# Performance Evaluation of Ferroboron Shielding Material after Irradiation in FBTR

Bijay Kumar Ojha<sup>1</sup>, V. Anandaraj<sup>1</sup>, M. Padalakshmi<sup>1</sup>, V.V. Jayaraj<sup>1</sup>, C. Padmaprabu<sup>1</sup>, Ran Vijay Kumar<sup>1</sup>, A. Vijayaragavan<sup>1</sup>, C.N. Venkiteswaran<sup>1</sup>, V. Karthik<sup>1</sup>, T. Johny<sup>1</sup>, Divakar R.<sup>1</sup>, S. Murugan<sup>1</sup> and Jojo Joseph<sup>1</sup>

<sup>1</sup>Indira Gandhi Centre for Atomic Research, Kalpakkam, India

E-mail contact of main author: bijay@igcar.gov.in

**Abstract**. Ferro boron has been identified as a candidate material for in–vessel radiation shielding application in future Fast Breeder Reactors (FBRs) in India that can result in significant cost savings. Out-of-pile physical and chemical characterization studies have established its neutron shielding property and long-term compatibility with 304L SS clad under sodium at the operating temperatures. An irradiation experiment was designed and carried out with the aim of establishing in-reactor performance of ferroboron shielding material over a target life-time of 60 years. This paper reports on post-irradiation examinations carried out on the experimental capsule. The results show that slumping of stack and helium release are not life limiting for deployment of ferroboron for in-core shielding in fast reactors.

Key Words: Ferroboron, neutron shielding, helium release, chemical interaction

#### 1. Introduction

In-reactor shielding around the core of a sodium-cooled fast reactor (SFR) is essential for significantly reducing leakage of high-energy neutrons from the core, thus reducing the activation of the primary coolant and maintaining acceptable dose levels in the steam generator area. The Prototype Fast Breeder Reactor (PFBR), which is in an advanced stage of construction at Kalpakkam, India, employs nine rows of shielding sub-assemblies with stainless steel and boron carbide as the shielding materials. Since the shielding subassemblies constitute a significant fraction of the core volume, any efforts towards reducing their number and replacing with a lower cost material, without affecting radiological protection, would lead to a significant capital cost reduction. A number of alternate shielding materials have been considered towards this objective.

For the Indian fast reactor programme, Keshavamurthy *et al.* [1] have experimentally investigated and found the shielding effectiveness of ferroboron to be good in spite of the lower boron content of up to 15% as compared to 78% in boron carbide. Further, Sunil Kumar *et al.* [2], on the basis of two-dimensional transport calculations, have concluded that this low-cost material is capable of satisfying radiological requirements on par with the shielding used in PFBR, with a marginal reduction in the activation of primary sodium. Out-of-pile studies on metallurgical interaction with cladding, chemical compatibility with sodium, etc. have been examined and found satisfactory [3, 4] for temperatures up to 600°C over the reference life-time of sixty years. However, for deployment in the reactor, performance issues such as pressure build up inside the cladding due to helium gas release, compaction of ferroboron stack and the metallurgical compatibility with the cladding material have to be evaluated over the planned life time of the reactor. To confirm the suitability of the material with respect to in-core performance, an accelerated test irradiation was carried out in Fast Breeder Test Reactor (FBTR) [3]. In this paper, results of post-irradiation examination (PIE)

of ferroboron after irradiating it to a fluence corresponding to a reactor life-time of sixty years is presented.

