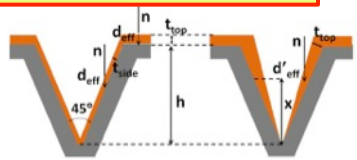


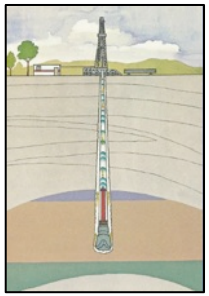






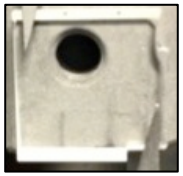
Increased efficiency



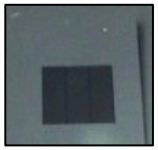


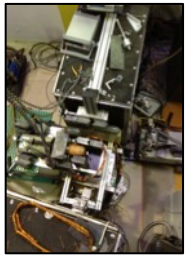
Other applications

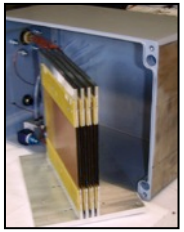
B. Alling et al., APL 98, 241911 (2011)
H. Pedersen et al., CVD 18, 221 (2012)
B. Guerard et al., Neutron News 23:4, 20 (2012)
A. Khaplanov et al., Proc. of ICANS XX (2012)
J. Correa et al., IEEE TNS, 60, 871 (2013)
B. Guerard et al., NIM A 720, 116 (2013)
I. Stefanescu et al., NIM A 727, 109 (2013)
A. Khaplanov et al., JINST 8, P10025 (2013)
I. Stefanescu et al., JINST 8, 12003 (2013)
F. Piscitelli et al., JINST 9, P03007 (2014)
A. Khaplanov et al., JPCS, accept. (2014)
F. Krejci et al., 14th ICAPTT proc, subm.
Etc.

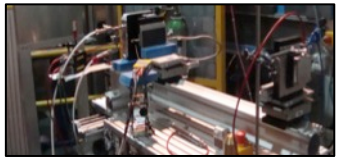


New substrates

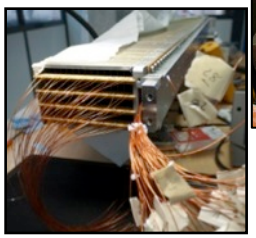




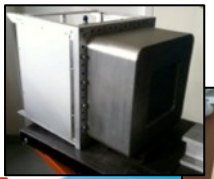


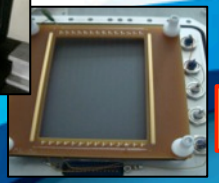


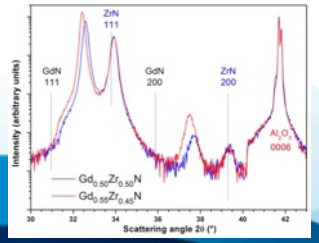
New detector types



Prototypes







New materials


Thank you
for your attention!
EUROPEAN
SPALLATION
SOURCE
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LINKÖPINGS UNIVERSITET
Linköping University

And remember that fingerprints in a vacuum system always shows up as bad adhesion…. ;-)