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# Design of a new experimental loop and of a coolant purifying system for corrosion experiments on EUROFER samples in flowing Pb-Li environment

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The use of ferritic/martensitic RAFM steels in PbLi blanket applications requires a better understanding of material compatibility related to physical/chemical corrosion phenomena in the 450-550°C temperature range. The impact of corrosion includes deterioration of the mechanical integrity of the blanket structure due to the wall thinning. Furthermore, serious concerns are associated with the transport of activated corrosion products by the PbLi coolant. In this frame, the present work aims to illustrate the design of a new experimental facility named LIFUS II (Lithium for FUSion II) intended to extensively investigate corrosion mechanisms related on coated (Al<sub>2</sub>O<sub>3</sub> based) and uncoated EUROFER samples at constant temperature of 550°C, for three different velocities and four different exposure times. Moreover, a “cold trap” purification system is designed to remove impurities and corrosion product dissolved in the liquid metal via upper concentration limits imposed by temperature-dependent solubility constrains.

Keywords: EUROFER corrosion, LIFUS II, cold trap.

## 1. Introduction

The reduced activation ferritic/martensitic (F/M) steel EUROFER is currently considered the reference structural steel for PbLi breeding blankets in future DEMO reactor [1]. Due to the extreme operating condition, EUROFER corrosion and corrosion product transport phenomena constitute strong limitations for the blanket design and for the PbLi loop safety (plugging phenomena and region with high concentration of activated materials). Available experimental data on the corrosion rate for F/M steels in flowing PbLi show a large dispersion, predicting values at high temperatures (450-500 °C) from 5 μm/yr to a few hundred μm/yr [2]. The same issue is observed for the solubility of iron in PbLi: data from different correlations are affected by a dispersion of several order of magnitude [3]. Moreover, detailed information about experimental conditions, such as the PbLi flow velocity and the amount of impurities dissolved in the PbLi alloy, are often missing. This lack of data precludes also a rational design of a PbLi purification system affecting the impurities source term and the maximum solubility value above which precipitation occurs. In this frame new experimental activities are foreseen in order to investigate EUROFER corrosion behaviour for temperatures and velocities significant for the HCLL, WCLL and DCLL Blanket design. The development of a new experimental apparatus, to be realized at the ENEA Brasimone R.C., is here proposed aiming at assessing corrosion rates on uncoated and coated EUROFER samples at a constant temperature of 550°C, for three different velocities 0.01, 0.1 and 1 m/s and four different exposure times 1000, 2000, 4000 and 8000 h.

## 2. Description of the facility

LIFUS II (see the P&ID reported in Figure 1) is a loop type experimental facility designed with a classical “eight shape”. Two test sections (*TS-1,2*) are installed in series in the hot leg of the loop with an operating PbLi temperature of 550°C. On the cold leg, operated at 400°C, are installed the electromagnetic pump (*EP-100*), the induction flow meter (*IFM-105*) and the by-pass to drift a portion of the total mass flow rate towards the cold trap (*CT-101, CT-102*) for the purification process.

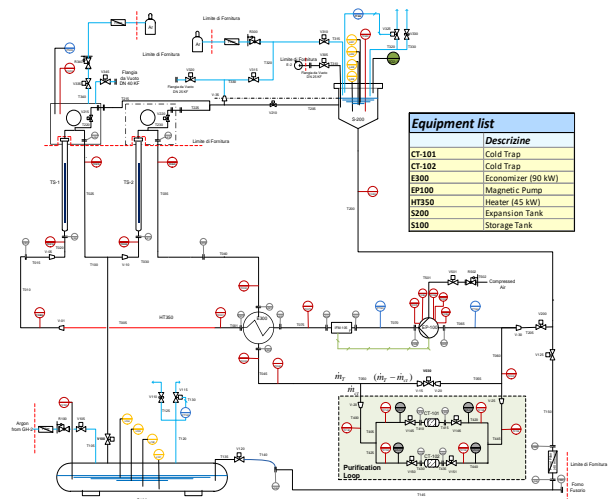
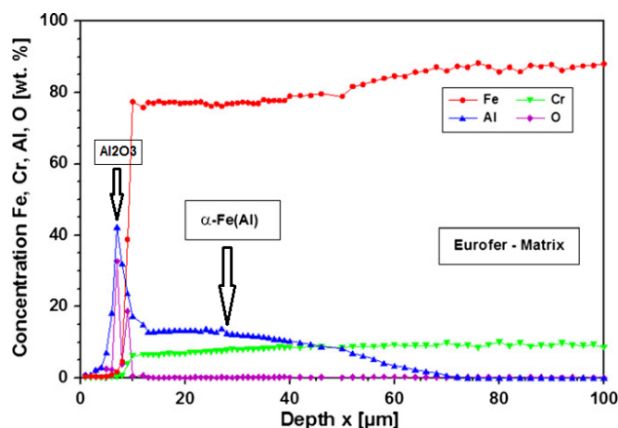