### 2. Experimental

### 2.1. Irradiation Experiment

Fig. 1 shows the sketch of the ferroboron irradiation capsule. The irradiation capsule was made up of two concentric stainless steel tubes with the inner tube having five partitions of nominal height 100mm each, in which the Ferro boron powder was packed to a density of 4.2 g/cc under pure argon atmosphere. Table I gives the chemical composition, phases present and relative density of Ferroboron filled in the sub-capsule. X-ray diffraction profile of the powder indicated that the major phase is FeB. Fe<sub>2</sub>B phase is present in very small volume fraction which dissolves back to FeB phase at high temperature. Table II gives the fabrication data of the ferroboron sub-capsules. The inner tube (clad) material is 304L stainless steel. The outer tube serves as secondary containment for preventing the ferroboron powder into the hot sodium in the unlikely event of breaching of inner tube. The inner tube has a nominal wall thickness of 1 mm. To facilitate puncturing of the capsule for gas extraction, wall thickness is reduced to 0.5 mm, locally at the central region of each capsule. The annular gap between the two tubes is filled with high purity helium for better heat dissipation. The central three partitions of the irradiation capsule are in line with the driver mixed carbide fuel column of FBTR. An intermediate plug was welded in the argon atmosphere to the outer tube with provision to evacuate and fill the annular gap with helium. The ferroboron irradiation capsule was locked in an experimental subassembly and irradiation was carried out at location (0407) of 4<sup>th</sup> ring in FBTR core for ~ 66 effective full power days to a fluence of 3.8 x  $10^{22}$  and a maximum displacement damage of 2.96 dpa close to core mid-plane, corresponding approximately to the centre of the 3rd sub-capsule. The FBTR inlet and outlet Sodium temperatures were 390°C and 475°C respectively.

Element	Boron	Silicon	Aluminium	Carbon	Sulphur	Phosphorus	Iron
Wt %	15.42	0.89	0.169	0.29	0.0061	0.0046	Bal.
Phases present	FeB & Fe <sub>2</sub> B						
Relative density	63 % of T.D (T.D – 6.7 g/cc)						

#### TABLE II FABRICATION DATA OF FERROBORON SUB-CAPSULE

Capsule No.	Internal	Quantity of	Powder fill	Density
(from the top of irradiation capsule)	diameter 'mm'	FeB filled 'g'	height 'mm'	g/cc
1	12.7	48.9	100.03	4.27
2	12.06	47.1	100.04	4.12
3	12.03	49.5	100.12	4.35
4	12.06	48.3	100.15	4.22
5	12.02	46.8	100.12	4.12

#### 2.2. Post Irradiation Examinations

After discharge from FBTR at the end of the irradiation campaign, the experimental subassembly containing the ferroboron capsule was received into the hot-cells of Radio metallurgy Laboratory (RML) for PIE [5]. The experimental subassembly was subjected to sodium cleaning to thoroughly remove sodium adhering to external and internal surfaces. The irradiation capsule was unlocked from the subassembly using specially designed and fabricated tools. Special fixtures were also designed and fabricated for lowering the irradiation capsule into the neutron radiography port through hot cell. Apart from neutron radiography (NR), Helium release measurements and metallographic examination of the clad section has been carried out.



Fig. 1. Sketch of ferroboron irradiation capsule.

# 2.2.1 Neutron Radiography

Neutron radiography (NR) was carried out in KAMINI reactor using indirect imaging technique [6]. The radiography of the capsule was carried out in both unirradiated and irradiated conditions at power level of 20 kW for an exposure time of 20 minutes. The radiography of irradiated capsule was carried out with multiple exposures so as to cover as many of the five sub-capsules as feasible. The images were taken with capsule in the two orientations (upright and inverted) to assess whether the ferroboron retained its pre-irradiation free flowing nature, or was compacted into a hard mass. Image processing routines such as edge detection and contrast enhancement through gamma correction were applied to the digitized images to quantify the dimensional details with an accuracy of  $\pm 0.2$  mm.

### 2.2.2 Helium release measurements

A custom built puncturing device was fabricated to extract the helium gas produced inside each sub-capsule by  $(n, \alpha)$  reaction. Fig. 2 shows the puncturing system installed inside the hot cell. Chamber was designed with a low dead volume of 1.6 cc to maximise the pressure in the sample vial for analysis by gas chromatography. The gas sample extracted was analysed by gas chromatograph using nitrogen as carrier gas.



Fig.2: Helium gas extraction set-up inside the hot cell

The irradiation capsule is inserted to the puncture chamber horizontally. The chamber encloses approximately the length of one sub-capsule (120 mm in length). Since the puncturing device can accommodate only one sub-capsule at a time, puncturing of each sub-capsule was done sequentially. A low-light capable digital camera was used to identify the puncturing locations in the capsule where the thickness of the clad wall is 0.5mm. The chamber volume was precisely measured using a calibration volume.

