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Corrosion experiments in flowing LBE at 450 °C

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Abstract

The corrosion behaviour of AISI 316L steel is being investigated in flowing lead–bismuth eutectic (LBE) at 450 °C. The tests are performed in the LECOR (lead corrosion) loop, at low oxygen activity in LBE. The preliminary results presented in this paper refer to AISI 316L stainless steel and show the start of a corrosion mechanism with a small transfer of Cr from the inner part of the steel matrix to the outer one. This paper reports only preliminary results, obtained after 1000 h of test.

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1. Introduction

In accelerator driven systems (ADS), one of the concerned issues is the material compatibility and corrosion in lead–bismuth eutectic (LBE). Liquid metal corrosion to the structural materials can proceed via various processes: species dissolution, formation of the inter-metallic compounds at the steels/liquid metal interface, and penetration of liquid metal along grain boundaries and in metal. The occurrence of these corrosion phenomenon depend on the experimental parameters, such as temperature, thermal gradients, solid and liquid metal compositions, velocity of the liquid metal and oxygen activity in LBE.

There is few data on the corrosion of steels in LBE available. This paper reports preliminary data on AISI 316L steel (Table 1) corrosion properties, exposed to LBE at 450 °C with very low dissolved oxygen concentration.

2. Experimental

The corrosion tests were performed in the LECOR loop. As shown in Fig. 1, the plant has a 'figure-of-eight' configuration, typical for a corrosion loop, with a high temperature branch running from the economiser to the test sections and a low temperature branch including the delivery and return pipes between the tank and the economiser, including the by-pass line. The vessel contains a mechanical pump, with submerged impeller, sized to provide the loop with a maximum liquid metal flow-rate of 4 m^3/h [1].

The three test sections TS1/2/3, containing the specimens to be investigated are placed downstream of the electrical heater and can contain up to 12 specimens each. Test sections 1 and 2 contain the corrosion specimens; test section 3 contains the solid Mg getter. The use of Mg is described later in the paper. In the steady state, the thermal power

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Table 1
The chemical compositions of the steels (wt%, balance Fe)

Element	Cr	Ni	Mo	Mn	V	Nb	Si	C
AISI 316L	17.3	12.1	2.31	1.8	–	–	0.35	0.02

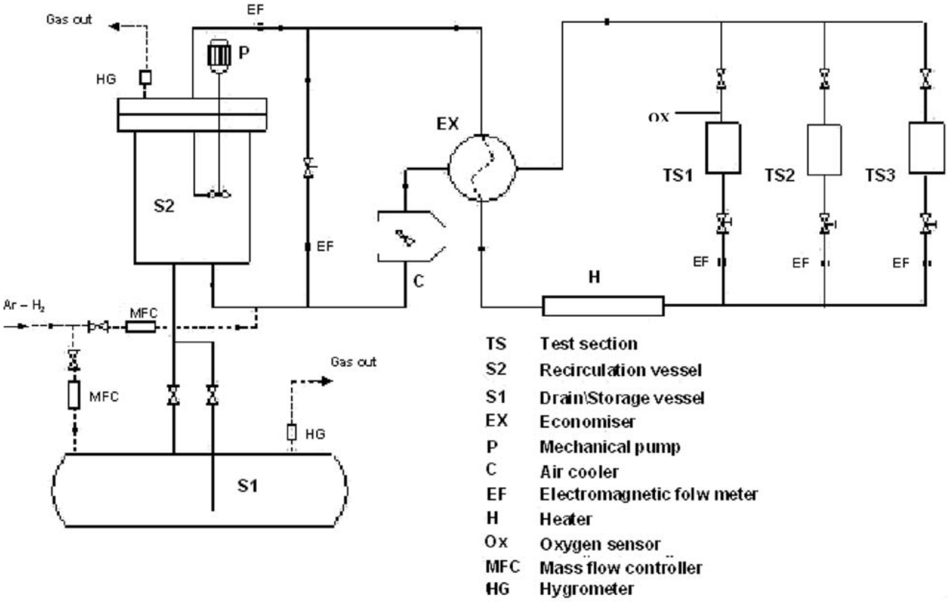


Fig. 1. The LECOR loop.

provided by the heater is equal to the power extracted by the forced air heat exchanger. This configuration enables a continuous transport of corrosion products from the hot region, where the test sections are located, to the cold region where they are partially released, simulating the actual behaviour of coolant fluid in a thermal production plant. The structural materials of the loop are as follows: the cold part is fabricated in austenitic steel, while the hot region is in ferritic steel representing a good compromise between the requirements of acceptable ductility and corrosion resistance in a liquid metal medium.