Figure 1: LIFUS II P&ID

The hot and cold legs are coupled through an economizer (*E-300*) consisting in a shell and tube PbLi/PbLi heat exchanger. The hot fluid ( $T_{h,in}$  550°C) enters tube sides and cools down to 450°C at the exit section of the heat exchanger. The cold fluid (400°C) instead, enters shell side and is heated up to 500°C. Considering the total mass flow rate of ~4.6 kg/s and a temperature jump across the economizer  $\Delta T_{ECO} \sim 100^\circ\text{C}$  the heat exchanged is about 87 KW. The PbLi in the shell side is then channelled towards the hot leg where, through a subsequent heating

modules (*HT-350*), reaches the operating temperature of 550°C (required power ~45kW supplied by a series of electrical heating bands installed on a 2" ½ pipe, Figure 1).

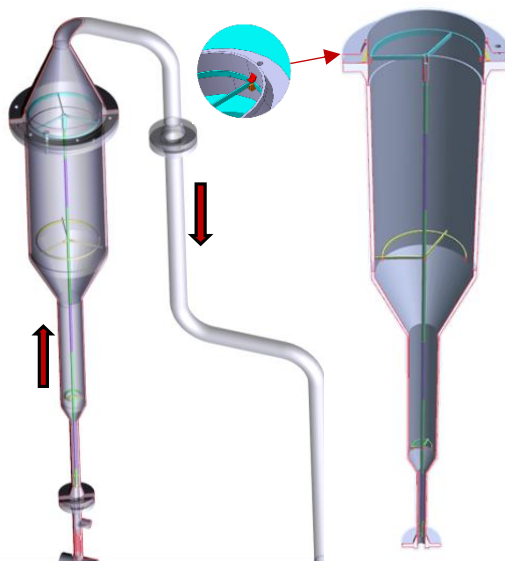
PbLi entering the cold leg needs to be further cooled to reach 400°C. This operation is accomplished splitting the total flow into a portion channelled towards the purification system where it is cooled down (e.g. 270°C), while the other portion at 450°C bypasses the purification system. The flow repartition and the Cold Trap (*CT*) outlet temperature are such that the two partitions re-join downstream the purification zone at a nominal cold temperature of 400°C. Thereby, the *CT* covers the dual function of loop cooling and purification system. Such a *CT* arrangement offers a flexible operation in terms of treated flow, permitting hence, a wider assessment of the purification system efficiency. From a heat balance the *CT* must be designed to remove ~45 kW in order to meet the rated cold leg temperature requirement of 400°C. The desired PbLi mass flow rate is guaranteed by an electromagnetic pump (*EP-100*) with permanent magnet (PMP, alternating travelling magnetic field generated by a rotating magnetic system with permanent magnets poles with alternating polarity) installed downwards the cold trap and operating at a temperature of 400°C. An induction mass flow meter (*IFM-105*) is used to monitor and control the loop mass flow rate. The reference structural material for piping and components (*HX Economizer, Test Sections, etc.*) is steel 2 ¼ Cr-1 Mo (*ASTM A335 Gr. P22*), whose relatively low corrosion rate (low Nickel content and consequent low degradation by precipitation) contributes to minimize the corrosion products dissolved in the alloy. The main pipeline of the circuit consists of 2" Sch. 80 XS pipes, which guarantees a relatively low PbLi piping velocity (0.25 m/s) minimising the pipeline corrosion. The circuit is completed by the storage vessel *S-100* with filling and draining lines, the expansion vessel *S-200* and a removable glove box installed above the test sections to ensure the specimens extraction in a controlled and protected atmosphere avoiding any contamination of the loop with oxygen during this phase. In order to reduce the plugging risk, together with the *CT* component design, a coating treatment of all the structural steel in PbLi is taken into consideration. According to [4-6], the *Al*-based coating suitable for operation in PbLi is a diffusion double layer where the external layer consists of the *Al-Fe* phase and the internal layer consists of  $\alpha$ -*Fe(Al)* phase. Above the double layer there is also a thin  $Al_2O_3$  layer which provides an enhancing protection feature. *Fe-Al* and, in particular,  $\alpha$ -*Fe(Al)* and  $Al_2O_3$  are the protective phases of the coating since they have low solubility in PbLi. Other *Al*-enriched phases (such as  $FeAl_2$  and  $Fe_2Al_3$ ) are unwanted and must be avoided since they are brittle and soluble in the liquid metal. An example of good protective coating for operation in PbLi is reported in [6], where the compositions of *Al* within each *Al*-phases are reported in Figure 2. Clearly visible are the external  $Al_2O_3$  layer with 40% of *Al* and the  $\alpha$ -*Fe(Al)* phases with about 15% of *Al*. The coating thickness is 50 µm.



