MECHANISMS OF CRACK INITIATION IN FERRITIC MARTENSITIC AND AUSTENITIC STEELS IN PBBI

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Abstract

The ferritic martensitic steel T91 is among the candidate materials for internal structural components of future Heavy Liquid Metal cooled reactors. The position of T91 on the list of the candidate materials is now being questioned due to the evidence of its sensitivity to liquid metal embrittlement (LME), mainly in PbBi eutectic. Therefore, a renewed interest is currently focused on austenitic steels, in particular on 1.4970 steel, as there is a lack of evidence of LME for them.

The main goal of this work was to provide an insight into crack initiation phenomena in Liquid Metals for the two different steels and the evaluation of possible mechanisms initiating and propagating the cracks.

Slow Strain Rate Tensile (SSRT) tests were carried out with flat specimens in PbBi at 300 °C with low oxygen content (about 10^{-8} wt. %) and, for comparison in air, up to the Ultimate Tensile Stress (UTS) point. 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 area. This specimen geometry can be used for determination of the threshold stress of the crack initiation.

The behaviour of the two materials was affected by the environment, as their cracking mode changed in PbBi when compared to the testing in air. Post-tests examinations were carried out with a Scanning Electron Microscope (SEM) equipped with Focused Ion Beam (FIB). The role of oxides formation and microstructure is discussed and correlated to the initiation of cracks.

Under these experimental conditions 1.4970 steel was not affected by the environment, as small plastic cracks were observed around the Ti-rich precipitates in both air and PbBi.

By contrast T91 steel showed a market tendency to the crack initiation and rapid growth in PbBi.

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

1. INTRODUCTION

Materials interaction with Heavy Liquid Metals has been a topic of wide interest for several years [1] because of worldwide interest on developing HLM-cooled nuclear reactors. This work focuses on the fundamental understanding of steel cracking conditions in HLM. In particular, two reference steels were considered and compared.

The phenomenon called Liquid Metal Embrittlement (LME) is characterized by the tendency of structural materials to low energy fracture under stress in contact with liquid metals; the phenomenon is typically associated with a change from ductile to cleavage-like fracture mode [2]. Extensive investigations have been conducted to study its characteristics and to understand the mechanism, but there are still many open questions on the initiation and mechanisms of propagation.

2. EXPERIMENTAL

*Material.*The 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 material was normalized at

1150 °C for 15 min with subsequent water cooling to the room temperature and finally annealed at 770 °C for 45 min, slow cooled in the air. The typical microstructure formed by this heat treatment consists of laths of martensite and original austenitic grains.

Austenitic 1.4970 steel (also known as 15-15Ti) of nominal composition (wt. %) Fe-15.95Cr-15.40Ni-1.49Mn-1.20Mo-0.52Si-0.44Ti was produced by Sandvik. The material was provided in from of a bar which was taken from intermediate step of a thin wall cladding tube production. The final step of the treatment for the supplied bar was homogenizing heat treatment at 1200 °C for 24 hours, reheating to 1240 °C-1260 °C and hot forging by hydraulic press. The microstructure of the section from which the specimens were machined had large grains and contained numerous large intergranular Ti-rich precipitates.

Specimens. Flat tapered specimens (**Figure 1**) [3] were fabricated by wire cutting using electrical discharged machining (EDM). One of the two large parallel surfaces was ground to 500-grid finish and the other one was polished to 1μm finish before testing.

