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Corrosion behavior of IN-RAFM steel with stagnant lead-lithium at 550 °C up to 9000 h

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ABSTRACT

Corrosion study of IN-RAFM (India specific reduced activation ferritic martensitic) steel with static lead-lithium, Pb-16Li has been carried out at 550 °C for different time durations, 2500, 5000 and 9000 h. Flat and tensile IN-RAFM samples were exposed to liquid metal. Exposed samples were analyzed for micro structural observation and chemical composition by scanning electron microscope equipped with EDX (energy dispersive X-ray spectrometer). Micro vickers hardness measurement and tensile testing were also carried out. Corrosion rate of IN-RAFM steel with static Pb-16Li at 550 °C is ~38 μm/year. After exposure to liquid metal, granule, nodule and pebble like structures were observed in the surface micrographs. Iron, chromium and manganese were found leaching from near surface regions from a depth of ~15 μm. Hardness reduction was observed up to a depth of ~15 μm after exposure to liquid metal. There was no significant reduction in the tensile strength. Dimpled ductile fracture was observed after exposure to liquid metal up to 9000 h.

1. Introduction

Lead lithium eutectic is one of the proposed coolant and breeder for fusion reactors [1–4]. In Indian LLCB (lead lithium ceramic breeder) test blanket module [5–7] concept, Pb-16Li is used as coolant, neutron multiplier and tritium breeder. IN- RAFM steel [8,9] is the candidate structural material for this test blanket module. This material has been developed from its surrogate material, modified 9Cr-1Mo steel (P91) by substituting highly radioactive element molybdenum by tungsten and niobium by tantalum [10]. Compatibility of structural material with liquid metal coolant is crucial for such an application [11–19]. For this, It is necessary to understand the various factors contributing to liquid metal corrosion, under different conditions. Static experiments, in which sample coupons of structural material, exposed to static liquid metal, maintained at a particular temperature can give preliminary understanding of corrosion mechanism. Once the material performance under static condition is known, extent of degradation of material can be evaluated due to introduction of velocity factor, in forced flow conditions. The driving factors for static condition are the maximum solubility of each element at the temperature of liquid metal and concentration gradient of individual alloying elements between the RAFM material and the liquid metal and the liquid metal and the crucible/vessel material. The crucible/vessel holding the liquid metal also plays an important role, as corrosion occurs both from the sample coupons of structural material as well as crucible material. If the crucible material is different from the material being tested, then the dissolution of individual alloying elements from the crucible can saturate the liquid metal, due to larger surface area of the crucible compared to that of specimens being tested. As two dissimilar metals (crucible material and the material being tested) are in contact with liquid metal, dis-similar metal mass transfer can occur, due to the thermo dynamic requirement that all element chemical potentials should be equal in all phases and redistribution of elements occur until the condition of equality of chemical potential is satisfied [20].

Static experiments have been carried out in the past, exposing RAFM steels such as CLAM, JLF-1, F82H-mod and 9Cr-ODS steels to liquid metals, such as lithium and lead-lithium [21–27]. To study the effect of dissimilar metal mass transfer, static corrosion experiments were carried out in different crucible materials [21,22]. Impurity effect over corrosion has been studied for lithium with dissolved oxygen and nitrogen. It is found that Impurities such as nitrogen in liquid meal can enhance the corrosion significantly [21,23]. Compatibility of different ferritic martensitic steels with lead-lithium has been studied worldwide in static and flowing conditions [11–34,36–64]. It is reported that, dissolution rates of ferritic steels in lead-lithium are an order of magnitude greater than in lithium [13].

The purpose of the present study is to investigate the effect of corrosion of IN-RAFM steel with stagnant Pb-16Li on microstructure and

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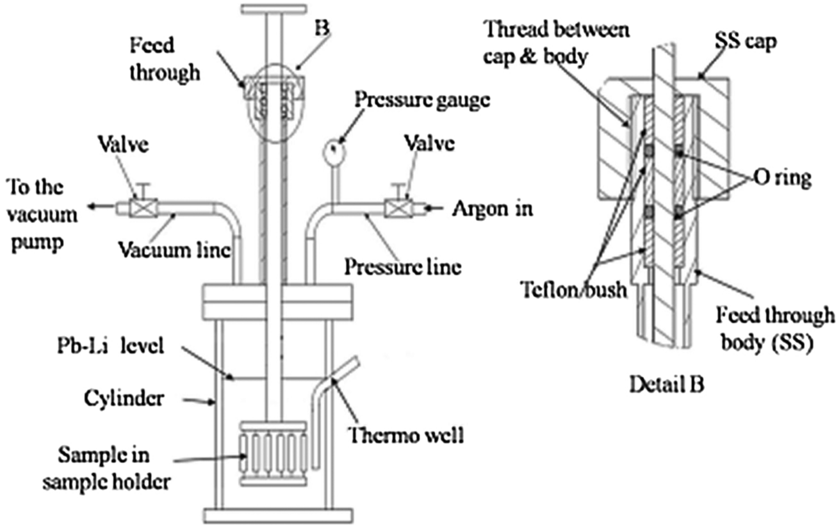

Fig. 1. Schematic of the experimental setup.

mechanical properties.

