INFLUENCE OF PBBI EUTECTIC ON THE CRACK INITIATION IN 316L AND T91 STEELS

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Abstract

Crack initiation of the ferritic martensitic steel T91 and austenitic steel 316L is currently under study. This is due to the evaluation ongoing in the EC for materials compatibility in Heavy Liquid Metals (HLM) environment, and, in particular, the issue of Liquid Metal Embrittlement.

Slow Strain Rate Tensile (SSRT) tests were performed with flat specimens in PbBi at 300°C with oxygen content in the range of about 10^{-6} wt.% down to 10^{-12} wt.% and, for comparison in air, up to the point of maximal stress (without or with minimum necking). Tests were performed with flat tapered specimens, which were meant to create a uniform variation of stress along the gauge length, with the maximum stress concentrated in the smallest cross-section area.

The cracking mode of T91 changed due to environment influence when compared to the testing in air, however the austenitic 316L seemed unaffected, even though at the lowest oxygen content, the crack numbers and morphology changed. Post-tests examinations were carried out with a Scanning Electron Microscope.

Keywords: Ferritic-martensitic steel, austenitic steel, lead-bismuth eutectic, tapered, crack initiation

1. INTRODUCTION

Lead bismuth eutectic (LBE) is a potential heat transfer liquid for future Gen-IV reactors. The Multi-purpose hYbrid Research Reactor for High-tech Applications (MYRRHA), currently under development in Belgium uses LBE as cooling liquid as well as spallation target [1]. One critical factor in the development is therefore the compatibility of structural materials with the cooling liquid. Particularly Liquid metal embrittlement (LME) is an important topic in the recent years [2] and it might lead to failure of components typically associated with a change from ductile to cleavage-like fracture mode [3].

Austenitic steel of 316 type belongs to one of the main austenitic alloy classes that has a good combination of strength, ductility, and toughness at low and high temperature, along with good formability, weld ability, and corrosion properties. The 316 steels also have reasonably good creep resistance at high temperatures. The ferrite content of these steels is an important point since ferrite aging at high temperatures can lead to some brittle phase precipitation and to a toughness decrease. Made of type 316 austenitic stainless steels are pressure boundary pipes and the primary circuit of pressurized water reactors. It is used as a manufacturing material for nuclear fuel clad tubes and fuel sub assembly wrappers in fast breeder reactors owing to its superior mechanical properties at elevated temperatures and good compatibility with liquid sodium [4]. The expertise on compatibility of stainless steels with sodium is not transferable to lead and lead alloys, due to the significant differences in their physics and metallurgic properties. Therefore, particular studying of materials in heavy liquid metals is necessary.

The Ferritic-martensitic steel T91 has, together with good corrosion resistance, excellent radiation and swelling resistance in fast neutron flux and high temperature mechanical properties. Over the last decades extensive work has been conducted to investigate the LME characteristics of T91 under a wide variety of conditions.

The main task of this paper is to provide an insight into crack initiation and to examine the material susceptibility to liquid metal embrittlement (LME) for steels T91 and 316L.

2. EXPERIMENTAL

2.1. Material

Ferritic-martensitic steel T91 (Grade 91 Class 2/S50460) of nominal composition (wt. %) Fe-8.9Cr-0.9Mo-0.4Mn-0.2Si-0.2V was produced by Industeel, Arcelor Mittal group. The steel was austenitized at 1050 °C with a holding time of 1 min/mm followed by water quenching. This was followed by an annealing treatment at 770 °C with a holding time of 3 min/mm followed by air cooling. The typical microstructure formed by this heat treatment consists of laths of martensite and original austenitic grains. The material properties at 300 °C according to EN ISO 6892 are: Ultimate tensile strength (UTS) 602 MPa, Yield strength (YS) 499 MPa.