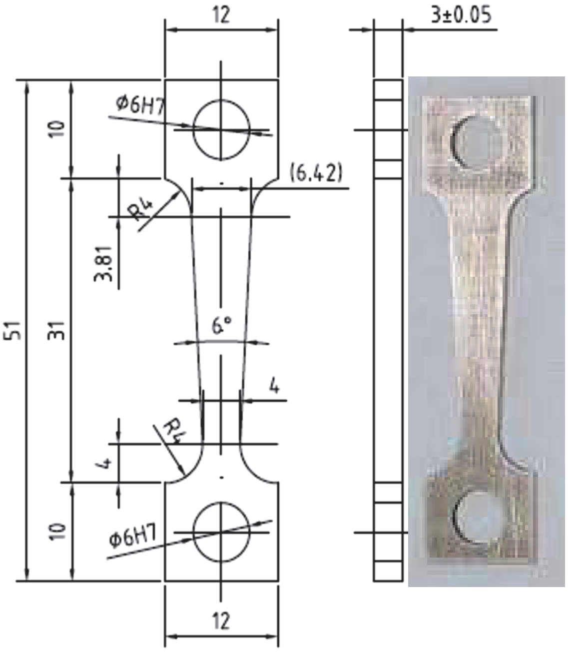

Figure 1 Tapered specimen

Experimental procedure. The tapered specimens were monotonic tensile loaded in the CALLISTO cell, a vessel containing PbBi (LBE) built on a Zwick/Roell Electromechanical Creep Testing machine, Kappa 50DS. CALLISTO is based on the 2-vessel concept, where the first container serves for preparation of the liquid metal (gas dosing). The liquid is then transferred to the second tank, where testing of the specimen is performed. **Table 1** contains the test matrix, giving a general view of the two materials being tested at the same temperature (300 °C) and strain rate (10^{-6} s^{-1}) in different environments, air and PbBi. The last column reports the oxygen content measured in the experiments in PbBi, with oxygen sensors (Bi/Bi_2O_3).

Table 1 Test Matrix

Steel	Environment	T [°C]	Strain Rate [s^{-1}]	O [wt. %]
T91	Air	300	10^{-6}	-
T91	PbBi			1-30 ×10^{-8}
1.4970	Air			-
1.4970	PbBi			4×10^{-8}

Post-test evaluation. After test, the specimens were observed and analysed in a dual beam FIB-SEM system LYRA3 GMU (fy TESCAN). The sample surfaces with cracks were recorded in a secondary electron mode at working voltage 20 kV and electron beam current ~1 nA. The FIB milling and polishing procedure using Ga^+ ions at working voltage 30 kV and ion beam currents ~1 nA and ~0.2 nA carried out to create a cross sectional view of a chosen crack. A Pt over-layer was used for protecting the cross section from damaging during the milling process.

3. RESULTS

3.1. Tensile tests

Slow Strain Rate Tensile (SSRT) tests were carried out at first up to rupture, then up to the maximum load, in order to keep uniform stress and strain in the tapered part and to avoid strain localization. The stress-displacement curves (**Figure 2**) showed that the environment did not have a marked effect. However, both steels show a slight decrease of the maximum stress value in PbBi compared to the one in air.

