Neutron irradiation ageing study of AluBor4 and LCB, Nov.-Dec. 2015 irradiations

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Keywords

Irradiation, boron, neutron, ageing

1 Introduction

The macroscopic behaviour under neutron flux of two boron containing materials has been investigated at ILL. The irradiations took place during the last cycle of 2015. After their radioactive cool down, they have been recovered from their irradiation shuttle and have been analysed.

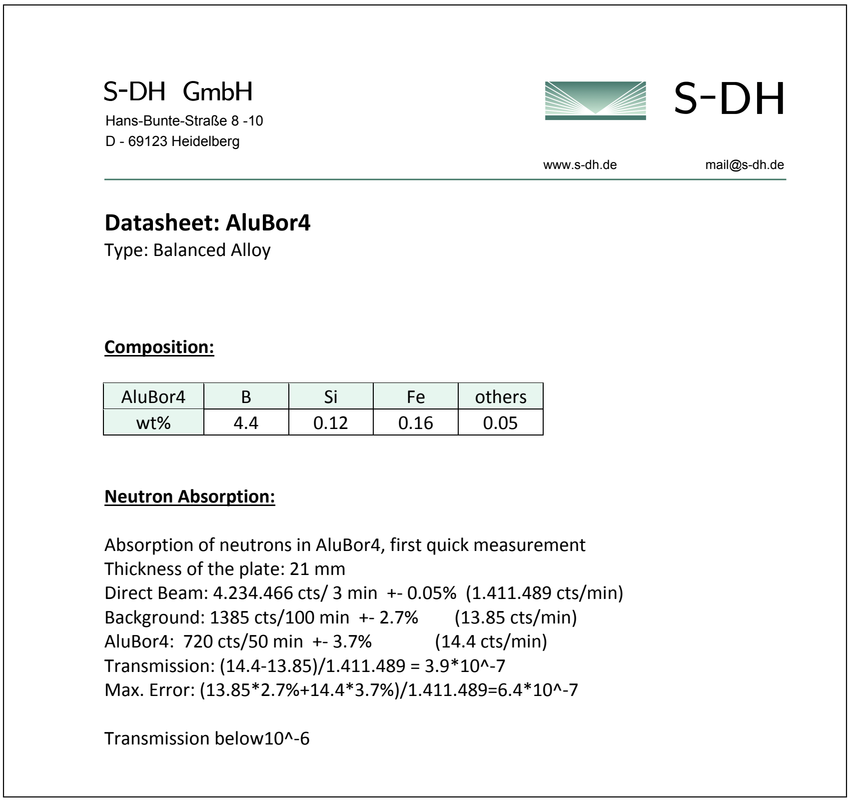
2 Materials and methods

2.1 Studied Materials

Two materials have been tested: a borated aluminium called AluBor4, and a mixture of B_4C and epoxy called LCB. All the samples were provided by S-DH and followed the geometrical specification linked to the different irradiation conditions foreseen.

AluBor4 is composed, in mass, of 95.27 % of aluminium and 4.4 % of natural boron. The rest of the mass being silicon, iron and trace elements. More details are gather in the data sheet provided by S-DH and reported in Fig. 1. The density is similar to the one of pure aluminium, which is around 2.7 g/cm^3. Neutron absorption data, extrapolated from the Fig. 1, are gathered in Table 1. The nuclear heating (in W/cm^3) is computed in the case of a T4 irradiation that yields an average flux around 2×10^{13} n/cm^2/s. Two geometries of samples have been tested: $10 \times 10 \times 1$ mm^3 and $14.5 \times 14.5 \times 10$ mm^3. The latter samples have been provided with $m = 3$ super-mirror coating.

LCB is composed, in mass, of 60 % of B_4C and 40 % of epoxy ($C_{15}H_{16}O_2C_{10}H_{22}N_2$). More details are gather in the data sheet provided by S-DH and reported in Fig. 2.



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Datasheet: AluBor4
Type: Balanced Alloy

Composition:

AluBor4	B	Si	Fe	others
wt%	4.4	0.12	0.16	0.05

Neutron Absorption:

Absorption of neutrons in AluBor4, first quick measurement
Thickness of the plate: 21 mm
Direct Beam: 4.234.466 cts/ 3 min +- 0.05% (1.411.489 cts/min)
Background: 1385 cts/100 min +- 2.7% (13.85 cts/min)
AluBor4: 720 cts/50 min +- 3.7% (14.4 cts/min)
Transmission: (14.4-13.85)/1.411.489 = 3.9*10^-7
Max. Error: (13.85*2.7%+14.4*3.7%)/1.411.489=6.4*10^-7