Austenitic steel 316L (ASTM A240-Ed02) was produced by Industeel, Alcelor Mittal group. The austenitic stainless steel was received as hot rolled and heat treated plates with a thickness of 15 mm. The solution annealing was done at 1050-1100 °C with a goal to reach a homogeneous microstructure as well as a homogeneous distribution of mechanical and corrosion properties compared to the hot rolled 316L. The typical microstructure is fully austenitic. The material properties at 300 °C according to EN ISO 6892 are: UTS 443 MPa, YS 181 MPa.

2.2. Specimens and procedure

Electrical discharge machining (EDM) was used to fabricate the specimen (**Figure 1**). One of the two parallel surfaces was ground to 500-grid finish and the other was polished to 1μm finish. The two nonparallel surfaces (the thin side) stayed as received after EDM.

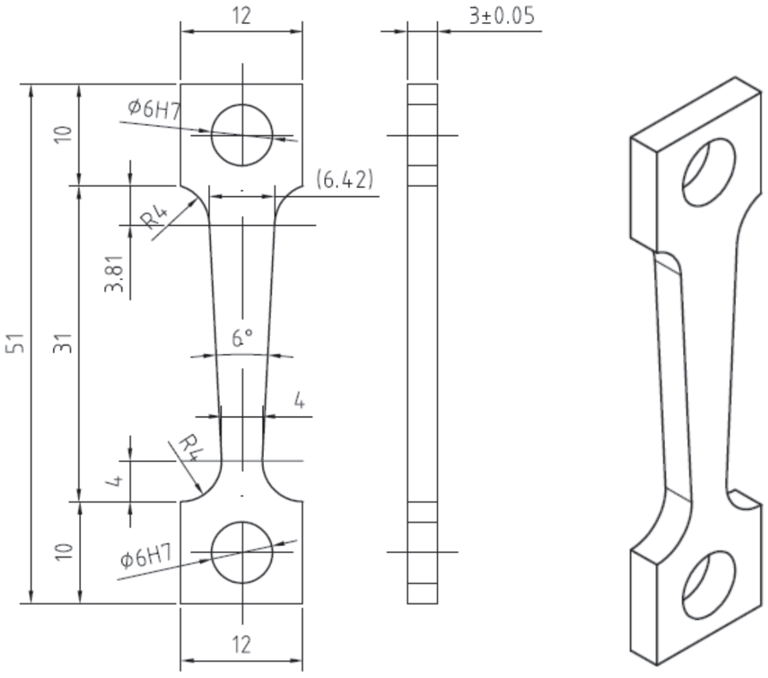

Figure 1 - Tapered specimen

The specimens were fitted into the testing cell for HLM environment. Whole cell+specimen alignment is placed in the Kappa 50DS, electromechanical creep testing machine from Zwick/Roell group. The testing space is protected by the overpressure of the inert atmosphere. The testing vessel is equipped with a gas regulation system and Bi/Bi_2O_3 oxygen sensors, which form together a system for the oxygen regulation inside the liquid metal. The system regulates the oxygen amount during the whole test procedure in the required limits.

The specimens were tested in tensile mode with constant extension rate 0.0012 mm/min, which is approximately equal to strain rate 10^{-6} s^{-1}. Both materials were tested at the same temperature (300 °C). Each material was tested in two environments (air and LBE with regulated oxygen amount) for comparison of the environmental influence. Tests were ended at two different conditions (at maximal applied load or after rupture). The complete test matrix is summarized for general overview in **Table 1**.

Table 1 - Test Matrix of the SSRT tapered specimens of steels T91 and 316 L

Steel	**Design.**	**Environment**	**Test end**	**T [°C]**	**Strain Rate [s^{-1}]**	**O [wt.%]**
T91	T3	Air	rupture	300	10^{-6}	-
	T1	LBE				6×10^{-6}
	T8	LBE				3×10^{-13}
	T9	LBE				10^{-8}
	T4	Air	max. load			-
	T10	LBE				2×10^{-8}
	T11	LBE				3×10^{-7}
316L	L3	Air	rupture			-
	L2	LBE				3×10^{-8}
	L6	LBE				3×10^{-12}
	L4	Air	max. load			-
	L5	LBE				2×10^{-9}
	L7	LBE				3×10^{-12}

Specimens were cleaned from LBE with a mixture of $C_2H_6O+H_2O_2+CH_3COOH$ (1:1:1). After exposure, the specimens were analyzed in a TESCAN Mira 3 - FEG SEM with Oxford Instruments EDX detector (Parameters of each SEM observation are highlighted in the figures for each specific specimen).