Before puncturing, the chamber was evacuated to a pressure of 0.1 Torr using a dry vacuum pump. Capacitance transducer having a precision of 0.1 Torr was used to measure the gas pressure in the chamber. Gas extraction was carried out on central sub-capsule and two sub-capsules on either side of central sub capsule. Each sub-capsule was punctured after ensuring satisfactory leak-tightness of the gas collection system. The extracted gas sample was collected in a 1.0 cc stainless steel sample vial. After puncturing, the sample vial was retained in the chamber for half an hour for homogenization.

Collected gas was analysed in a gas chromatograph using nitrogen as a carrier gas to identify and quantify the gases present in the capsule. Packed column of molecular sieve 5 Å was used as the stationary phase in the gas chromatograph. The sample eluted from the column was

detected using a thermal conductivity detector (TCD). The chromatograph was calibrated using a standard gas mixture of Helium and Argon. The column size, carrier gas flow-rate, temperature of the column and the detector were optimized for obtaining good separation of the peaks of the sample constituents. Since the vial pressures were sub-ambient, the sample loop was evacuated to a pressure of 0.1 Torr before injecting the gas sample to be analysed. The sample injection pressure was noted using a capacitance manometer after feeding the gas sample into the sample injection loop of gas chromatograph.

The peak areas of the sample constituents were obtained from the chromatogram. The chromatogram obtained during the analysis of gas extracted from one of the capsules is shown in Fig. 3. The peak areas of each species were converted to number of moles in the 1cc sample from the respective calibration curve. These values were multiplied by the volume correction factor to obtain the corresponding values in the capsule. The volume of the gases in the capsule and the partial pressures were deduced using the ideal gas law equation. The percentage of helium released from the ferroboron matrix to the capsule was calculated based on the theoretical volume of gases generated due to  $(n, \alpha)$  reaction. The pressure inside the sub-capsule was calculated by measuring the dead volume inside the sub-capsule after puncturing and the total partial pressures was measured from gas chromatography analysis.



# **Retention time (minutes)**

*Fig 3: Chromatogram obtained during the analysis of gas extracted from one of the ferroboron subcapsules.* 

#### 3. Results and Discussion

The neutron radiographs of the ferroboron capsule in the pre-irradiation and post irradiation conditions are shown in Fig. 4 and 5 respectively. The ferroboron stacks in the unirradiated and irradiated conditions were compared (Table III). Due to dimensional limitations on the pre-irradiation exposures, pre-irradiation measurements were made only on the 1<sup>st</sup> and 3<sup>rd</sup> sub-capsule.

Sub-capsule No.	Ferro-boron Pre-irradiation (mm)	Slumping (mm)	
1	100.6	100.1	0.5
3	100.0	99.7	0.3*

Note: Gap was observed within the stack as shown in the radiograph



Fig. 4. Pre-irradiation neutron radiograph (a) raw image (b) processed image



Fig. 5. Post-irradiation neutron radiograph (a) raw image (b) processed image

It can be seen from the radiographs that the initial gap between the top of the stack and the end plug in the unirradiated condition was found to shift to another location of the ferroboron stack in the irradiated condition. This suggests that the ferroboron stack above the gap could have got compacted. However the gaps were irregular and discontinuous. It can be concluded from radiographic images that the slumping of the ferroboron stack on accelerated irradiation equivalent to the target reactor life-time of 60 years is limited to a maximum of 1mm in 100 mm pre-irradiation stack height.

Puncture tests of the sub-capsules indicate that the maximum pressure due to helium release is 0.16 MPa. Table IV gives the pressure in the three sub-capsules subjected to puncture tests and the helium partial pressure. It can be seen that the internal pressure in the capsules is very nominal. Hence there is no concern with respect to the pressure build up due to release of helium. Gas chromatography analysis of the sample gas revealed that the constituents are Helium and Argon. Argon is the pre-fill gas. Partial pressures of each constituent was estimated from the peak areas obtained from the chromatogram by correlating with calibration curves obtained using analysis of known composition of gas mixtures and the respective peak areas. It can be seen from the table that the 2<sup>nd</sup> capsule has relatively higher helium release than the other two sub-capsules. This is attributed to the relatively higher sodium temperatures towards the top of the irradiation capsule facilitating higher gas release.