Transmission below10^-6

Figure 1: AluBor4 data sheet provided by S-DH.

g/mol	Element	wt %	g/cm^3	mol/cm^3	at/cm^3	$\Sigma_{(n,\alpha)}$ (cm^{-1})	Q(W/cm^3) in T4
27.0	Al	95.27	2.57	9.53×10^{-2}	5.74×10^{22}		
10.8	B	4.4	0.119	1.10×10^{-2}	6.62×10^{21}	5.08	41
28.1	Si	0.12	3.24×10^{-3}	1.15×10^{-4}	6.65×10^{19}		
55.8	Fe	0.16	4.32×10^{-3}	7.74×10^{-5}	4.66×10^{19}		
	Others	0.05	1.35×10^{-3}				

Table 1: Some characteristics about AlB4.

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Radiation Absorber LCB	Date: Nov 03, 2011	Harald Haese, haese@s-dh.de

Keywords : Oncology, Treatment, Multileaf Collimator, shielding of neutron radiation, cover of motion controller (FPGA), cooling/thermal tranportation.

	1. Description
	A high energy linear accelerator produces x-ray and neutron radiation. Neutron radiation produces errors in the electronic devices like motion controller of the leafs-blades. We developed a special shielding (LCB) to cover all the electronic devices and avoid errors from neutron radiation. The LCB has a high thermal conductivity and with the big surface a high cooling rate for the electronic devices. For many years, LCB from S-DH are used for MLC's by Siemens.
	2. Specification
2.1	Color: Black Density: 1.74 g/cm³ Content: Boron-Carbid (B_4C) : 60 % wt $C_{15}H_{16}O_2$ $C_{10}H_{22}N_2$: 40 % wt
2.2	Shape: LCB can produced in almost any shape and volume
2.3	
2.4	Production Capacity last 3 years: 10.000 piece per year

Figure 2: LCB data sheet provided by S-DH.

Extrapolated neutron absorption data are gathered in Table 2. The nuclear heating (in W/cm^3) is computed in the case of a H9-Tomograph irradiation that yields an average flux around 3×10^9 n/cm^2/s. Two geometries of samples have been tested: $10 \times 10 \times 0.4$ mm^3 and $14.5 \times 14.5 \times 10$ mm^3.

2.2 Neutron irradiation methods

2.2.1 Homogeneous irradiation

Homogeneous irradiation was conducted in the T4 tube of the reactor on AluBor4 samples with dimensions around $10 \times 10 \times 1$ mm^3. It lasted 3 days with a reactor power around 43 MW. This equipment provides a thermalised neutron flux that yields an unperturbed intensity of 2×10^{13} n/cm^2/s, when the reactor is operated at 50 MW. The sample holder

Element	wt %	g/cm^3	mol/cm^3	at/cm^3	$\Sigma_{(n,\alpha)th}$ (cm^{-1})	Q(W/cm^3) in H9
B_4C	60	1.044	$1.9{\times}10^{-2}$	$1.1\ {\times}10^{22}$		
$C_{15}H_{16}O_2C_{10}H_{22}N_2$	40	0.696	1.7×10^{-3}	1.1×10^{21}		
B				4.6×10^{22}	34.9	0.042
H				4.0×10^{22}		
C				3.8×10^{22}		
N				2.1×10^{21}		
O				2.1×10^{21}		

Table 2: Some characteristics about LCB.

used for that experiment is made so that samples are in contact with the light water. The natural water flow maintains the sample temperature below 100 °C. More details on that experiment can be found in the publication from Boffy *et al.* [1]. The effective flux during the experiment, also known as perturbed flux, has been measured with the neutron activation analysis of zirconium foils embedded in the sample holder. This is necessary as the *in situ* flux is highly dependent upon the absorption properties of the samples and the reactor power. It is worth mentioning that the average flux in the sample is 72 % of the one at its surface. This is due to the self-shielding brought by the high absorption cross section of the boron-10 nuclei.
LCB could not be tested in that configuration due to the risk of deterioration of epoxy under irradiation, and a consequent release of foreign materials in the light water pool of the reactor. Polymers, in general, are known to degrade quickly under irradiation.

The aim of that experiment was to determine if the density of AluBor4 was modified by thermal neutron irradiation.