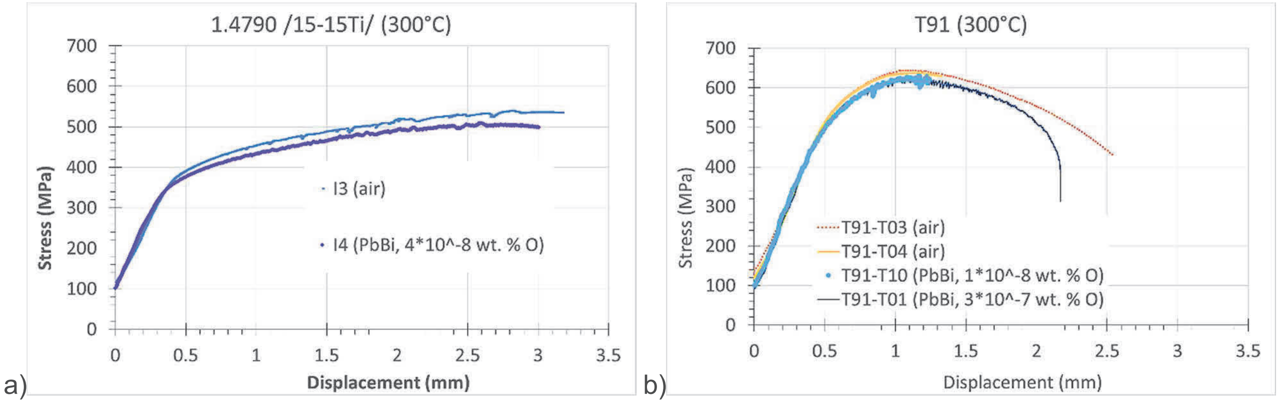

Figure 2 Stress-Displacement curves for a) austenitic 1.4790 and b) ferritic-martensitic T91 steels

3.2. Post-test examination

3.2.1. The steel 1.4970

After test, the specimen surface was observed to characterize surface cracks. On the ground and polished surfaces, there were numerous crack openings around the Ti-rich precipitates. In particular, in air on the ground surface there were the cracks and slip bands marking plastic straining besides the grinding steps (**Figure 3a**). Even though the plastic marks were more numerous after testing in LBE, they still had features of plastic deformation and shallow cracks developed inside some of them (**Figure 3b**).

3.2.2 The steel T91

On the surface of the specimens loaded to rupture in air, there appeared shallow surface cracks (**Figure 4**), which initiated within slip bands. While on the polished surface these slip lines are significant, on the ground one these features are hidden in the roughness of the surface finish. However, the character of the surface cracks is similar, likely ductile.

On the specimen tested in LBE up to the UTS, it was observed formation of semi-brittle cracks (**Figure 5**). In locations around the lowest diameter much more ductile features were observed (**Figure 5a**) than in the location of lower stress (**Figure 5b**) where the last (the farthest away from the neck) crack appeared. For the last crack, the maximum reached stress was 550 MPa (89 % of UTS, 109 % of Rp0.2) and the plastic strain was lower than the total one. These cracks were very likely initiated owing to LME.

For a more detailed investigation of the crack initiation, another specimen, which was loaded in PbBi up to rupture, was used for the FIB cutting and observation of the surface cracks. In particular, the features showed in **Figure 6a** and in the cross section (**Figure 6b**) highlighted the presence of surface cracks. As initiated the cracks were longer on the surface in comparison to their depths. On the image, a two-layer oxide (about 0.3 µm thick), which is in general identified as an outer Fe_3O_4 and an internal $FeCr_2O_4$ spinel type oxide, can be clearly distinguished.