**Figure 2: Composition of Al, Fe, Cr and O within an Al-based diffusion coating above the EUROFER steel**

### 3. Test section

Two identical test sections (*TS-1* and *2*, Figure 1 and Figure 3), vertically arranged placed in sequence and working at constant temperature, are foreseen in the hot leg of the loop (550°C). The first *TS* will be adopted for short and medium-term tests (1000, 2000, 4000 h) while the second one will be used for long term test (8000 h). Each *TS* is designed with three specific cross section characterized by increasing the inner diameter (1", 3" and 10") in order to provide the requested velocities inside each *TS* (respectively 1.0, 0.1 and 0.01 m/s).



**Figure 3: Test Section**

For each velocity section, a set of 6 specimens are foreseen (3 uncoated + 3 coated with outer diameter of 10 mm and a length of 37 mm (M5), screwed together to form a rod to be inserted inside the test section). Dummy elements are foreseen in the *TS* region where cross section variation occurs. The samples rod will be fastened in the upper part of the *TS* while kept free to expand in the lower part to avoid stresses due to different thermal expansions. In Table 1 the main parameters that characterizes the *TS* geometry and the flow conditions are reported.

**Table 1: TS main parameter**

Quantity	Unit	Sec-1	Sec-2	Sec-3	NOTE
$v$	m/s	1	0.1	0.01	Section velocity
$A_p$	m <sup>2</sup>	4.79 10 <sup>-4</sup>	4.69 10 <sup>-3</sup>	4.81 10 <sup>-2</sup>	Flow Area
$D_i$	m	26.64 (1" Sch.40)	77.92 (3" Sch.40)	247.65 (10" Sch.60)	Sec inner diameter (Size)
$d_h$	m	1.66 10 <sup>-2</sup>	6.79 10 <sup>-2</sup>	2.38 10 <sup>-1</sup>	Hydraulic diameter
$L$	m	0.304	0.466	0.55	Section length
$Re$	-	1.56 10 <sup>5</sup>	65055	22199	Reynolds number
$m$	kg/s	4.6			Mass flow rate
$\mu$	Pa s		1.02 10 <sup>-3</sup>		PbLi viscosity at 550°C
$\rho$	kg/m <sup>3</sup>		9541.5		PbLi density at 550°C
$d$	m		0.01		Sample outer diameter

The proposed corrosion-resistant coating is an Aluminium-based layer consisting of an amorphous matrix of  $Al_2O_3$  with nano-crystalline inclusions obtained by means of *PLD* (Pulsed Laser Deposition) technique in the *CNST* (Center of Nano-Science and Technology) laboratories at *IIT* (Italian Institute of Technology) [7,8].