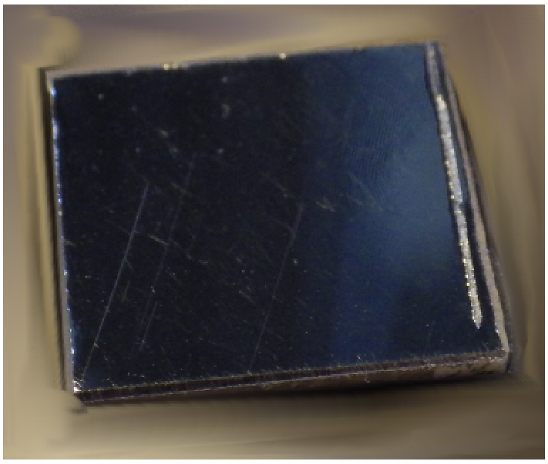
2.2.2 Grazing incidence irradiation

Another sample holder developed recently at ILL allows to create, in T4, irradiation conditions combining: high thermal flux, grazing incidence, and a sample temperature below 100 °C. Details on that sample holder will be published soon. Neutron flux intensity on sample surface was measured with zirconium foils. For the same reasons as for previous set-up, solely AluBor4 was characterized with that technique.

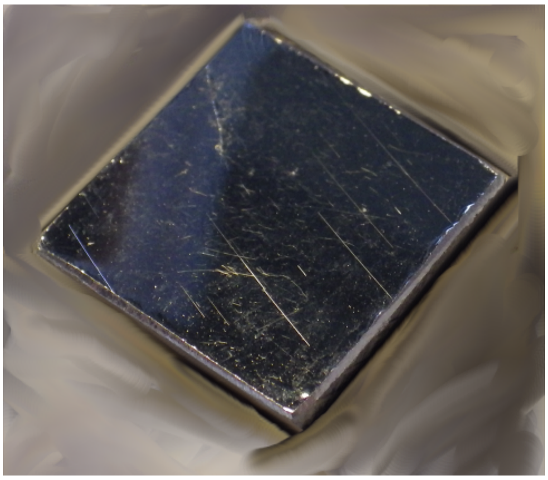
The aim of that experiment was to determine if the super-mirror coating was damaged by the thermal neutron irradiation. Pictures of the pristine samples are reported in Fig. 3. The experiment lasted 17 days and the reactor was running at 43 MW

2.2.3 Frontal irradiation

LCB samples have been irradiated in the Neutrograph test instrument [2]. That experiment aimed primary to detect by eye inspection an degradation of the samples. The Neutrograph is fed by the H9 thermal neutron beam which has a 3×10^9 n/cm^2/s thermal flux plus a fast neutron component of about 0.1 %. For both sizes of sample, $10 \times 10 \times 0.4$ mm^3 and $14.5 \times 14.5 \times 10$ mm^3, the specimens were oriented so that the beam was hitting per-



(a) First sample



(b) Second sample

Figure 3: AluBor4 samples coated with $m = 3$ super-mirror. Only the shown surface, which is the one coated, will be irradiated by thermal neutrons. Dimensions: 14.5×14.5 mm^2.

pendicularly their larger side. Considering the thickness of the two sample kinds, the beam attenuation at opposite surface of irradiation, for 1.8 Å neutrons, was 75 % and 97 %, respectively. The total beam fluence accumulated was 3.6×10^{15} n/cm^2. For the thinner samples, due to the self-shielding phenomenon, the average fluence over the whole sample was 1.9×10^{15} n/cm^2.

The density of thinner samples has been measured before and after irradiation to have a general idea of macroscopic behaviour of LCB under flux.

2.3 Characterization technique

To monitor the density evolution of the thin samples with received fluence, we have employed the hydrostatic weighing method. A density determination kit has been installed on a Mettler-Toledo AE240 weighing scale. Both materials have been measured with that technique. Even though the flux was very heterogeneous inside LCB samples, the rationale was to have a general idea of the macroscopic behaviour of that material.

For the 10 mm thick samples, the characterization was an eye observation of the surface aspect.

3 Results and discussion

3.1 AluBor4

3.1.1 Density variation

Once recovered and counted with a germanium gamma-detector, the zirconium foil yielded that the perturbed flux at sample surface was 8.94×10^{12} n/cm^2/s. This corresponds to