During normal plant operations the total liquid metal flow-rate available to the test sections is adjusted through the by-pass line, while the liquid metal flow-rate in each test section is automatically controlled by electro-pneumatic regulation valves using feedback signals from electromagnetic flowmeters.

Many different materials are being investigated in the LECOR loop. This paper reports the preliminary results for AISI 316L steel.

The as-received materials were machined to cylindrical specimens with 10 mm diameter and 50 mm length. Before placing the specimens into the test sections of the loop, they were degreased and weighed. The dimensions of each specimen were measured. There was no additional heat treatment to the specimens after machining. The specimens were fitted together in the test sections by fixing their ends with pins. The test section temperatures of the test sections were 450 °C, and the cold branch temperatures of the loops was about 300 °C. The oxygen concentration in LBE were maintained in the range of 10^{-8}–10 wt%, by means of solid stoichiometric Mg addition (which acted as an oxygen getter) directly inside the test section 2 and mixture of Ar/H_2 gas bubbling. The oxygen concentration was measured by an electrochemical sensor (Russian made at IPPE, Obnisk), placed in the TS1, which gave a constant current output of 670–630 mV, corresponding to the above defined concentration range [2–5]. The correct amount of Mg was determined by checking the dissolved oxygen concentration using the electrochemical sensor. The total duration of the tests is forecasted in 4000 h. Three specimens were removed after the loop draining for analysis after 1000 h of exposure time.

After the LBE exposure tests, one specimen of each material has been immersed in a fresh solution (CH_3COOH: H_2O_2: $C_2H_5OH = 1:1:1$) at room temperature to remove the adherent LBE. Specimens were then cleaned and dried for visual examination and weight measurement. Specimens without cleaning were cut and polished for cross section examination by optical microscopy (OM) and scanning electron microscopy (SEM). Thus, the surface that underwent optical analyses was not polished or cleaned. Energy dispersion X-ray spectroscopy (EDS) was used to analyse the chemical composition of the adherent corrosion products and bulk materials.

3. Results

As already said, the preliminary data shown in this paper are related to AISI 316L steel specimens. The EDS micrographies do not show deep LBE penetration into the steel matrix (Figs. 1 and 2). Only some areas seem affected by corrosion, with a maximum measured depth of 5 μm (Figs. 3–5). In none of the analyzed surfaces, LBE appears to stick to the steel surface. There are some detachments of thin steel layers, very slightly Cr enriched when compared with unexposed specimens due to elemental migration inside the matrix (Fig. 6). The specimens are being weighted and weight variations will be analyzed in parallel. In some areas, some residual, extremely thin, natural oxides can be

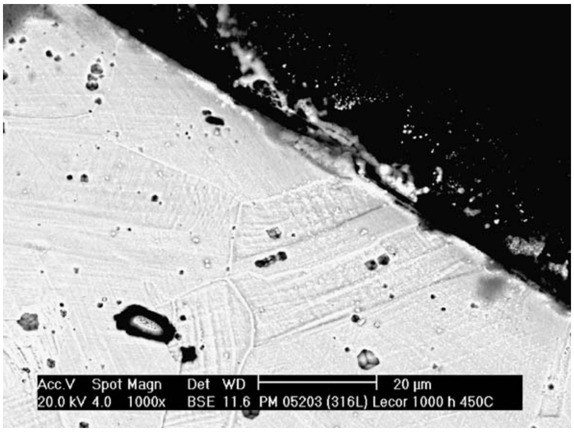


Fig. 2. 20 μm scale SEM micrograph of AISI 316L steel. Exposure time 1000 h, *T*: 450 °C.

