# CHARACTERISATION OF CRACK INITIATION AND GROWTH IN AUSTENITIC 1.4970 STEEL IN PbBi

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## Abstract

The development of Generation IV nuclear reactors and accelerator driven systems (ADS) is supporting a wide range of work on the compatibility of stainless steels with heavy liquid metals (HLM).

The austenitic steel DIN 1.4970 (also known as 15-15Ti) is being qualified for use in heavy liquid metals because of its lack of sensitivity to Liquid Metal Embrittlement. The aim of this study is to look into cracking modes and their main characteristics in order to reach a clear idea of mechanisms.

Flat tensile, tapered specimens were loaded in PbBi at 300 °C with low oxygen content and, for comparison in air, up to the Ultimate Tensile Stress, UTS, point and rupture. The specimens had one ground and one polished surface, to highlight also the effect of the surface finish.

The behaviour of the steel was not notably affected by the environment, when compared to testing in air. In both cases, the cracking mode was mainly ductile and affected only by the presence of large precipitates in the steel matrix.

Post-tests examinations were carried out with SEM and EBSD. The role of microstructure towards crack initiation and propagation is here described.

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

**Keywords**: Crack initiation, austenitic steel, lead-bismuth eutectic, metallography

## 1. INTRODUCTION

In the nuclear field, austenitic stainless steels are among the main candidates for components of fast reactors. Austenitic stainless steels have long been a favoured choice for fast reactor cladding material due to good creep resistance, resistance to irradiation-induced void swelling, high-temperature mechanical strength and ductility, and established fabrication technology [1,2]. For these reasons, type 15-15Ti material which is a Ti-stabilized austenitic stainless-steel alloy, is one of most suitable for nuclear industry.

15-15Ti has a specifically tailored composition, especially regarding the carbon and titanium content [1]. When Ti is added to the steel as stabilizer, it forms fine TiC precipitates and thus effectively binds the free carbon. The absence of dissolved carbon eliminates the formation of chromium carbides and hence reduces the corrosion sensitization of grain boundaries. Under appropriate annealing conditions, fine, nm-sized TiC precipitates form which act as defect recombination centers during irradiation [3,4,5]. Ti-modified austenitic stainless steels are often cold-worked to increase their mechanical properties. Therefore, the cold-worked steels have higher crack growth resistance at high stress intensity levels compared to solution-annealed stainless steel. The cold-working promotes the precipitation of carbides which are liable to precipitate on slip planes produced by pre-strain and creep strain [6,7].

For its properties, 15-15Ti is one of the best candidates for high temperature components of nuclear reactor of IV generation Lead-cooled fast reactor [8,9]. The main problem in Lead-cooled fast reactor development is the compatibility of the structural materials (steels) with the coolant as well as the corrosion of structural components. When steel comes in contact with liquid metal and is simultaneously under loading conditions, Liquid Metal Embrittlement (LME) could occur. LME is 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. LME is usually most severe just above the melting point of the liquid metal and it disappears with increasing temperature [10]. Although there is a great interest on these steel properties, only few data on their characterization can be found in the literature.

The aim of this work is the study of the 15-15Ti stainless steels crack properties both in air and in PbBi. For this purpose, the crack behaviour was investigated at 300 °C, with low oxygen content and, for comparison in air, up to the Ultimate Tensile Stress point and rupture, following by a microstructural examination in order to characterise crack initiation and propagation.

## 2. EXPERIMENTAL

*Material of* Austenitic steel 1.4970 (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 the thin wall cladding tubes production. The supplied bar underwent 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.

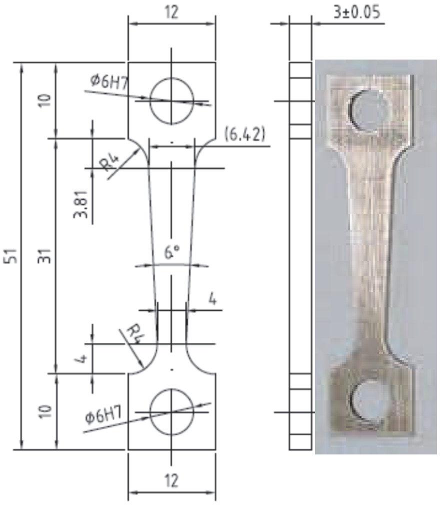

**Figure 1** Tapered Specimen: dimensions and general appearance

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

*Experimental procedure*. The tapered specimens were 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 concepts, where the first container is for the preparation of the liquid metal (oxygen dosing). The liquid is then transferred to the second tank, containing holders and specimens. In this work we report the main observations for 2 specimens, one loaded in LBE and one in air for reference, both at 300C. The oxygen content in LBE was measured during the experiments, with oxygen sensors (ref. Bi/Bi2O3).

After testing, specimens were analysed in a SEM LYRA3 GMU on the surface and cross section. Moreover, a SEM MYRA3 GMU was used for the EBSD analyses.

## 3. RESULTS

*Stress-displacement.* This test was performed with specimen up to the maximum load in air (I3) and with specimen up to rupture (I7). **Figure 2** shows the stress-displacement curves at the minimum cross section of the tapered specimens in the two environments. Yielding and maximum stresses have very similar values in air and PbBi. The oscillations indicate to dynamic strain ageing effect occurring in the steel at the temperature and the strain rate. This graph shows that the environment did not have any effect on the mechanical properties of the material.