For this purpose, a static experiment was conducted, in which IN-RAFM steel samples were exposed to static liquid metal, Pb-16Li, at 550 °C temperature. Here after Pb-16Li will be referred to as Pb-Li. This paper discusses the experimental details and the results obtained.

2. Experimental set up

Schematic of the experimental set up is shown in Fig. 1. Lead-lithium chunks were loaded in a cylindrical vessel, made of stainless steel 316 L. Inner diameter and thickness of the vessel were ∼78 mm and 5.5 mm respectively. A ‘K’ type thermocouple was introduced into the thermo well, welded on one side of the vessel. A three kW heater coil was wound over the circumference of the chamber to melt lead-lithium chunks. Six flat and six tensile IN-RAFM steel samples were fixed to a sample holder. It was fixed at one end of the feed through, which is used to move the sample holder up and down in the vessel. Size of the IN-RAFM steel samples exposed to Pb-Li was 35 mm (l) x 9.5 mm (w) x 1.6 mm (t). Samples are shown in Fig. 2. Chemical composition of IN-RAFM steel and SS316 L are given in Tables 1 and 2 respectively. Chemical composition of liquid metal is given in Table 3.

Initially, stainless steel vessel was evacuated to 10^{-3} mbar, raising the temperature of the vessel along with the chunks up to 200 °C. Then the vacuum system was isolated and high purity argon cover gas was introduced. Temperature of the chamber along with the chunks was slowly raised to 550 °C. Once all the chunks melted and the melt temperature was maintained at 550 °C, the sample holder was slowly lowered with the help of feed through arrangement to dip the samples into the liquid metal melt. Volume of Pb-Li used in the experiment ∼382 cm^3. A positive pressure of 1.5 bar was maintained over the liquid metal melt throughout the experiment.

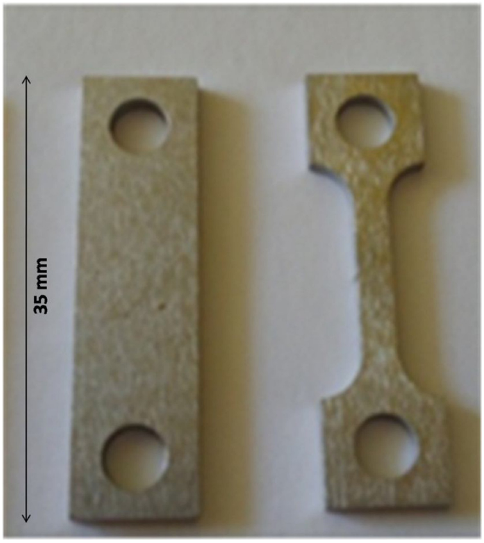

Fig. 2. Flat and tensile samples.

In this setup, three experiments were carried out for different time durations, 2500, 5000 and 9000 h. Two flat and two tensile samples were removed from the chamber after each exposure time. To remove the adherent Pb-Li, the samples were cleaned using cleaning solution, consisting of acetic acid, hydrogen peroxide and ethyl alcohol in 1:1:1 ratio. The samples were cleaned and weighed till similar consecutive readings were obtained. Weight measurements were taken using Sartorius precision weighing balance with a precision of ± 0.01 mg. After cleaning, the samples were cut and molds were prepared for metallographic examination. The molds were ground using different grade Silicon carbide (SiC) emery papers and finally polished with alumina paste to achieve mirror finishing.

3. Results and discussion

3.1. Weight change

Weight of the samples was measured before and after exposure to liquid metal for different time durations, i.e. for 2500, 5000 and 9000 h. From weight loss measurements, dissolution of the sample per unit area per year (dissolution rate) and thinning of the sample per year (corrosion rate) were calculated. In this calculation, it was assumed that the surface area of the sample before and after exposure to liquid metal was same.

Weight loss, dissolution rate and corrosion rate are given in Table 4. From weight loss measurements, corrosion rate of IN-RAFM steel is estimated to be ∼ 38 μm/year.

Weight loss vs. exposure time is shown in Fig. 3. Weight loss is non linear with respect to time.

From Table 4, it can be seen that though weight loss increased with exposure time, corrosion rate decreased with exposure time. Dissolution rate and corrosion rate were found to be nearly same after 2500 h and 5000 h exposure but decreased after 9000 h exposure. This could possibly be due to the formation of saturated layer of dissolved elements near the sample surface. Non linear pattern of corrosion rate could be