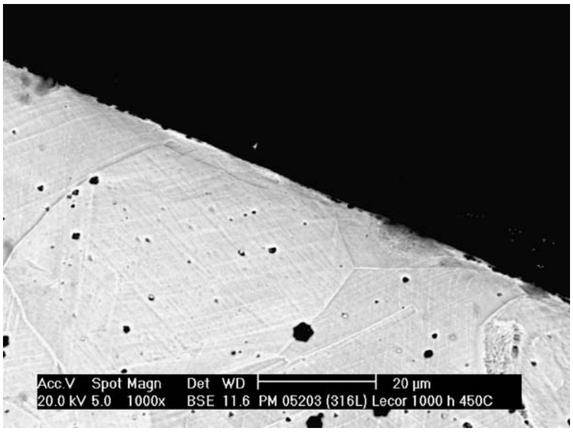


Fig. 3. 20 μm scale SEM micrograph of AISI 316L steel. Exposure time 1000 h, *T*: 450 °C.

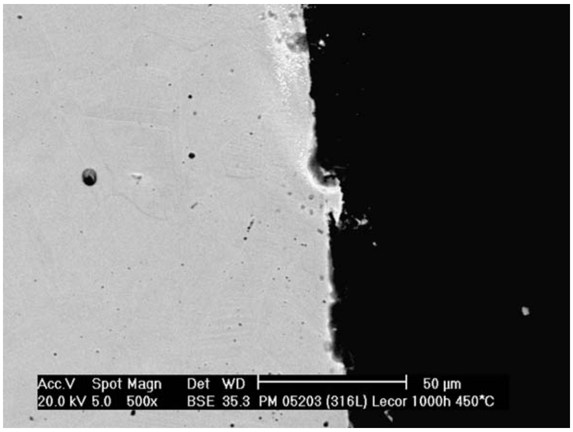


Fig. 4. 50 μm scale SEM micrograph of AISI 316L steel. Exposure time 1000 h, *T*: 450 °C.

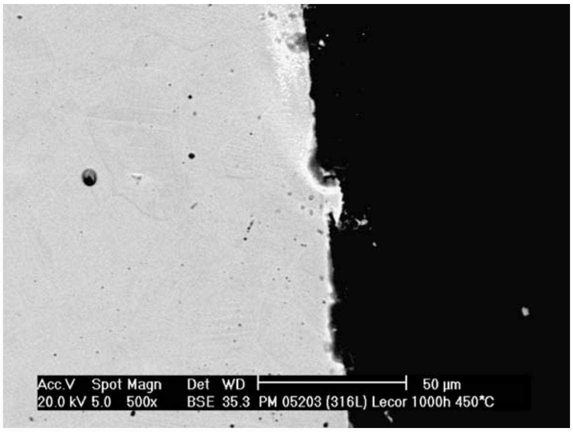
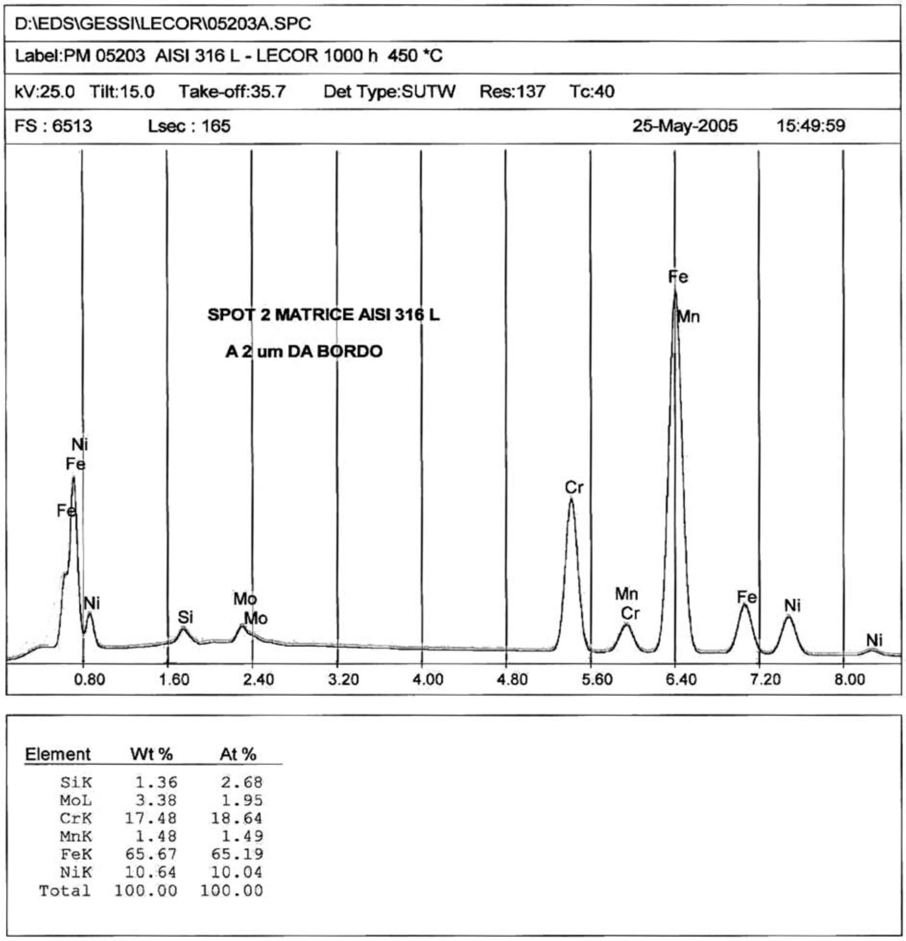


