



# Chemical compatibility study of lithium titanate with Indian reduced activation ferritic martensitic steel



Sagar Sonak<sup>a,\*</sup>, Uttam Jain<sup>a</sup>, Rumu Halder<sup>b</sup>, Sanjay Kumar<sup>a</sup>

<sup>a</sup> Fusion Reactor Materials Section, Bhabha Atomic Research Centre, Mumbai 400 085, India

<sup>b</sup> Material Science Division, Bhabha Atomic Research Centre, Mumbai 400 085, India

## HIGHLIGHTS

- Chemical compatibility between  $\text{Li}_2\text{TiO}_3$  and Indian RAFM steel has been studied at ITER operating temperature.
- The lithium titanate chemically reacted with ferritic martensitic steel to form a brittle and non-adherent oxide layer.
- The layer grew in a parabolic manner as a function of heating time.
- Diffusion of oxygen (from  $\text{Li}_2\text{TiO}_3$ ) appears to be controlling the oxide layer.

## ARTICLE INFO

### Article history:

Received 30 March 2015

Received in revised form 20 July 2015

Accepted 25 July 2015

Available online 12 August 2015

### Keywords:

Lithium titanate

Ferritic martensitic steel

Corrosion

TBM

## ABSTRACT

Chemical compatibility between lithium titanate and Indian reduced activation ferritic-martensitic steel (In-RAFMS) was studied for the first time under ITER operating temperature. Lithium titanate required for the study was synthesized in-house. Coupons of In-RAFMS were packed inside lithium titanate powder and heated at 550 °C up to 900 h under inert argon atmosphere. The lithium titanate chemically reacted with ferritic martensitic steel to form a brittle and non-adherent oxide layer. The layer grew in a parabolic manner as a function of heating time. Microstructural and phase evolution of this oxide layer was studied using XRD, SEM and EPMA. Iron and chromium enriched zones were found within the oxide layer. Diffusion of oxygen (from  $\text{Li}_2\text{TiO}_3$ ) appears to be controlling the oxide layer.

© 2015 Elsevier B.V. All rights reserved.

## 1. Introduction

India has proposed Lead-Lithium Cooled Ceramic Breeder (LLCB) concept of Test Blanket module to be tested in upcoming International Thermonuclear Experimental Reactor (ITER). The technical data on the feasibility and compatibility of these materials in ITER environment will provide the valuable technical data for the design and operation of DEMO fusion reactors [1,2]. The Indian LLCB-TBM design involves reduced activation ferritic-martensitic steel as the structural material, lithium titanate ceramic pebbles as tritium breeders and liquid lead lithium eutectic as tritium breeder, neutron multiplier and coolant. Tritium will be generated in situ during reactor operation by ( $n,\alpha$ ) reaction with  $\text{Li}^6$  in the tritium breeding zones [3–5]. Tritium breeding zone consists pebble beds of  $\text{Li}_2\text{TiO}_3$  with diameter in the range of 300–600  $\mu\text{m}$  [6]. The heat generated in tritium breeding zone will be extracted by flowing Pb-Li eutectic flowing adjacent to the  $\text{Li}_2\text{TiO}_3$  bed packed in In-RAFMS

columns. The structural material (In-RAFMS) will be in continuous contact with  $\text{Li}_2\text{TiO}_3$  pebbles at one side and flowing liquid Pb-Li eutectic at another side.

