

Corrosion mechanisms of Eurofer produced by lithium ceramics under fusion relevant conditions

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ABSTRACT

Advanced lithium-rich ceramic materials are under consideration for the helium cooled pebble-bed breeder unit prototype in ITER. Although chemical compatibility issues with reduced activation steels have in general been associated with liquid breeders, corrosion has also been observed to be produced by some solid compositions under specific conditions. In this work, the mechanisms including the corrosion process considering different lithium ceramics (lithium orthosilicate and lithium metatitanate) are examined, together with the influence of temperature and exposure time. The effect of the composition of the purge gas, intended for tritium extraction, is also evaluated. Corrosion in Eurofer was found to severely occur for metatitanate composition, where temperature plays a major role. Corrosion in Eurofer is produced all over the sample (out of contact areas), as a consequence of the gas swept.

1. Introduction

The helium cooled pebble bed (HCPB) concept for DEMO includes as fundamental elements a reduced activation ferritic-martensitic (RAFM) steel as structural material, lithium ceramics in the form of pebbles as tritium breeder, beryllium-based neutron multipliers, and helium as coolant [1]. The chemical compatibility of structural and functional materials is an essential issue to be addressed, given the high corrosion susceptibility of Eurofer in contact with lithium based materials [2]. Although corrosion processes in RAFM steels have been mainly associated with liquid breeders, recent studies showed that corrosion may also be important for some solid materials and under certain conditions [3,4]. In fact, chemical compatibility may be a decisive feature to be considered in the selection of the most suitable solid breeders.

Lithium metatitanate (Li_2TiO_3) and lithium orthosilicate (Li_4SiO_4) are the two main candidate materials for this purpose. Although orthosilicate contains higher lithium content than metatitanate, its mechanical properties are poorer, therefore mixtures of both with different proportions are proposed [1,5]. However, great differences in the corrosion behavior for both candidates have been observed which must also be taken into account, as discussed in the present paper.

The environmental conditions, particularly gasses that are in contact with the metal, will decisively influence the corrosion process. Hence, special attention should be paid to the reactivity of these two ceramics with the purge gas. Tritium extraction from the pebble bed is

carried out by means of a sweep gas, mainly composed of helium, at a pressure of ~ 0.2 MPa, and mass flow rate ~ 0.1 g/s [6]. The addition of small amounts of hydrogen, which would be the tritium carrier element, is established. More recently, the addition of H_2O instead of H_2 has been proposed, since the affinity of tritium is higher for water than for hydrogen and the extraction yield is expected to be higher [7]. The interaction with hydrogen does not seem to be a problem. Although it could produce partial reduction of the material, the reduced species would not affect the tritium release properties; however the interaction with water could be much more complex. Lithium orthosilicate is hygroscopic [8], so it easily absorbs water molecules on its surface. At high temperature, water could penetrate through the grain boundaries and partially dissolve the pebble; however, lithium metatitanate does not interact with water and the possible problems of corrosion could be related with secondary phases.

This paper presents the results of Eurofer corrosion by lithium orthosilicate and metatitanate under fusion-relevant conditions of temperature and purge gas mass flow. These results were obtained by advanced analytical techniques.

2. Materials and methods

The reduced activation stainless steel used in the experiments is Eurofer, and the characteristics of the pebbles are shown in Table 1. Eurofer samples ($7 \times 7 \times 4$ mm³) were kept in direct contact with the ceramic pebbles for up to 3700 h, in a special cylindrical chamber with

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Table 1
Characteristics of the pebbles.

Name	Manufacturer	Diameter (μm)	Fabrication method	Formulation	Raw materials	Ref
K-S	KIT	200–400	Melt spraying Kalos	$\text{Li}_4\text{SiO}_4 + 10\%\text{Li}_2\text{SiO}_3$ excess of SiO_2	$\text{LiOH} + \text{SiO}_2$	[13]
J-T	JAEA	900–1000	Emulsion method	$\text{Li}_{2+x}\text{TiO}_{3+y}$ excess of Li (Li/Ti = 2.2)	$\text{LiOH}\cdot\text{H}_2\text{O}$ and H_2TiO_3	[14]

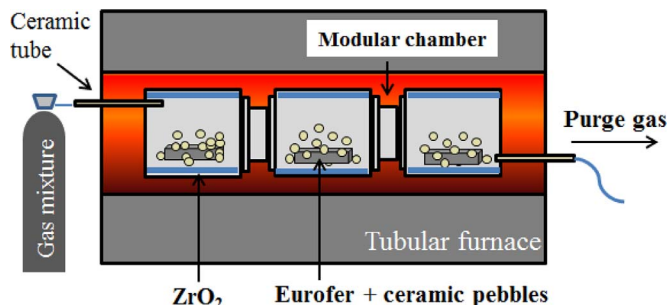


Fig. 1. Experimental assembly for corrosion tests.

a modular design manufactured at CIEMAT in which up to three breeders can be tested simultaneously (Fig. 1). Chamber inner walls are covered with ZrO_2 for protection against corrosion, and a stainless steel mesh has been installed between consecutive modules in order to prevent possible breeder mixture as a consequence of the sweep gas. A purge gas flow $\sim 1\text{cc}/\text{min}$ is maintained through the different chamber sections, which is controlled by a screw flowmeter. The chamber is introduced in a tubular furnace that permits testing temperatures up to 600°C . Corrosion tests have been carried out for two different ceramic pebbles at 350°C and 550°C for up to 3700 h, and for two different purge gas compositions: He/H_2 (98/2%vol) and $\text{He}/\text{H}_2\text{O}$ (99.8/0.2% vol).