Fig. 5. 50 μm scale SEM micrograph of AISI 316L steel. Exposure time 1000 h, *T*: 450 °C.



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Label:PM 05203 AISI 316 L - LECOR 1000 h 450 °C

kV:25.0 Tilt:15.0 Take-off:35.7 Det Type:SUTW Res:137 Tc:40

FS : 6513 Lsec : 165 25-May-2005 15:49:59

Element	Wt %	At %
SiK	1.36	2.68
MoL	3.38	1.95
CrK	17.48	18.64
MnK	1.48	1.49
FeK	65.67	65.19
NiK	10.64	10.04
Total	100.00	100.00

Fig. 6. EDS spectrum at 2 μm from the boundary layer showing Cr enrichment.

observed. The presence of Mg has been evidenced in a microanalysis, being the consequence of the stoichiometric addiction of the getter itself, directly into the test section.

4. Discussion

The behaviour shown by the AISI 316L steel can be defined as expected. When comparing the EDS micrographs with the ones obtained after the 400 °C test, a similar corrosion effect is observed [6,7]. In both cases the reducing medium started a slow corrosion process, consisting in an elemental dissolution, function of the different steel elements solubility in LBE. The extremely slight Cr enrichment as well as the small Ni depletion of the outer steel layers confirms the effect of this elements higher solubility in LBE when compared with the other steel constituting elements. Thus, in the 400 °C test a clearer ferritization was evidenced [6,7], while in this new experiment the process seems only about to start. This evidence can be related to the smaller time step, (1000 h instead of 1500) that was chosen, in order to acquire a better knowledge of the early phenomena. The extremely slight Cr enrichment together with the Ni depletion can be considered as the result of elemental migration from the centre of the steel matrix to the outer layers.

The LBE did penetrate inside the steel, but without any obviously noticeable preferred path. Again, this behaviour is found also in the 400 °C experiment.

5. Conclusion

The main conclusions are summarized as follows:

- After 1000 h of exposure, a corrosion process based on elemental dissolution has started.

- In only very few areas the LBE penetrated into the steel matrix, down to a maximum of 5 μm, but without preferential paths.
- The comparison with previous experiments gave quite good consistency, even though a direct comparison will be more consistent with a larger time scale.
- The active control/monitoring system for the loop again gave good results, confirmed also by the micro analyses.

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