

Deliverable Number:		D 21.10
Deliverable Title:		Report on production parameter optimisation
	Delivery date:	[36]
	Leading beneficiary:	3
	Dissemination level:	PU
	Status:	Finished
	Authors:	K. Zeitelhack et al.

Project number:	283883
Project acronym:	NMI3-II
Project title:	Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy
Starting date:	1 <sup>st</sup> of February 2012
Duration:	48 months
Call identifier:	FP7-Infrastructures-2010
Funding scheme:	Combination of CP & CSA – Integrating Activities

#### **Description:**

The detection efficiency which can be achieved by using a single solid boron converter in a gaseous detector is limited by the fact that absorption length for thermal neutrons is larger than the ranges of the reaction products in boron, which are around ~3.5  $\mu$ m for the 1.47-MeV alpha particle and ~2 $\mu$ m for the 0.84-MeV Li ion. Because of the relatively low neutron detection efficiency of a single layer, up to 30 layers are necessary to achieve~ 50% detector efficiency for thermal neutrons. Consequently, one of the major challenges in developing gas detectors based on solid <sup>10</sup>B is the need to deposit uniform <sup>10</sup>B layers of ~ 1 $\mu$ m thickness over a large area.

TUM has evaluated the performance of  $B_4C$  coatings from different manufacturers, produced both by magnetron sputtering and electron beam evaporation. Homogeneity, transmission and detection efficiency measurements have been made using the TREFF cold neutron beam at FRM 2 and a prototype detector developed within task 21.2.3. Figure 1 shows some of the coatings evaluated. Figure 2 shows a compilation of detection efficiency data measured for various coatings for  $\lambda = 4.7A$ neutrons in comparison to GEANT4 simulation calculations.



Figure 1: Photographs of some of the coating evaluated at TUM.





As shown in Figure 2 simulation and measurement show good agreement and no significant differences have been found in coating performance.

The BNC team have also explored production techniques for solid boron converters. Both magnetic sputtering and electron beam evaporation techniques are available at the BNC campus. A suitable  ${}^{10}B_4C$  target was procured for the experimental magnetron sputtering machine of the Mirrortron company and this machine was used to deposit films. Details of the coating results and characterisation of three of the films is given below. Electron beam evaporation of  ${}^{10}B$  films has also been carried out. The films were deposited on twenty aluminium blades provided by the ILL for a prototype multigrid detector. Radiographic tests of the blades were satisfactory and adhesion seemed to be good as well. However, following the success of the ESS, ILL and Linkoping collaboration in producing high quality  ${}^{10}B$  films by magnetron sputtering, further development of film production at BNC has not been pursued.

Electron beam evaporation is usually more costly and thus magnetron sputtering is the preferred process. Linköping University is well set up to produce samples for R and D purposes in a timely fashion and thus Linköping has been chosen as the source of experimental coating for all work performed further on in this programme.

# **REPORT** on B<sub>4</sub>C layer production at BNC (results of the first trial deposition)

## **Procedure conditions :**

- DC- sputter
- -W = 2 kW
- Ar pressure 6 militorr/nitrogen
- Target substrat distance 7cm
- T = 51 hours

## Results

There were three plates mounted on the carrige of the LESKER made sputter machine. One bare Al-based alloy plate and one Al-alloy plate coated by Ni layer of sizes of 20x80x0.5 mm<sup>3</sup>. These plates were provided by ILL detector lab.

In addition a 2 m thick pure aluminium plate having 50x50 mm<sup>2</sup> surface was also mounted the carrige.

The thickness of the deposited layers were mesured at the reflectometer of the Neutron Spectroscopy Department of the Wigner Research Centre by using neutron transmission at wavelength  $\lambda$ = 4.28 Å.

All measurements were done by collecting more than 270000 events. The obtained results are given in the Table I.

#### Table I.

Substrate	Thickness of the	Effecive thickness		
	substrate:d(mm)	:	Remarks	Photos of the

		t <sub>в4C</sub> (μm)		prepared layers
Al(Ni coating)	0.498	0.14	The B₄C layer was	
			terribly pock-	Figure No.4
			marked (see the	
			photo)	
Al(no Ni coating)	0.492	1.12	The B₄C layer	
			seems to be	Figure No.5
			homogeneous	
Al(no Ni coating),	0.492	1.07		
repeted at				
2013.12.04				
Al (Hungarian)	1.965	1.96	The B₄C layer	
			seems to be	Figure No.6
			homogeneous	

Transmission of all substrates was also measured. The obtained results are seen in Table II.

### Table II.

substrate	transmission	
Al(Ni coating)	0.948	
Al(no Ni coating)	0.965	
Al (Hungarian)	0.987	

#### Surface profile analysis

Using ZYGO NEWVIEW 700 3D Optrical Surface Profilers Laser Interferometer a rather small surface parts (less than 0.2x0.2 mm<sup>2</sup>) chosen by chance on the surface of the deposited layers were studied. The corresponding features are shown below. It is clearly seen that the surface roughness follows as a replication that of the substrat, i.e. the stripes corresponds to the scrattches appearing during the procedure of production of the substrates.



Figure 3.  $B_4C$  layer thickness variation on Ni coated Al substrate provided by ILL. The rather large thickness variation is caused by the pock-marked coating.



Figure 4.  $B_4C$  layer thickness variation on Al substrate provided by ILL. The stripes are the inhibit property of the surface being observable even by unaided eyes.



Figure 5.  $B_4C$  layer thickness variation on Hungarian Al substrate. The blue spots appeared due to remainders of the cleaning fluid.



Figure 6. The photographic view of the  $B_4C$  layer deposited on the Ni-coated Al substrate.



Figure 7. The photographic view of the  $B_4C$  layer deposited on the bare Al substrate



Figure 8. The photographic view of the B<sub>4</sub>C layer deposited on the Hungarian Al substrate.

## **Concluluding remarks**

The valleys and hills seen on the Figures 3, 4 and 5 are nothing to do with the thickness variation of the  $B_4C$  layer. They are simply the repliaction of the surface relief of the substrate formed during its production.

The thicknesses given in the Table I are the effective ones and thus they are not applicable for the calculation of the compactness of the  $B_4C$  layer.

Figure 6 demonstrates the presence of rather large spots not coated by  $B_4C$ . The  $B_4C$  coating was not formed when removing the substrate from the carrige. Obviously the  $B_4C$  particles were not able to stick to the substrate when thet reached it.

In Figure 7 it can be seen that the  $B_4C$  layer coated the substrate much more homogeneously. The only exceptions are the upper and lower edges of the substrate where some badly coated stripes are seen. The reason for this affect is not yet clear.

The homogenity of the layer deposited on the 2 mm thick pure Al substrate shown in Figure 8 is the best. The photo looks so dark beceause the surface of the layer was so smooth that it was reflecting anything in its vicinity. In order to get rid of any reflected shapes the plate was surounded by a black screen. The reflection of the black screen gave a black view of the deposited layer.