Investigation of Irradiation Induced Phase Formation at Ferroboron and SS 304L Clad Material Interface

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1. Introduction

Ferroboron has been identified as a candidate material for in–vessel radiation shielding application in future Fast Breeder Reactors (FBRs) in India considering 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 accelerated irradiation experiment has been carried out with the aim of establishing in-reactor performance of ferroboron shielding material for a target life-time of 60 years in a commercial FBR. Slumping of the Ferroboron within the sub-capsule, generation of Helium gas and ferroboron- clad chemical interaction have been evaluated as a part of Post Irradiation Examinations.

2. Irradiation of Ferroboron capsule in FBTR

The irradiation capsule was made up of five sub-capsules each having 100mm length with Ferroboron powder packed to a density of 4.2 g/cc under argon atmosphere. Irradiation capsule had another outer sleeve serving as a secondary containment for the FeB powder. The displacement damage undergone by the five sub-capsules was in the range of 0.50-2.96 dpa. The maximum displacement damage of 2.96 dpa corresponds to the life time fluence undergone in a commercial fast breeder reactor.

3. Experimental methods

Post Irradiation Examination was carried out in RML hot cells for evaluating the effects of ferroboron on SS 304L cladding material after irradiation. Before taking up the microstructural examinations on the clad to evaluate chemical interaction, the capsule was subjected to Neutron radiography to evaluate the slumping of FeB powder column and puncture tests to quantify the helium release due to (n,α) reaction. 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 10B (n, α) 7Li reaction was estimated to be 0.16 MPa, low enough to produce any significant stress on the clad. Thus Neutron radiography and He release measurements did not indicate any concern on the performance of FeB as a shielding material.

The clad sections from different sub-capsules were metallographically prepared inside the hot cell up to 1 micrometer diamond finish followed by etching for optical microscopy. The samples were examined using an optical microscope inside the hot cell.

The clad section, which indicated maximum clad damage, was further investigated using Scanning Electron Microscopy to evaluate the nature of interaction and formation of phases due to the chemical interaction. On the same sample, phase investigation was also carried by XRD with specimen stage capable of moving in three orthogonal directions (1 μm sensitivity). The clad section is placed on zero diffraction plate and XRD pattern were recorded from the area of 2.1mm x 4.3 mm across the thickness of the clad sample surface with 300 µm x 300 µm step size along X and Y direction respectively.

4. Microstructural examination of Ferroboron clad

The optical microscopic examination revealed reduction in wall thickness in the central sub-capsule of ferroboron clad (Fig.1) which has seen the peak displacement damage. The maximum clad wall thickness reduction measured using optical microscopy was around 200 micrometer.

The SEM examination confirms the clad attack leading to formation of interaction phases at the FeB-clad interface. The extent of interaction is found to be varying along the circumference of the clad inner surface. In addition, the interaction was also found to be varying both radially and circumferentially (Fig.2).

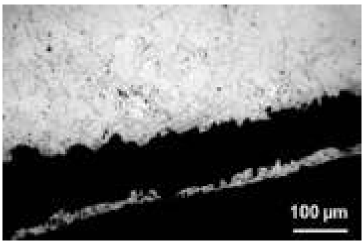

Fig. 1 Microstructure of the SS 304 L clad showing the clad attack clad

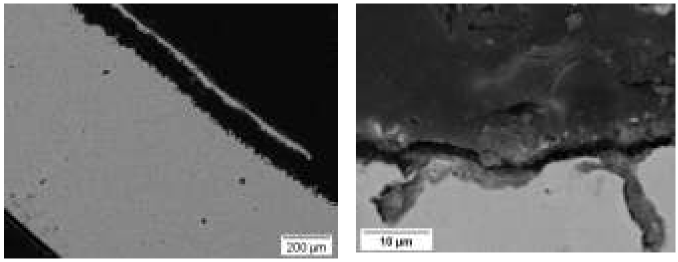

Fig. 2 SEM micrographs indicating chemical interaction between FeB and SS 304L clad

5. Phase Investigation by XRD

From the line scan across the clad wall thickness, distinct XRD patterns were recorded. Towards the inner side of the clad, a Lithium rich secondary phase formed due to chemical interaction between the clad and the FeB shielding material. The interaction zone measured from XRD was found to be in agreement with the values obtained from SEM analysis. As per the crystallography open database (COD) it was found that the interaction phase is matching with $Fe_{0.5}Li_{0.217}$. The XRD pattern obtained from a scan point at the Fe-Li phase is given in figure 3.

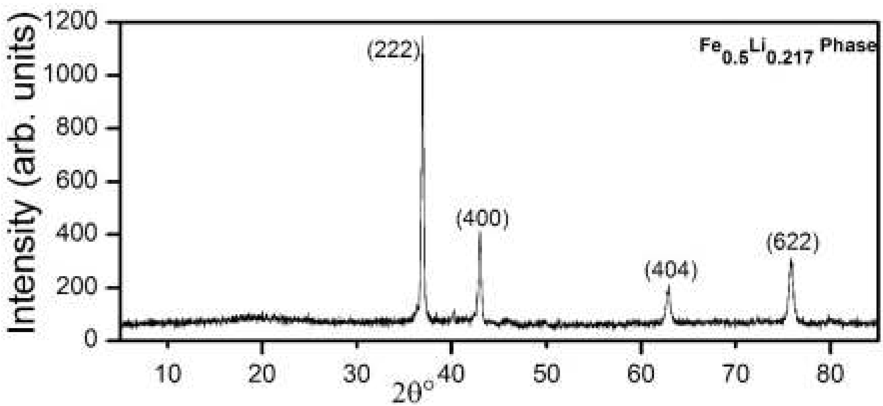

Fig. 3 XRD pattern at the interface region showing the Fe0.5Li0.217 interaction phase.

Lithium is formed due to (n, α) reaction with Boron as given bellow:

$^{10}B + {}^{1}n \rightarrow [{}^{11}B]^{*} \rightarrow \alpha + {}^{7}Li + 2.31$ MeV

Nascent "Li" atom, having higher affinity to Fe, is smaller enough to diffuse easily at inner surface of the clad and forms the $Fe_{0.5}Li_{0.217}$ phase.

6. Conclusion

From the accelerated irradiation experiment of the FeB shielding subassembly for the target life-time of 60 years indicated the clad damage due to interaction of ferroboron to form a Li rich discontinuous phase at inner side of the clad tube. The extent of clad damage due to "Li" attack is found to be limited to ~200 micrometers, which is not a life limiting factor for the shielding subassembly during its service life.

References

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