3. RESULTS

3.1. SSRT tests

SSRT tests (**Table 1**) are shown as load-displacement curves in **Figures 2** and **3**. The environment did not have any effect on maximal load neither on T91 nor 316L steels. However the measured displacement of the T91 specimens differs and it is decreasing with decreasing oxygen content. The T91 elongation decreasing occurs after reaching maximal load but no further decrease was observed after reaching 10^{-8} wt.% of the oxygen amount. The results from 316L stainless steel elongation show a negligible influence of the oxygen amount with respect to absolute values and the deviations, which is probably caused by using two machines with different compliances.

The stress values due to maximal applied load are between 394±7 MPa (the widest area of the tapered specimen) and 632±12 MPa (the narrowest area) for T91 steel, between 308±6 MPa (the widest area) and 494±10 MPa (the narrowest area) for 316L steel. This is valid for specimens tested up to UTS, though it cannot be applied for specimens loaded up to rupture.

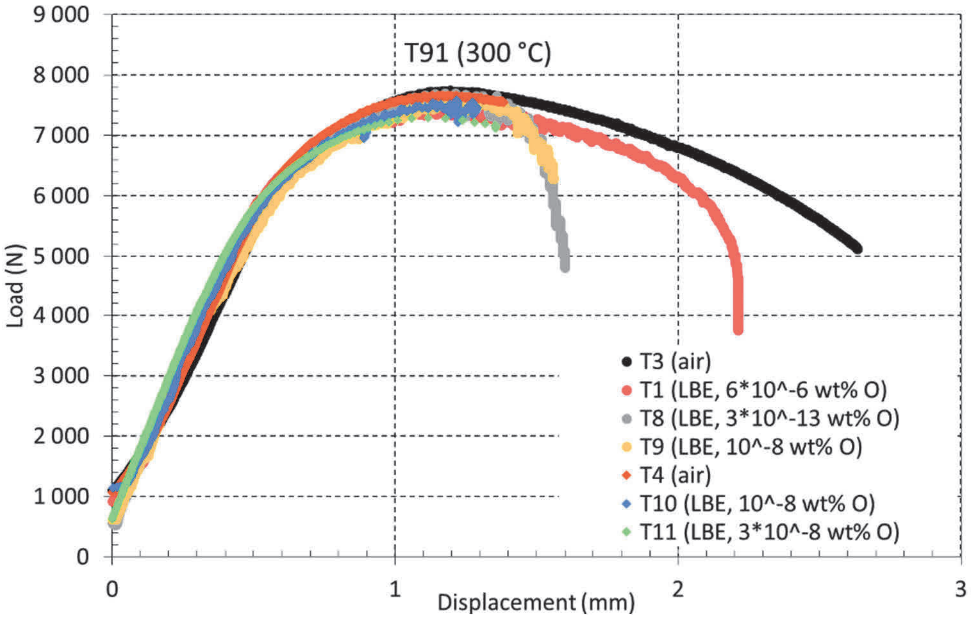