Over the years, a few reports have been published on the compatibility studies of liquid lead lithium eutectic with In-RAFMS or similar ferritic-martensitic steels [7–12]. The weight loss and degradation in mechanical properties of steel in the presence of liquid lead lithium eutectic is reported. However, no such studies are available on the chemical interaction of  $\text{Li}_2\text{TiO}_3$  either with In-RAFMS or ferritic-martensitic steel. Considering the solid-solid nature of reaction between steel and lithium titanate, the chemical interaction is likely to be slower as compared to the interaction of steel with liquid lead lithium eutectic. This could be one of the reasons for limited data available on present system. However, recently Hernández et al. [13,14] studied the corrosion compatibility of  $\text{Li}_4\text{SiO}_4$  and  $\text{Li}_6\text{SiO}_5$  with Eurofer-97 in ITER relevant conditions. They observed formation of corrosion layers up to 700 nm for  $\text{Li}_4\text{SiO}_4$  and layers up to 5000 nm  $\text{Li}_6\text{SiO}_5$  for exposure time of 2660 h. Phase analysis showed corrosion layers consisting mixed oxides of Fe, Cr and Li. These layers were brittle, porous and fragile. The formation of brittle and fragile nature of corrosion

\* Corresponding author.

E-mail address: [sagarsonak@gmail.com](mailto:sagarsonak@gmail.com) (S. Sonak).

**Table 1**  
Chemical composition of In-RAFMS steel. All values are in %wt.

C	P	S	Ti	v	Cr	Mn	Ta	W	N	O	Fe
0.12 ± 0.001	0.002 ± 0.001	0.002 ± 0.001	0.002 ± 0.001	0.21 ± 0.02	9.02 ± 0.45	0.57 ± 0.01	0.07 ± 0.02	1.46 ± 0.03	0.03	0.01	Rem.

layer is deleterious to the long term performance of In-RAFMS steel as a structural material. The corrosion from ceramic breeders, together with corrosion from liquid lead lithium eutectic may pose operational difficulty in the long term.

The present work studies the interaction of  $\text{Li}_2\text{TiO}_3$  with In-RAFMS. The  $\text{Li}_2\text{TiO}_3$  powder required for the study was synthesized in-house. The steel specimens were exposed to  $\text{Li}_2\text{TiO}_3$  powder up to 900 h. The nature of oxide layer formed on the surfaces was characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) and electron probe microanalysis (EPMA) techniques. The growth mechanism of oxide layer is evaluated and possible mechanism is discussed.

## 2. Experimental

Lithium titanate required for the present study was prepared in-house from the solid state reaction of lithium carbonate and titanium oxide. A homogeneous mixture of lithium carbonate and titanium oxide was wet milled in alcohol medium. This wet milled powder mixture was then dried in vacuum oven and heated at 1000 °C for 2 h in open atmosphere. The product obtained was lithium titanate as confirmed by X-ray diffraction.

The In-RAFMS specimens required for the interaction studies with lithium titanate were fabricated from the as received sheet of In-RAFMS. A detailed chemical composition of In-RAFMS is presented in Table 1. The microstructure is tempered martensite structure with chromium and tungsten rich coarse  $\text{M}_{23}\text{C}_6$  carbides on grain boundaries and fine tantalum and vanadium rich MX type carbides inside the grains.

The In-RAFMS steel specimens had dimensions of length: 10 mm width: 10 mm and thickness: 5 mm. These specimens were polished up to 1  $\mu\text{m}$  diamond cloth and packed inside the powder bed of  $\text{Li}_2\text{TiO}_3$ . Before packing, the  $\text{Li}_2\text{TiO}_3$  powder was calcined at 300 °C and kept inside vacuum desiccators to eliminate possible contamination of moisture. 5 g of Lithium titanate powder was used in the present study. Steel coupons of In-RAFMS were kept inside the calcined powder bed and compacted under the load of 200 MPa at room temperature in a steel die using a hydraulic press (Fig. 1). This was to ensure that the In-RAFMS pellets will be well surrounded by calcined  $\text{Li}_2\text{TiO}_3$  powder and isolated from outside atmosphere. The pellets obtained after cold compaction now consisted steel specimens surrounded by  $\text{Li}_2\text{TiO}_3$  powder. The pellets had the diameter of 12 mm and thickness of 10 mm. These pellets