The theoretical yield of helium atoms after the irradiation is  $2.24 \times 10^{20}$  per cm<sup>3</sup> of ferroboron for the middle capsule. Based on the above theoretical yield value and the measured helium content in the capsule, the helium release works out to around 3.3 %.

Sub-capsule	Estimated values		Post irradiation examination results					
location	$^{10}B$	Helium	Void	Gas	Helium	No of He	Не	
(from the top	depletion	yield	volume	pressure	partial	atoms	release	
of irradiation	%	(No of	in the	in the	pressure	released	fraction	
capsule)	70	atoms/cc	capsule	capsule	(torr)	per cm <sup>3</sup>	%	
		of FeB)	(cc)	(MPa)		of FeB		
$2^{nd}$	2.9	2.07E+20	3.5	0.16	762	7.82E+18	3.77	
3 <sup>rd</sup>	3.0	2.24E+20	3.7	0.17	725.4	7.48E+18	3.34	
4 <sup>th</sup>	2.6	1.91E+20	4.0	0.10	618.1	6.7 E+18	2.57	

# TABLE IV: HELIUM RELEASE & PRESSURE BUILD UP

### 4. Discussion

Ferroboron is a promising shielding material which can result in substantial cost savings in fast breeder reactors. Out of pile characterization such as thermophysical properties and high temperature metallurgical compatibility with the clad and interaction with sodium had indicated excellent compatibility. An accelerated test irradiation of this material to evaluate the irradiation performance was conceived to obtain the confidence and planning further irradiation experiments based on the feedback from the present work. Though the extrapolation of the results from accelerated tests is challenging because of the kinetics, damage rate etc and its influence on the various parameters such as the gas release, the initial results from this irradiation experiment and performance evaluation has provided encouraging results to pursue irradiation experiments with this material for obtaining data at higher duration of irradiation.

# 5. Conclusions

Post Irradiation Examinations have provided valuable data on the irradiation behavior of the ferroboron shielding material. Neutron radiographs indicated that the slumping of the ferroboron stack is limited to a maximum of 0.5 mm in a 100 mm pre irradiation stack height. The maximum pressure released due to  $B^{10}$  (n,  $\alpha$ ) Li<sup>7</sup> reaction was estimated to be 0.16 MPa, low enough to produce any significant stress on the clad. PIE of the ferroboron capsule irradiated in FBTR has indicated that slumping of stack and helium release are not life limiting for deployment in fast reactors. Microstructural examination of the clad sections is underway to evaluate the chemical interaction.

# 6. References

- [1] KESHAVAMURTHY, R. S. *et al.*, "Experimental Measurements of Neutron Attenuation in the Advanced Shield Material Ferro Boron in KAMINI Reactor", Energy Procedia, 7 (2011), 273.SUNIL KUMAR, D. *et al.*, "A feasibility study of ferro-boron as in-core shield material in fast breeder reactors", Nucl. Engg. Design, B240 (2010), 2972.
- [2] KESHAVAMURTHY, R.S. et al Proceedings of the International Conference on Fast Reactors and Related Fuel Cycles (FR 13), Paris, France, 4–7 March 2013, International

Atomic Energy Agency, STI/PUB/1665 (ISBN:978-92-0-104114-2), Paper No. IAEA-CN-199/231, May 2015.

- [3] RAI, A. K. *et al.*, "Investigation of high temperature phase stability, thermal properties and evaluation of metallurgical compatibility with 304L stainless steel, of indigenously developed ferroboron alternate shielding material for fast reactor applications", Nuclear Engg. Design, **241** (2011), 2787.
- [4] RAJ, B. *et al.*, Post Irradiation Examination of mixed (Pu, U)C fuels irradiated in the Fast Breeder Reactor, IAEA proceedings IAEA-TECDOC-1039 on "Influence of high dose irradiation on core structural and fuel materials in advanced reactor", 47, 1998.
- [5] KASIVISWANATHAN, K. V., VENKATRAMAN, B. AND RAJ, B., "Neutron radiographic facilities available with KAMINI", Proc. of 6<sup>th</sup> World Conference on Neutron Radiography, Edited by Shigenori Fujine, Hisao Kobayashi and Keiji Kanda, Gordon and Breach Science Publishers, Japan, 117-120, 1999.