Figure 2 - Load-Displacement curves for ferritic-martensitic steel T91

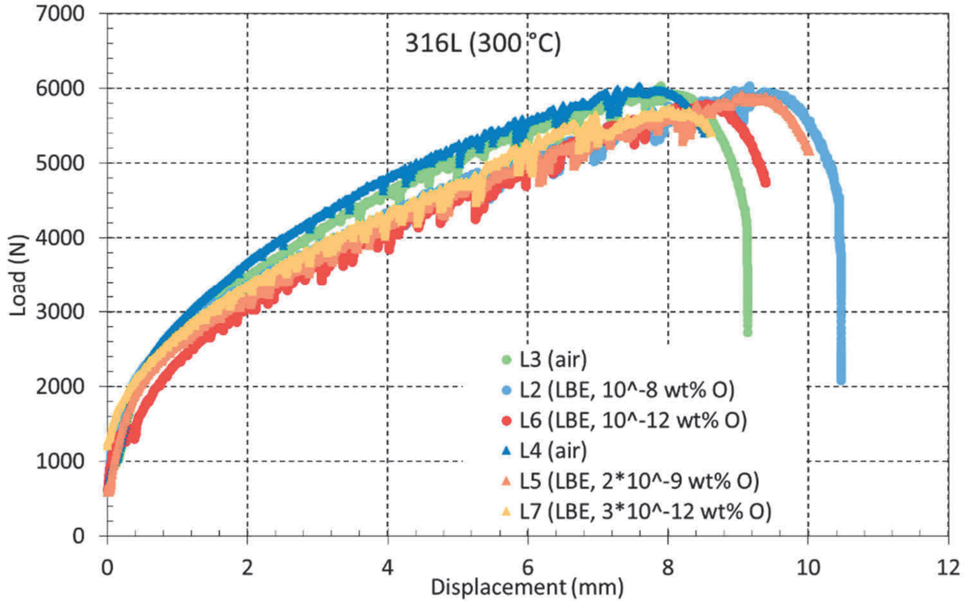

Figure 3 - Load-Displacement curves for austenitic steel 316L

3.2. SEM examination

On the edge of the ground-EDM surface, up to approx. 1.5 mm from the fracture surface, several microns long plastic cracks (up to 25μm deep) were observed on the T91 specimen tested in air (**Figure 4, right**). On the specimen T1, tested up to rupture in LBE, deeper cracks (up to 200μm) were observed (**Figure 5, right**). Moreover, on the surface of the specimens, small cracks were observed: in air (**Figure 4, left**), small cracks were observed to start along slip planes and around precipitates; in LBE (**Figure 5, left**), several thin cracks were observed, as a result of the oxide damage and slightly bigger developed also around precipitates. The dimensions of the cracks in both environment are comparable,

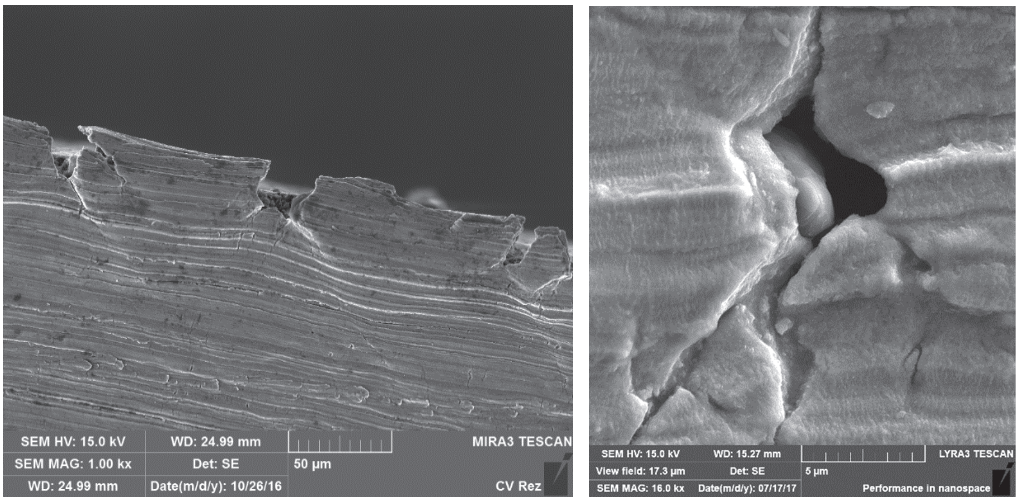

Figure 4 - Specimen T3, rupture (300°C, air), (left) detail of cracks at the edge (right) detail of a crack on the specimens ground surface