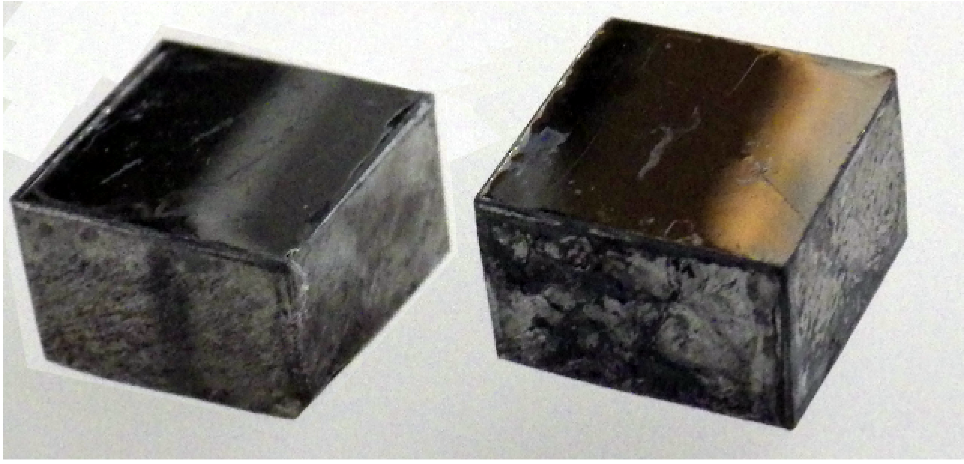


Figure 4: AluBor4 samples coated with $m = 3$ super-mirror after irradiation. Fluence on the irradiated surface: 7.70×10^{17} n/cm^2.

an accumulated fluence on sample surface of 2.31×10^{18} n/cm^2 and an average value in the whole sample, due to the self-shielding, of 1.66×10^{18} n/cm^2.
The pristine density was measured to be 2.681 ± 0.007 g/cm^3 and the irradiated one 2.671 ± 0.004 g/cm^3. This gives a density variation of $-0.37\% \pm 0.29$ %. Considering the uncertainty on that value, one cannot truly infer the bulk behaviour of AluBor4 under thermal neutron irradiation. However, one could think that the material is slightly swelling due to the (n,α) reactions. Though, considering the fluence it was exposed to, the change in density appears smaller than the one of N-BK7 or S-BSL7.

These results call for further irradiation experiments with different fluences, say from 0.1 to 2.5×10^{13} n/cm^2, and with an improved accuracy of the measurement procedure.

3.1.2 Super-mirror aspect after irradiation

The activation analysis of a zirconium foil informed us that the flux on the sample was around 5.24×10^{11} n/cm^2/s which yields, for 17 days of irradiation, a fluence of 7.70×10^{17} n/cm^2. Figure 4 shows the samples once recovered from the irradiation shuttle. The super-mirror did not show major degradation. One can see traces of aluminium oxidation on the mirror which could due to the fact that the surface is stuck to an aluminium part. The super-mirror does not detach from the AluBor4. Together with these samples, S-BSL7 and N-BK7 had been put in the shuttle. These latter did not show degradation either. N-ZK7 and Borofloat have been tested with that set-up at a comparable fluence and have shown multiple fractures on the irradiated surface.

3.2 LCB

3.2.1 Density evolution for homogeneous irradiation

The pristine density was measured to be 1.6411 ± 0.0008 g/cm^3 and the irradiated one 1.621 ± 0.003 g/cm^3. Let us recall that the average fluence, taking into account the self-shielding, was 1.93×10^{15} n/cm^2. This gives a density variation of $-1.24\% \pm 0.18$ %, which means that the sample is swelling. The magnitude of variation could be compared to the shrinking of Borofloat and N-ZK7 that achieve such a value around 1.5×10^{17} n/cm^2 and 4×10^{17} n/cm^2, respectively. This result shows that LCB is reactive under irradiation, already at low fluence.

Concerning the aspect of the thick sample, no modification could be seen by eye inspection.

Even though the accumulated fluence was relatively low compared to the one engaged in a guide, the LCB density appears to react under irradiation. This can be caused of by the $B^{10}(n,\alpha)$ reactions but also by the γ rays interactions with the network. If one only considers the (n,α) reactions, the energy deposited in the material, throughout the experiment, is around 16.4 MGy, with a rate of 92 kGy/h. Such values are similar to the ones found in the studies from Devanne *et al.* [3, 4], where they show that gamma irradiation induces a modification of the epoxy network. Hence, the density measurements highlighted the necessity to test LCB under grazing incidence irradiation, and with a mirror coating, before any large scale deployment as mirror substrate.

4 Conclusion

These experiments have shown that AluBor4 appears to be relatively stable under homogeneous thermal-neutron irradiation. Similarly, the adhesion of Ni-Ti super-mirror seems to resist to irradiation, at least below 100 °C. These results call for further experiment to validate the use of super-mirror coated AluBor4 in high radiation areas.

Macroscopic measurements have shown the reactivity of LCB to thermal neutron irradiation: its density decreases of $1.24\% \pm 0.18$ % at a fluence of 1.93×10^{15} n/cm^2. Therefore, the next step would be to test the material in grazing incidence conditions and with a mirror coating to see if it has an impact on reflectivity performances.

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