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Corrosion of Fe9Cr1Mo steel in stagnant liquid lead–lithium eutectic

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ABSTRACT

The corrosion behaviour of Fe9Cr1Mo steel samples in a static $Pb_{83}Li_{17}$ eutectic melt at 823 K in a specially designed necked quartz capsule is investigated. The samples are kept isothermally for 500, 1000, 1500 and 2000 h. The changes in microstructure and depletion of alloying components of the sample kept for 2000 h have been studied using scanning electron microscope (SEM), energy disperses X-rays (EDS) and electron probe beam microanalysis (EPMA). Weight loss and thinning of the walls has been calculated by gravimetric analysis method for all the samples. Using this data the change in thickness per year is calculated. Preferential dissolution of major constituent elements namely iron and chromium from the samples with the formation of a ferritic–martenstic layer is observed. Percentage mass loss with respect to time follows a parabolic curve which indicates non linear mechanism of corrosion.

1. Introduction

Thermonuclear reactor (fusion reactor) is a potential alternative and high energy density source of energy to achieve the growing demands of energy in future. Substantially less amount of nuclear waste generation, eco-friendly, limitless fuel source and high energy density captivate its importance. The thermonuclear reactor based on magnetic confinement plasma of deuterium–tritium (D–T) fuel cycle in which huge amount of thermal energy will be liberated by fusion reaction of tritium and deuterium confined within the magnetically suspended plasma [1,2]. The thermal energy thus produced should be extracted through suitable coolant to transform into usable form of energy, for example electrical energy. The key issues related to thermonuclear reactor are the selection of competent coolant, to extract the huge amount of thermal energy, and structural material, competent with the coolant and withstand against the high radiation and high thermal stresses [3–5]. Many structural materials including various classes of stainless steels, vanadium & vanadium based alloys and $SiC–SiC_f$ composite have been proposed for the reactor. The pre-eminent appropriate material will be selected by considering its thermo-mechanical properties, capability to withstand against high neutron flux and compatibility with coolant [6]. In this context behaviour of the surrogate material with the potential coolant is an important issue to be discussed before selection and development of material for this specific structural application. Fe9Cr1Mo steels or P91 is a structure materials presently used in the nuclear fission reactors worldwide [7,8] because of its unique physical properties such as high tensile strength, high hardness, fatigue resistance and resistance to irradiation. It appears promising candidate surrogate material for the development of fusion reactor first wall structure component with a suitable modification in the composition to stand against high neutron flux (neutron flux in fusion reactor will be substantially higher compared to usual nuclear reactor based on fission reaction) [9–11] and compatible with the proposed coolant. The eutectic $Pb_{83}Li_{17}$ alloy has been proposed as a coolant and secondary tritium breeding material in lead lithium ceramic breeder test blanket module (LLCB-TBM) in the international thermonuclear experimental reactor (ITER) [10]. The lithium component of the alloy participates in tritium breeding while neutron multiplication takes place in lead which eventually enhances the tritium breeding performance of lithium. As per the available literature data corrosion properties of $Pb_{83}Li_{17}$ is substantially higher compared to other proposed coolant materials such as pure lead and pure lithium. India is one of the participants of the ITER project and developing LLCB (lead lithium ceramic breeder) TBM (test blanket module) and HCSM TBM for testing in ITER. LLCB blanket concept aims to optimize the use of both $Pb_{83}Li_{17}$ and ceramic breeder with helium and $Pb_{83}Li_{17}$ as coolants. The TBM structural materials are under optimisation and Fe9Cr1Mo steel has been selected as a surrogate material [12,13]. In this context the corrosion behaviour of coolant $Pb_{83}Li_{17}$ with surrogate materials Fe9Cr1Mo in stagnant condition is essential to design and develop a suitable composition to be used as structural material compatible to $Pb_{83}Li_{17}$ coolant. The corrosion rate is affected by various factors relevant to the ITER–TBM design [14–16]. The investigation of the corrosion behaviour of the $Pb_{83}Li_{17}$ melt on Fe9Cr1Mo is quite essential both in dynamic as well as in stagnant condition of the eutectic melt to understand the corrosion mechanism and to develop a suitable composition resistant to corrosion.

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Table 1
Chemical compostion of P91 samples used in the present investigation.

Elements	Mo	Si	Mn	Cr	Fe
Percentage	1.01	0.35	0.48	9.08	Balance

Table 2
Chemical composition of $Pb_{83}Li_{17}$ eutectic.