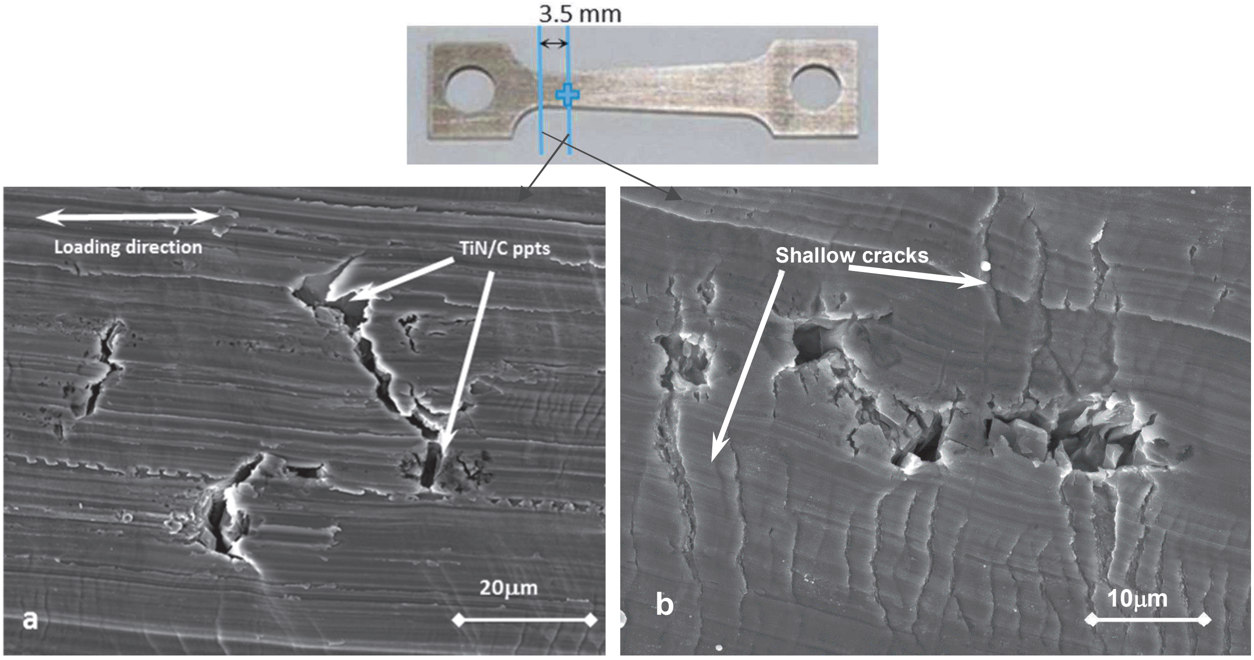

Figure 3 a) Tapered specimen loaded in AIR. B) Tapered specimen loaded in PbBi

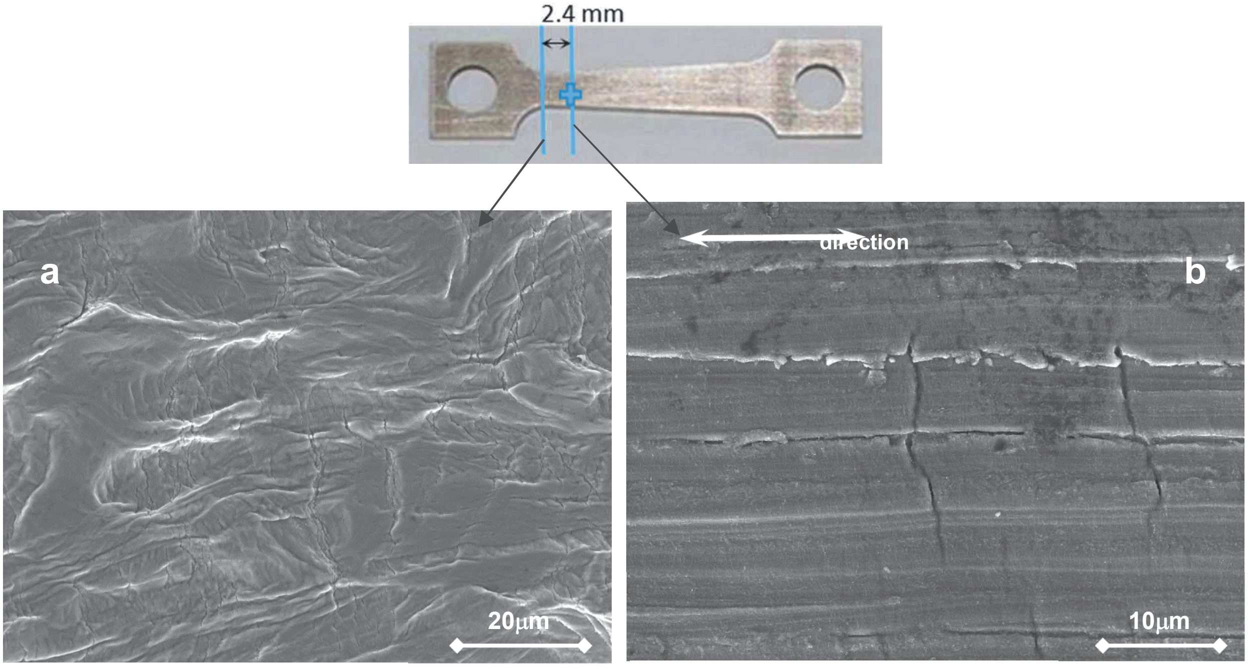

Figure 4 T91 tapered specimen loaded in AIR to rupture:
a) slip lines in necking area of the polished surface, b) the ground surface cracks