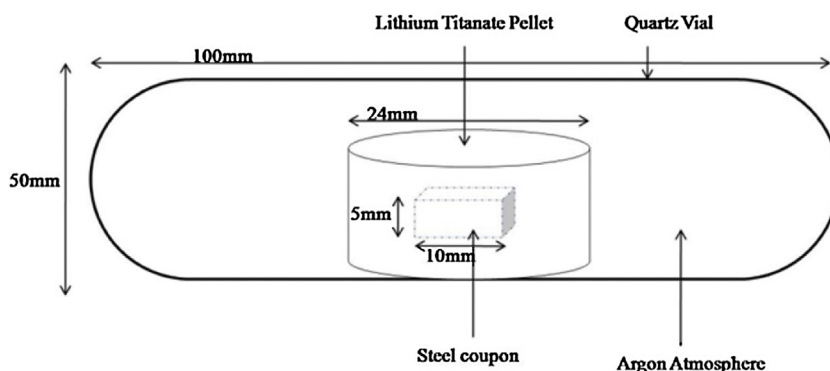
consisting steel coupons were encapsulated under argon environment at one atmosphere in quartz vials and heated at 550 °C for 100, 200, 400 and 900 h. Fig. 1 shows the complete experimental apparatus used in the study. After completion of desired heating cycles, the In-RAFMS pellets were gently removed from the  $\text{Li}_2\text{TiO}_3$  cover. The interface of the In-RAFMS exposed to  $\text{Li}_2\text{TiO}_3$  was analyzed by X-ray diffractometer in standard theta–two theta geometry with a step size of 0.005°. Standard  $\text{Cu K}\alpha$  radiation source was used in the machine for the study. The diffraction profile was obtained at the tube voltage of 40.0 kV and tube current of 40 mA respectively.

The microstructure of reacted steel face at the cross section was observed under scanning electron microscope (SEC SNE-3000). The elemental compositional analysis across the interface between  $\text{Li}_2\text{TiO}_3$  and In-RAFMS was performed using Electron probe micro analyser (CAMECA SX 100). The samples were analyzed with 20 kV acceleration voltage and 20 nA stabilized beam current. The beam size was kept at  $\leq 2 \mu\text{m}$ . The points for analyses were selected at regular intervals of  $\leq 1 \mu\text{m}$  depending upon the diffusion length by scanning the sample across the interface of  $\text{Li}_2\text{TiO}_3$  and In-RAFMS.

## 3. Results and discussion

The visual examination of all the In-RAFMS samples showed formation of a dark colored layer over the surface. It was apparent that the reaction took place between In-RAFMS steel and  $\text{Li}_2\text{TiO}_3$ . The phases forming over the surface and their structural evolution were studied using XRD analysis. Fig. 2 shows the X-ray diffraction profile of the surface layer formed over the specimen heated for 900 h. The XRD analysis identified the surface layers as  $\text{Fe}_3\text{O}_4$  phase (ICDD PDF: 079-0418). Oxides of other alloying elements of In-RAFMS or Li and Ti were not observed in the XRD analysis.

The microstructural characterization of corrosion layer was done using Scanning Electron Microscopy (SEM). The In-RAFMS steel specimens recovered after heating in powder bed were sliced along their cross sections, polished and viewed under scanning electron microscope. Fig. 3 shows the microstructures of the cross section of samples obtained after heating for 100, 200, 400 and 900 h. The SEM images clearly show the formation of a continuous but non-adherent corrosion layer at the edges. A marginal difference in contrast was observed near the metal and oxide layer interface indicating formation of a double layer. The phase composition of inner double layer, however, could not be detected by XRD owing to the limitation in penetration depth of X-ray beam.



**Fig. 1.** A RAFMS steel sample packed inside the compacted lithium titanate powder.

Download English Version:

<https://daneshyari.com/en/article/6745979>

Download Persian Version:

<https://daneshyari.com/article/6745979>

[Daneshyari.com](https://daneshyari.com)