#### 4. Purification system

The *CT* system consists of a heat and mass transfer device, where a supersaturated solution of impurities is generated as the result of PbLi cooling, causing the crystallization of the impurities both on the immovable surfaces and in suspension. The principle of the *CT* is to maintain the impurity equilibrium concentration in the loop below the PbLi solubility at 400°C (cold zone temperature). Such an apparatus has the purpose of collecting the impurities generated during the operation, avoiding therefore, the corrosion products precipitating in the loop. Although the purification principle is relatively simple, the design difficulties of a cold trap are significant and are related to the lack of reliable experimental data or empirical correlation on:

- steel components solubility in PbLi (e.g. *Cr* and *Fe*);
- the kinetics governing the precipitation phenomena;
- the total source term of the corrosion products.

For a generic system solute/solvent system with a source term and provided with a purification apparatus, the impurities concentration  $C(t)$ , in ppm, can be obtained from a balance equation:

$$C(t) = C^\infty \left( 1 - e^{-\frac{\eta \dot{m}_{ct}}{M} t} \right) \quad (1)$$

where  $M$  is the PbLi total mass in the system (kg),  $\dot{m}_{ct}$  is the mass flow in the *CT*,  $\eta$  the *CT* efficiency and  $C^\infty$  the asymptotic concentration ( $t \rightarrow \infty$ ) defined as:

$$C^\infty = \left( \frac{S}{\eta \dot{m}_{ct}} + C_{ct}^{Sat} \right) \quad (2)$$

Where  $S$  is the source term ( $\mu\text{g/s}$ ),  $C_{ct}^{Sat}$  represents the iron solubility at the minimum *CT* temperature,  $T_{ct}$  (being the iron the main component). Concerning the *CT* efficiency, it can be generically defined as:

$$\eta = \frac{C_{in} - C_{out}}{C_{in} - C_{ct}^{Sat}} \quad (3)$$

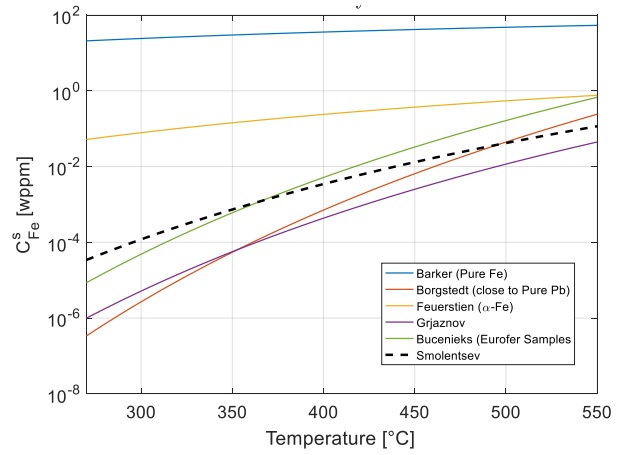
and in an initial assessment, it is assumed to have the same form of the *CT* efficiency defined for the sodium purification system:

$$\eta = \frac{1}{1 + p\tau^q} \quad (4)$$

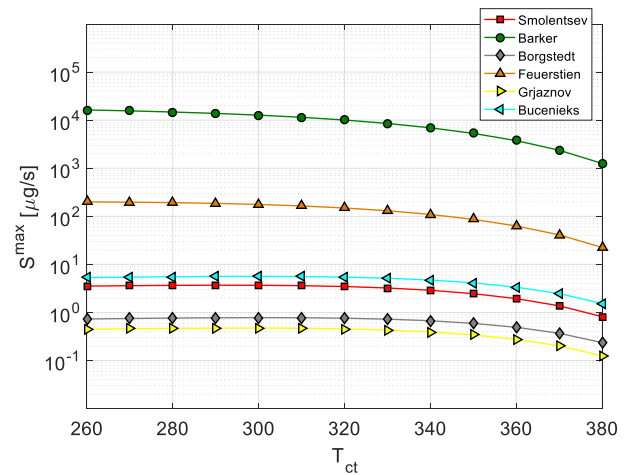
where  $\tau$  represents the fluid resident time (min) in the *CT* and  $p$ ,  $q$  are coefficients set equal to 122 and 3.4 respectively for sodium *CT* [10]. The appropriateness of such a correlation for PbLi corrosion system will be evaluated experimentally with chemical analysis of PbLi sampling upstream and downstream the *CT*.