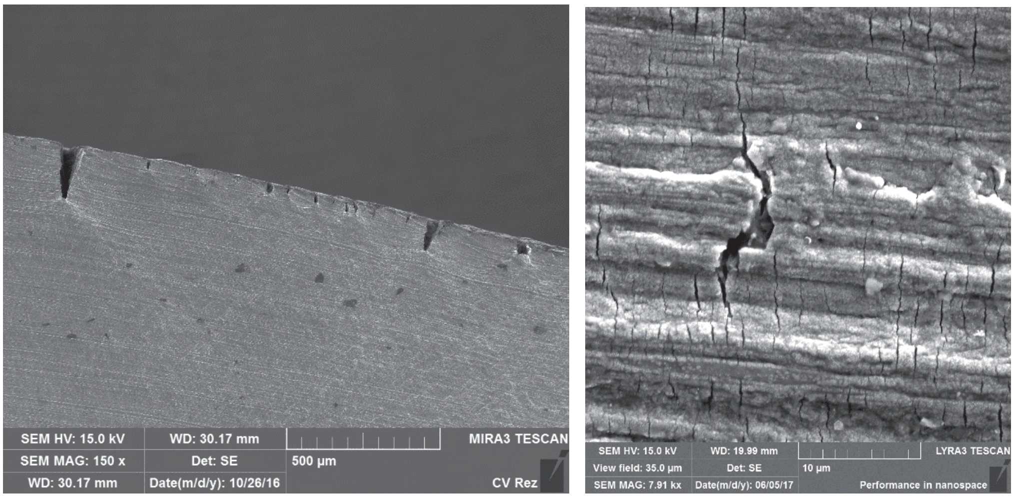

Figure 5 - Specimen T1, rupture (300 °C, LBE, $6 \cdot 10^{-6}$ wt% O), (left) detail of cracks at the edge (right) detail of a crack on the specimens ground surface

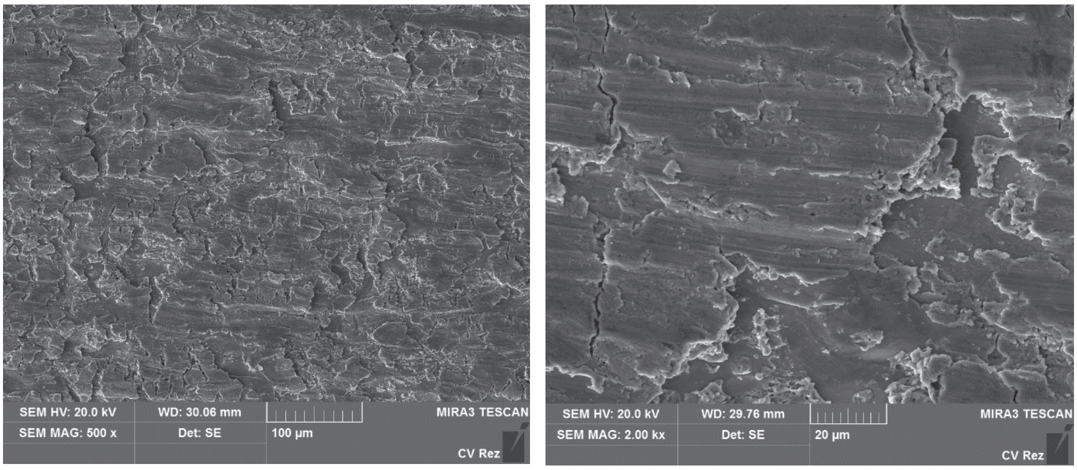

Figure 6 - Specimen L3, rupture (300 °C, air),
(left) general vies of cracks of the surface (left) detail of cracks characteristics