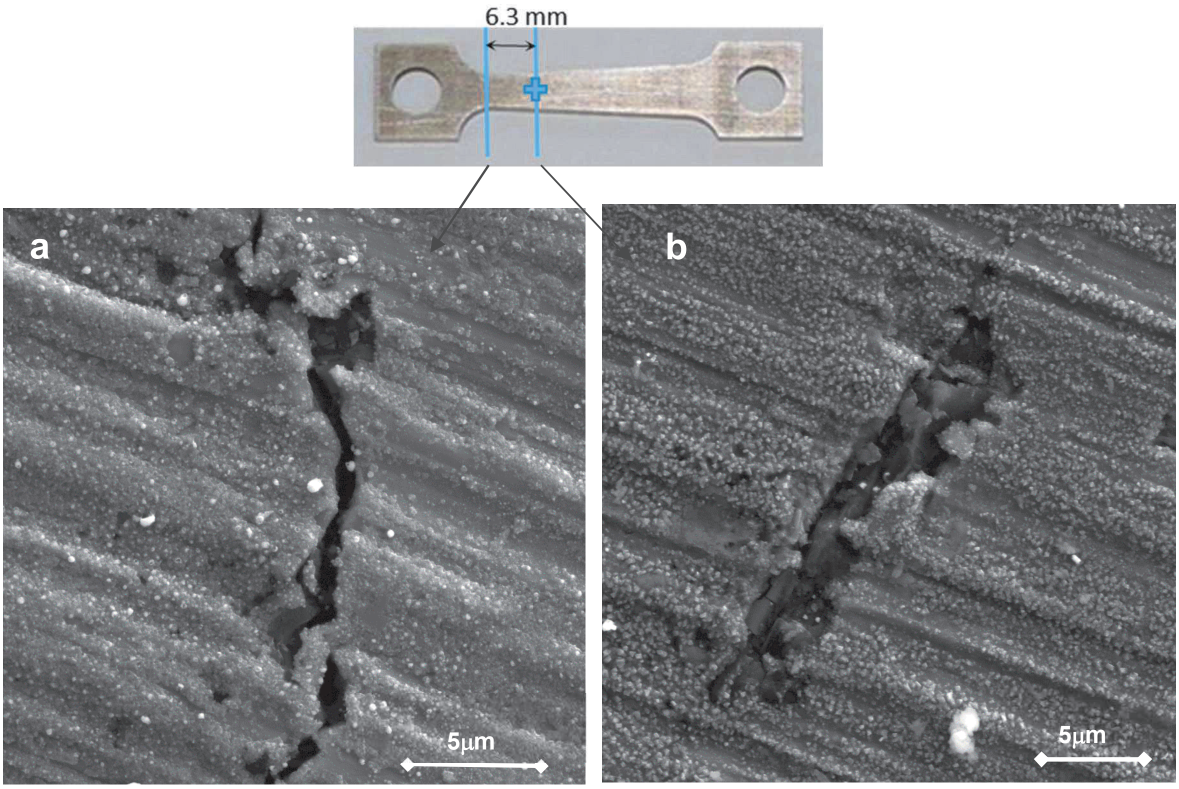

Figure 5 T91 tapered specimen loaded in PbBi up to maximum load, details of the ground surface; crack appearance in a) higher and b) lower stressed regions as marked in the upper picture

