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Formation of Al_2O_3/FeAl coatings on a 9Cr-1Mo steel, and corrosion evaluation in flowing Pb-17Li loop

Sanjib Majumdar [a,*], Bhaskar Paul [a], Poulami Chakraborty [b], Jugal Kishor [a], Vivekanand Kain [a], Gautam Kumar Dey [b,c]

[a] High Temperature Materials Development Section, Materials Processing & Corrosion Engineering Division, Bhabha Atomic Research Centre, Trombay, Mumbai, India

[b] Materials Science Division, Bhabha Atomic Research Centre, Trombay, Mumbai, India

[c] Materials Group, Bhabha Atomic Research Centre, Trombay, Mumbai, India

HIGHLIGHTS

- Al_2O_3/FeAl coating produced on P91 steel by pack aluminizing and heat treatment.
- Corrosion tests of coated steel conducted in flowing Pb-17Li loop at 500 °C for 5000 h.
- Coating was protective against molten metal corrosion during prolonged exposure.
- Self-healing protective oxides formed in the cracks generated in aluminide layers.

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ABSTRACT

Iron aluminide coating layers were formed on a ferritic martensitic grade 9Cr-1Mo (P 91) steel using pack aluminizing process. The formation of different aluminide compositions such as orthorhombic-Fe_2Al_5, B2-FeAl and A2-Fe(Al) on the pack chemistry and heat treatment conditions have been established. About 4–6 μm thick Al_2O_3 scale was formed on the FeAl phase by controlled heat treatment. The corrosion tests were conducted using both the FeAl and Al_2O_3/FeAl coated specimens in an electromagnetic pump driven Pb-17Li Loop at 500 °C for 5000 h maintaining a flow velocity of 1.5 m/s. The detailed characterization studies using scanning electron microscopy, back-scattered electron imaging and energy dispersive spectrometry revealed no deterioration of the coating layers after the corrosion tests. Self-healing oxides were formed at the cracks generated in the aluminide layers during thermal cycling and protected the base alloy (steel) from any kind of elemental dissolution or microstructural degradation.

1. Introduction

Producing the clean energy from thermonuclear fusion and hydrogen fuel driven power generating systems has been the main focus of research in recent times. India has participated in the International Thermonuclear Experimental Reactor (ITER) program, and proposed to place Lead–Lithium cooled Ceramic Breeder (LLCB) Test Blanket Module (TBM) in ITER with the primary objectives to (i) breed tritium with Tritium Breeding Ratio (TBR) > 1, and to (ii) extract the high grade heat with acceptable thermal efficiency [1,2]. Reduced activation ferritic martensitic steel (RAFMS) having a microstructure comprising of tempered martensitic with coarse $M_{23}C_6$ carbides, rich in Cr and W on the lath boundary and fine intra-lath Ta and V rich MX precipitates, is considered the reference structural material for future fusion power reactors [3]. The development of a suitable coating is considered as essential to improve the performance of the material (RAFMS) used in test blanket module (TBM). The main purpose of forming the coatings on martensitic steels is to (i) provide electrical insulation for mitigating magneto hydrodynamic (MHD) effects in self-cooled liquid metal (Pb-Li) systems, (ii) produce an effective tritium permeation barrier capability, (iii) improve the corrosion resistance against liquid metals at high temperatures, and (iv) provide helium containment to reduce helium leakage into the

* Corresponding author.
E-mail addresses: sanjib@barc.gov.in, sanjib731@gmail.com (S. Majumdar).

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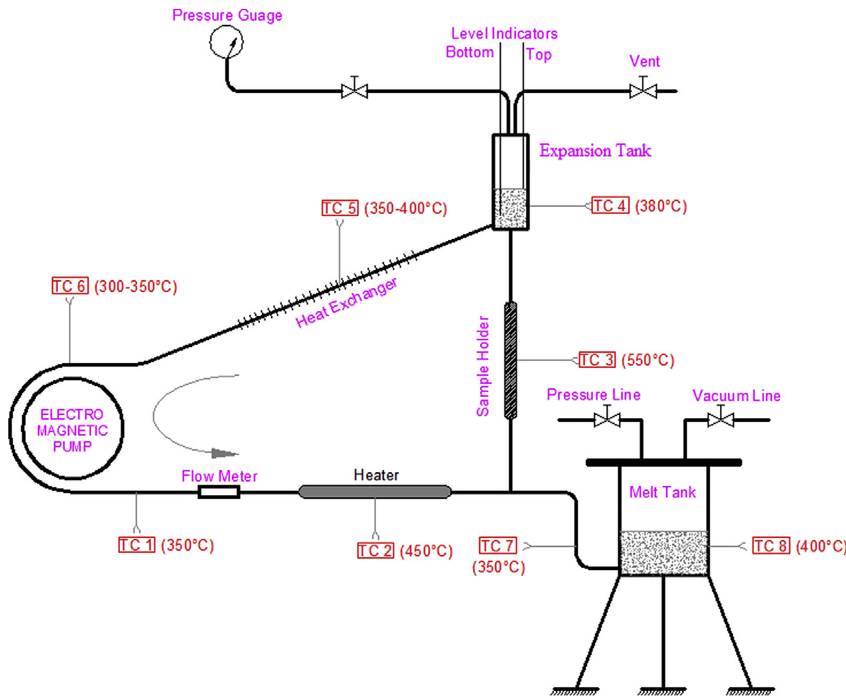