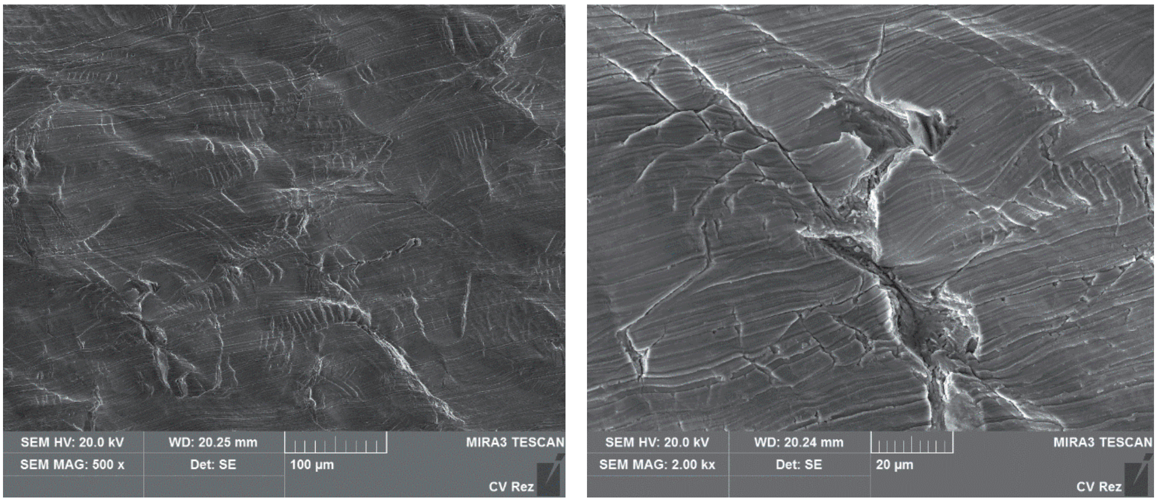

Figure 7 - Specimen L7, to UTS (300 °C, LBE, $3{\cdot}10^{-12}$ wt% O), (left) detail, the necking area - distribution of cracks, (right) detail of a crack

Austenitic steel 316L has minimum cracking on the edges after testing in air (**Figure 6**), only localised cracking was observed on the specimens stopped at the maximal load (**Figure 7**). Cracks were observed independently of the environment. Their initiation was connected with the formation of slip planes and the local presence of precipitates.

The clearest evidence of LME was observed only in the case of the specimen T1 (as well as T8 and T9), where fast growth and earlier failure of the specimen occurred. This difference was not observed for the 316L in these experimental conditions. These observations are suggesting that crack initiation is not a critical issue for the ferritic-martensitic steel, up to the UTS, even if cracks may start at lower load. For the crack to grow critically and the occurrence of LME, plastic deformation after the UTS is necessary.

4. CONCLUSION

Tapered specimens of austenitic 316L and ferritic-martensitic T91 steels were loaded in air and PbBi at 300°C and at the constant strain rate:

- The test results of T91 steel show crack faster growth after the UTS. The influence of LBE- oxygen content on UTS was negligible, however, the measured displacement to rupture was different and it decreased with decreasing oxygen content. Plastic strain, after the UTS is a necessary condition for a critical crack propagation, LME.
- The results from 316L stainless steel showed crack initiation in both environments and experimental conditions. The influence of LBE and oxygen amount on UTS value was negligible, although the measured displacement of the specimens was always higher in LBE compared to air. Crack initiation was affected by the surface finish, but it does not appeared to be affected by the environment (nor the growth).

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REFERENCES

[1] ABDERRAHIM, H. A., "Multi-purpose hYbrid Research Reactor for High-tech Applications a multipurpose fast spectrum research reactor," *Int. J. Energy Res.,* vol. 36, pp. 1331-1337, 2012.

[2] OECD/NEA, Handbook on Lead-bismuth Eutectic Alloy and Lead Properties, Materials Compatibility, Thermal-hydraulics and Technologies, 2015, pp.17-23

[3] LYNCH, S. P., Metal-induced embrittlement of materials, *Materials Characterization, 1992,* 28, 279-289

[4] DESU, R., KRISHNAMURTHY, H. N., BALU, A., GUPTA, A. and SINGH, S., "Mechanical properties of Austenitic Stainless Steel 304L and 316L at elevated temperatures," *Journal of Materials Research and Technology.* 2016. no. 5, p. 13-20.