In a preliminary design, the available correlations for determining iron solubility in PbLi (Figure 4) were investigated to determine the maximum corrosion source as a function of  $T_{ct}$ , (Figure 5) obtained from eq. (2) imposing  $C^\infty < C_{400^\circ\text{C}}^{Sat}$  in order to avoid any metal precipitation in the cold zone of the loop ( $T_{cold}=400^\circ\text{C}$ ):

$$S^{\max} = \eta \dot{m}_{ct} \left( C_{400^\circ\text{C}}^{Sat} - C_{ct}^{Sat} \right) \quad (5)$$



**Figure 4; Iron solubility in PbLi**



**Figure 5: Maximum allowable corrosion source**

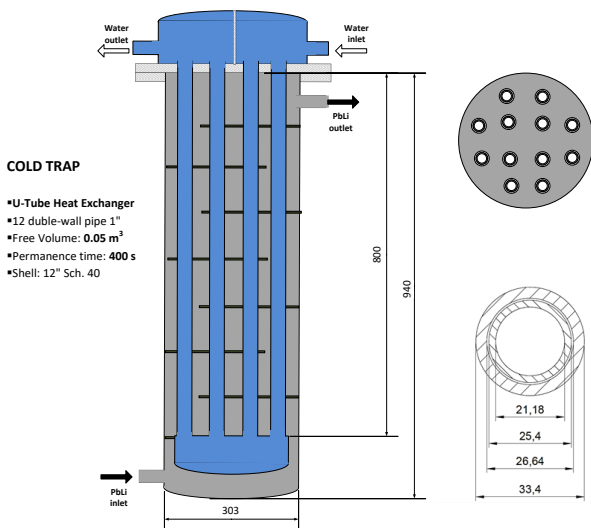
According to the Smolentsev [2] correlation, chosen as reference, the maximum source term allowable in order to avoid precipitation of corrosion products in the cold leg of the loop ( $T \sim 400^\circ\text{C}$ ) for a *CT* temperature in the range 270-300°C is about 3.6  $\mu\text{g/s}$  (0.11 kg/yr).

The saturation concentration significantly decreases in the range from 450 to 250°C, for this reason a minimum *CT*

temperature of  $T_{CT}=270^{\circ}\text{C}$  is fixed to keep an acceptable safety margin from the solidification temperature ( $235^{\circ}\text{C}$ ). For  $T_{CT}=270^{\circ}\text{C}$  the minimum flow bypassed in  $CT$  is about  $1.3\text{ kg/s}$  (being the  $CT$  cooling power fixed to about  $45\text{ kW}$ ). The maximum temperature in the  $CT$  is  $400^{\circ}\text{C}$  if we consider to process all the  $\text{PbLi}$  mass flow rate.

Another significant parameter to be defined is the  $CT$  residence time,  $\tau$ , affecting the deposition kinetics and therefore the  $CT$  efficiency. This parameter depends on the  $CT$  volume,  $V_{CT}$  and is inversely proportional to  $\dot{m}_{ct}$ . The value of  $V_{CT}$  has been set to about  $50\text{ l}$ , in order to obtain a residence time maximum value  $\tau \approx 400\text{ s}$  (for  $\dot{m}_{ct} = 1.3\text{ kg/s}$ ) and a minimum value  $\tau \approx 110\text{ s}$  (for  $\dot{m}_{ct} = 4.6\text{ kg/s}$ ). Regarding the layout of the  $CT$ , a shell and tube heat exchanger with 2-pass tube side configuration is proposed.  $\text{PbLi}$  enters, shell side, at  $450^{\circ}\text{C}$  and exits at  $270^{\circ}\text{C}-400^{\circ}\text{C}$ . The inner diameter of the shell is  $303.23\text{ mm}$  (12" Sch. 40) and the velocity of the  $\text{PbLi}$  is in the range  $2-6\text{ mm/s}$  respectively for  $1.3$  and  $4.6\text{ kg/s}$ . A series of baffle assures both an effective liquid metal circulation and an increased  $CT$  precipitation surface. The preliminary sketch of the  $CT$  is reported in Figure 6.