Fig. 1. Schematic of the Pb-17Li loop used for corrosion tests of the coated P 91 steel specimens.

plasma chamber. The other desired characteristics of the coatings are good irradiation resistance, higher thermal conductivity, mechanical integrity with the substrate material, and uniformity in complex geometries. Various options including Al_2O_3/Fe-Al, Er_2O_3, Cr_2O_3, TiC, TiN, and SiC are investigated as the coating material [4–16].

Alumina/Fe-Al coating is the most promising as tritium permeation barrier with the potential to generate a tritium permeation reduction factor (TPRF) of the order of 100–1000 compared to the permeation through bare steel [4,12,14]. Amongst the different coating techniques such as CVD [4–7,12], PVD, HDA [8,11], electrochemical deposition [10], the halide activated pack cementation (HAPC) technique has its distinct advantages of forming the desired coating on larger size components and complex geometries. The technological issues related to removal of coating layers from the surface near the weld to avoid its penetration in the weld zone, and to minimize the number of welds the bending of tubes before the application of coating are addressed earlier [5–7]. HAPC is basically a CVD type process in which the Al is deposited on the substrate surface from its halide vapour produced in-situ [17,18]. The corrosion behaviour of the ferritic martensitic steels in static and flowing liquid metal (Pb/Pb-Li) is reported earlier [19–29]. The dissolution of the matrix elements Cr and Fe, and also a change in microstructure up to certain depth from the surface of contact with the liquid metal has been observed. However, a limited literature data [10,11,30,31] is available addressing the liquid metal corrosion behaviour of aluminide and alumina coated steels. In the present investigation, the formation of aluminide coating layers on a surrogate martensitic material (grade P91 steel) for fusion reactor application using HAPC technique has been studied. The coated steels were subsequently heat treated to form alumina/Fe-Al layers. Liquid metal corrosion tests of the coated specimens were conducted in a flowing molten Pb-17Li (at. %) loop. The specimens obtained before and after the corrosion tests were characterized using different techniques.

2. Experimental procedure

2.1. Formation of coating

The chemical composition (in wt.%) of the grade P91 steel used in the present investigations is 0.30–0.60Mn, 0.20–0.50Si, 8.00–9.50Cr, 0.85–1.05Mo, 0.18–0.25V, 0.06–0.10Nb, 0.03–0.07N and balance Fe. For optimizing the parameters of the pack aluminizing process the samples of uniform cross sections 15 mm × 10 mm × 2 mm were cut from a thick plate of P91 steel.

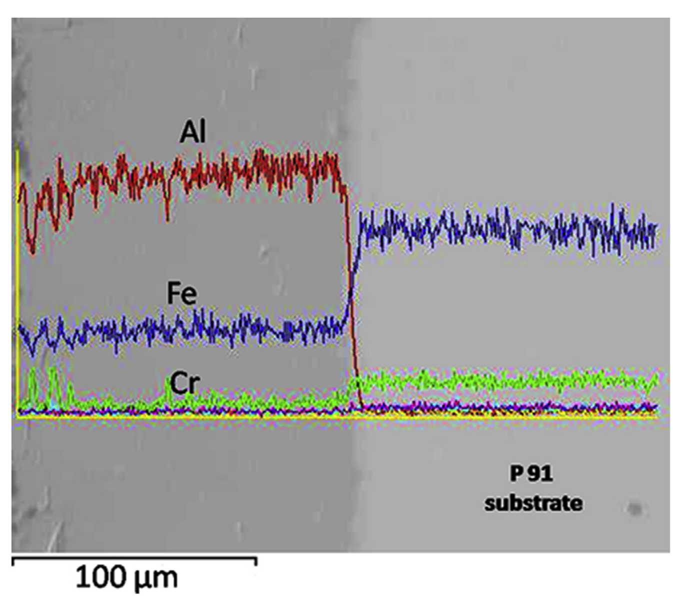

Fig. 2. SEM cross-sectional image and EDS line profiles for the pack aluminized coating formed at 650 °C using a pack composition $10Al-4NH_4Cl-86Al_2O_3$.