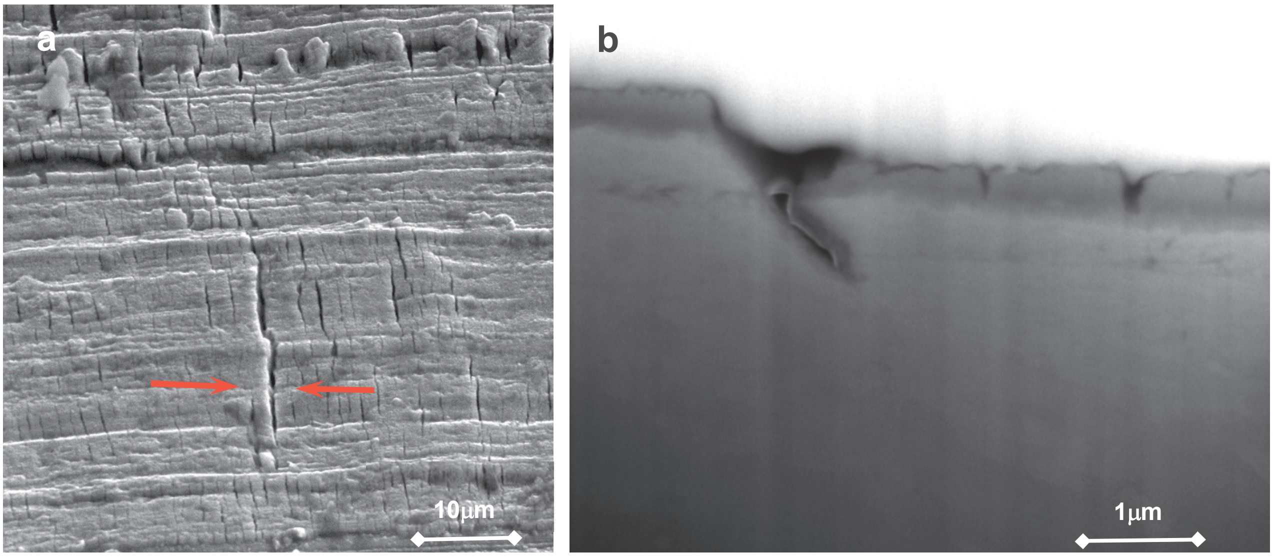

Figure 6 SEM detail of surface cracks in T91, in the necking region about 2 mm from fracture. a) Top view of surface cracks (red arrows point to the location of the FIB cut) and b) FIB cross section of the surface cracks

The cracks initiated in the outer more brittle magnetite layer and they are temporarily stopped at the interface with the internal spinel (**Figure 6b**). One of the observed cracks grew into the bulk (about 1 μm deep) at an angle of about 45°. The crack was most likely initiated in a slip band under the surface as it shows a step between oxide layers on the opposite sides of the crack. This step resulted in opening of the crack which is visible on the surface. However, the crack has lack of PbBi inside that suggests a lack of environmental effect.

Indeed, there appear to be a sufficient oxide layer to prevent the direct contact between the media and the steel. Further investigation is ongoing.

In summary, the observation showed that local plasticity is an important part of the crack initiation in both steels in air as well as in LBE.

4. CONCLUSION

Tapered specimens of austenitic 1.4790 and ferritic-martensitic T91 steels were monotonically loaded in air and PbBi at 300 °C:

- The austenitic steel showed ductile behaviour in air with surface slip bands and openings around Ti-rich precipitates. In LBE, superficial semi-brittle cracks developed in the slip bands.
- The T91 surface cracks were initiated and propagated in air and LBE. In LBE, most of the shallow cracks are limited to the outer Fe_3O_4 oxide layer. Deeper ductile cracks were filled with a thin oxide preventing wetting from the liquid metal.
- Plastic strain is a necessary contribution to crack initiation in T91 in LBE, in order to observe LME.
- Threshold stress for LME crack initiation in T91 was evaluated to be 550 MPa.

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