The corrosion layer formed after the tests has been studied by scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM / EDX, Zeiss Auriga Compact/Bruker XFlash). Also, the distribution of light elements, such as lithium, has been investigated by secondary ion mass spectroscopy (SIMS, Hiden Analytical).

Gaseous species involved in the corrosion process were studied by thermally induced desorption (TID). For this, ceramic pebbles for the two compositions under examination were first subjected to the purge gas for a shorter period of time 24 hours but at a higher temperature (700°C) in the same modular chamber, and then mounted in a separate vacuum system equipped with an oven and a quadrupole mass spectrometer (Pfeiffer PrismaPlus QMG 220 residual gas analyzer). Absorbed gases in the ceramic pebbles in the range of 1–100 amu can be detected with sensitivity $\geq 10^{-14}$ mbar.

3. Results and discussion

Fig. 2 shows cross sectional SEM micrographs of the corrosion layer produced in Eurofer after 3700 h in contact with either K-S or J-T lithium ceramic pebbles at 550°C , and for two different purge gas compositions. While a thin and homogeneous corrosion layer is produced in the metal surface for the K-S pebbles ($\leq 1\mu\text{m}$ thickness), severe corrosion is observed for J-T material regardless of the purge gas, which can be clearly seen even at low magnification. The layer produced by the J-T pebbles appears porous and fragmented, the different grey tonalities corresponding with different compositions.

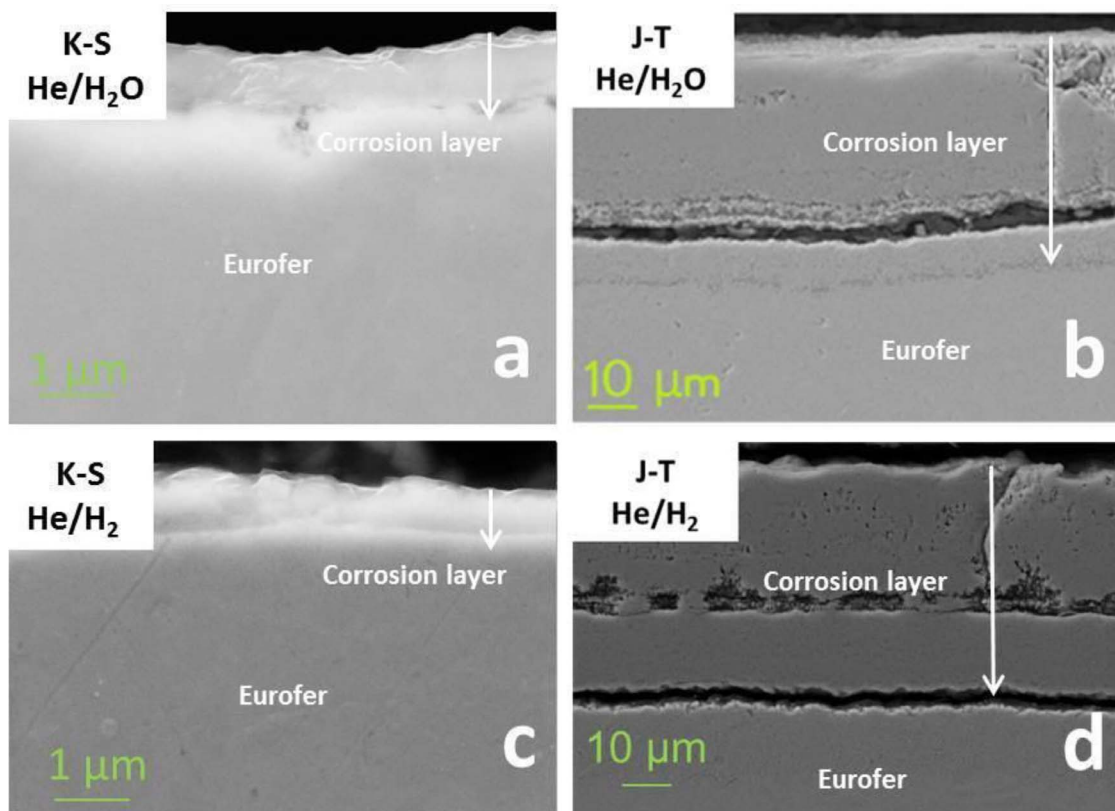


Fig. 2. Corrosion layer produced on Eurofer after 3700 h of exposition at 550°C .

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