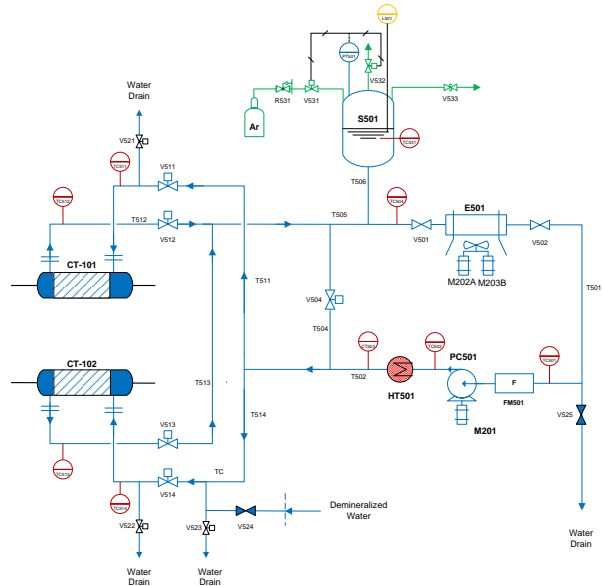
The purification system will be arranged in a parallel configuration in order to ensure the continuous operation of the experimental facility (if needed, the flow will be diverted in the second  $CT-102$ , while the first one ( $CT-101$ ) can be drained and disassembled for maintenance or reconditioning, e.g. removal of the plugged impurities). Upstream and downstream the  $CT$ s a sampling devices is foreseen to extract the circulating  $\text{PbLi}$  and to analyze its impurities content aiming at assessing the  $CT$  efficiency.



**Figure 6: Preliminary CT configuration**

The secondary side of the purifying system consists in a pressurized water loop (see the P&ID Scheme in Figure 7). The pressure of the secondary loop is set to  $20\text{ bar}$  ( $T_{sat}=212^{\circ}\text{C}$ ), the inlet and outlet water temperatures are  $160^{\circ}\text{C}$  and  $193^{\circ}\text{C}$  respectively, with a water mass flow rate range of  $0.2-1\text{ kg/s}$ . The water flows through a bundle of  $12$  ( $U$ -tube configuration), double-wall tubes with a stainless steel powder filling the gap between the tubes (Figure 6). This geometry provides a double

physical separation between the two fluids that allows the thermo-mechanically decoupling of the two tubes through the gap. The inner tube is a 1" 14BWG pipe while the outer tube is 1" Sch.40. The gap thickness is  $0.62\text{ mm}$  with a powder heat transfer coefficient about  $1/10$  of that of the stainless steel [9].



**Figure 7: Water secondary system**

The Air Cooler,  $E-501$ , is designed to remove the power from water secondary fluid while, the heating system  $HT-501$  is considered in order to pre-heat the water at the operating conditions.

## 5. Conclusions

A new facility named LIFUS-II, to be constructed at ENEA Brasimone RC, has been designed to investigate the RAFM/ $\text{PbLi}$  corrosion aspects for the current BBs concepts. The LIFUS-II experimental campaign will be devoted to evaluate the corrosion rate of the reference steel and the chemical compatibility of a new  $Al$ -based coating proposed as anti-corrosion and anti-permeation barrier in flowing  $\text{PbLi}$ . Therefore, uncoated and coated EUROFER samples will be exposed to different  $\text{PbLi}$  flow velocities ( $1.0$ ,  $0.1$  and  $0.01\text{ m/s}$ ) at a temperature of  $550^{\circ}\text{C}$ . Experimental data will be collected at different exposure times ( $1000$ ,  $2000$ ,  $4000$  and  $8000\text{ h}$ ).

Finally, a  $CT$  preliminary design is proposed together with the sampling system to maintain impurities concentration below the cold zone saturation point, avoiding precipitation in the coldest part of the loop and providing detailed information about experimental conditions. Moreover, the experimental campaign will be useful in order to assess the parameters affecting the purification efficiency.

## Acknowledgments

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