Elements	Pb	Li	Ni	Bi
Percentage	Balance	0.59	0.02	0.01

Magnetic effects on the corrosion are another aspects to be discussed. Sufficient investigations on corrosion behaviour of different structural materials in a dynamic $Pb_{83}Li_{17}$ flow were reported earlier [17–22]. The dynamic corrosion study substantially affected by the erosion of the corroded surface which provides always a fresh surface to the molten $Pb_{83}Li_{17}$ alloy and enhanced the corrosion rate. Erosion–corrosion is limited to moving metallic interface. Common example arises when there is a rapid relative movement between fluids and metal parts, such as molten eutectic in present case, swirling along pipes or impellers churning in vats. Therefore exact pattern of corrosion due to dissolution and diffusion of the alloying component into the molten $Pb_{83}Li_{17}$, whose mechanism is governed by the difference in chemical potential, could not be properly explained. In the static molten $Pb_{83}Li_{17}$ condition corrosion of the structural material with the molten metal interface are by dissolving an alloying component into liquid metal by mass transfer of the alloying component of structure material and liquid metal ($Pb_{83}Li_{17}$) penetration into the solid structure material. Thermodynamically the corrosion phenomena governed by the diffusion of the soluble component due to chemical potential difference in the alloying components between the surface of the sample and the bulk of the melt. The results of such phenomena are accompanied by thinning of the walls of the structural material, degrading the mechanical properties and other undesirable effects. In the past, several corrosion tests were conducted in liquid $Pb_{83}Li_{17}$, the materials analyzed were austenitic steels and ferritic–martensitic steels [23–29]. Moreover, the tests were also performed in static conditions [30,31]. But as per the author's knowledge the time dependent static corrosion behaviours of Fe9Cr1Mo steel with the exact composition presented in the Table 1 is not reported. In the present study the corrosion of Fe9Cr1Mo with molten $Pb_{83}Li_{17}$ was investigated in a static condition at 823 K temperature in the interval of 500, 1000, 1500 and 2000 h. In spite of slight variation in composition in the whole text P91 will be used for the Fe9Cr1Mo steel and eutectic alloy for $Pb_{83}Li_{17}$.

2. Experimental

Commercially available P91 steel was used to fabricate the corrosion samples. The average surface areas of samples were 6.6 cm^2 with thickness 1.5867 mm. Shape and design of the sample was illustrated in Fig. 1A. Chemical analysis (using ICP-AES technique and wet chemical analysis) report of the P91 sample compositions are presented in the Table 1. Samples were annealed at 1250 K under high vacuum (10^{-7} Torr) condition to reduce the residual stress during sample preparation. All the surfaces of the samples were mechanically cleaned upto three delta finish using emery paper and diamond paste followed by chemical cleaning to remove the surface impurities. The weight of the samples was recorded before its uses for the corrosion experiment. The density of the samples (7672.5 kg/m^3) was measured by Archimedes principle. The samples were encapsulated along with ~0.5 L eutectic alloy in a high pure helium ($O_2 \leq 1$ ppm, $N_2 \leq 2$ ppm) filled necked vitrified quartz tube of wall thickness about ~3 mm. A schematic diagram of the encapsulated sample is shown in Fig. 2. Neck dimension of the quartz tube was kept less than the dimension of the corrosion sample to prevent the floating of the sample above the molten eutectic due to density difference. Both the compartments were filled with solid eutectic alloys initially and sample were placed in the lower compartment so that after melting of the eutectic alloy P91 samples should completely dipped into eutectic alloy in the lower partition. The chemical composition of the eutectic alloy is presented in Table 2. The encapsulated samples were kept in resistance heating furnace at 823 K temperature for different time interval. It was insured that sample should completely dipped inside the eutectic alloy. Several sets of experiments were conducted at the identical condition. After the completion of desired duration (500, 1000, 1500 and 2000 h) the samples were removed by breaking the vitrified quartz capsule. The samples after 2000 h experiments without cleaning were shown in Fig. 1B. The sample extracted was covered with the lead lithium alloy indicate that at this temperature and after 2000 h the wetting of the surface occurred. The solidified eutectic alloy slackly sticks to the samples surface were removed by gently scratching. The remaining eutectic alloy from the

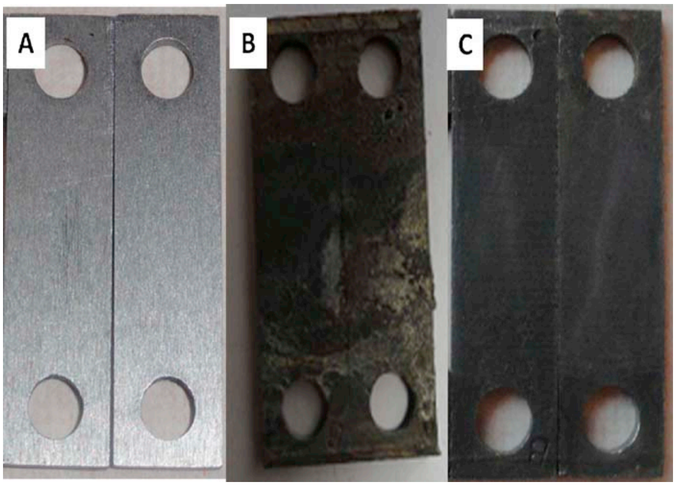

Fig. 1. P91 Sample (A) annealed and polished (B) corroded before cleaning (C) lithium cleaned.

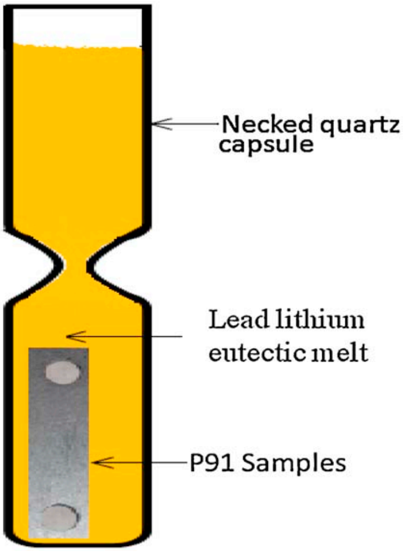

Fig. 2. Schematic diagram of the necked quartz and sample so that the sample should always remains inside the molten eutectic alloy.