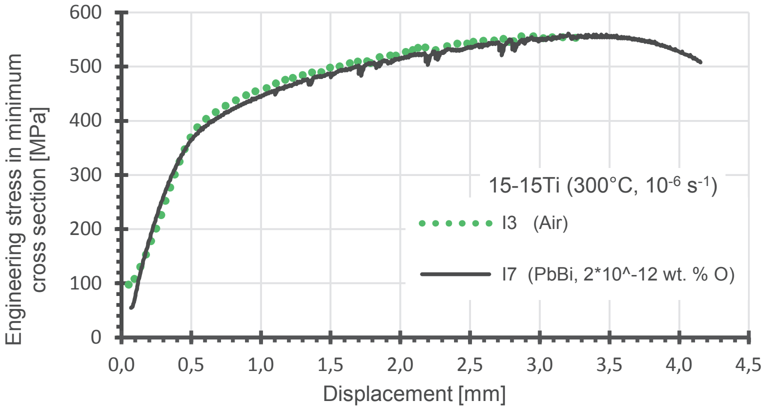

**Figure 2** Stress-displacement graph of 15-15Ti specimen in air and PbBi exposures

*Microscopy.* Observation of specimen surfaces after exposure highlighted the presence of very small cracks (max 5μm long) and marks along slip planes, as a result of the plastic deformation. All these features had a ductile appearance and in both specimens cracks had similar characteristics.

Observation of the ground (**Figures 3 a,b**) and polished (**Figure 3 c**) specimen showed presence of cracks around the Ti-rich precipitates. The polished surface of the specimen showed similar features (**Figure 3 c**), however, the cracks were crossing the Ti-rich precipitates and not the steel matrix.

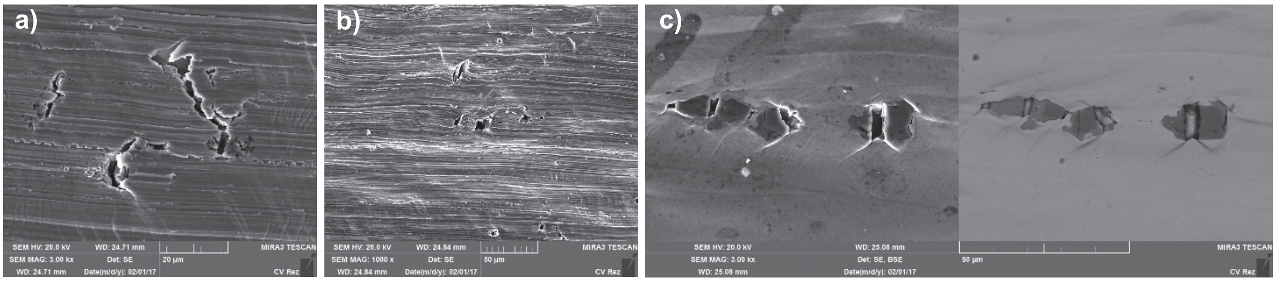

**Figure 3** Surface observation of ground (a,b) and polished (c) 15-15Ti specimen after air exposure

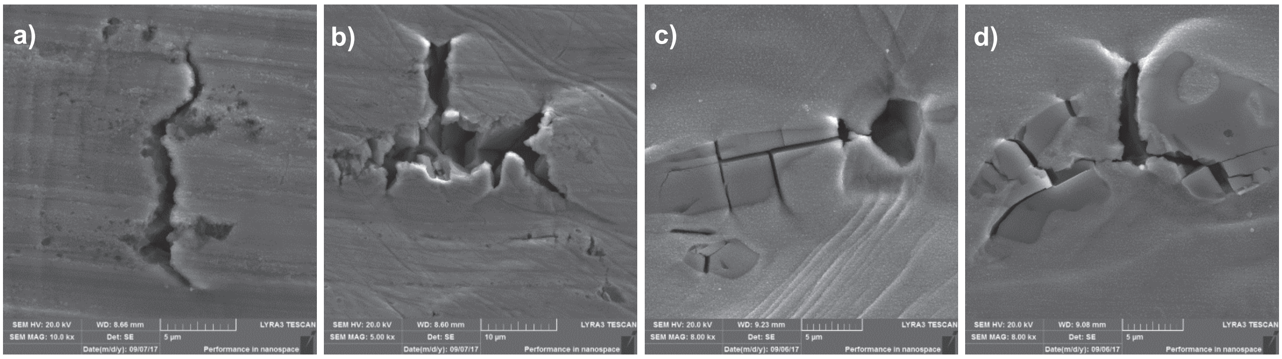

**Figure 4** Surface observation of ground (a,b) and polished (c,d)15-15Ti specimen after PbBi exposure

Similar to the specimen exposed in air, the cracks were observed to be surrounding the precipitates of ground specimen (**Figures 4 a,b**) and going through the precipitates in the polished side (**Figures 4 c,d**). The clusters of dense shallow cracks (**Figure 4c**) in the neck of ground specimen are not observed after air exposure. Cracks of ground specimen were apparently bigger and opened in comparison with polished side of specimen. Polished side did not show many opened cracks.

For the specimen exposed in LBE, a study of the cross-section was also carried out, in order to characterize the cracks (**Figure 5**). Cross-section showed small cracks, not deep into the steel. Wherever Ti-rich precipitates were observed (**Figures 5 c and d**) it was evident that their fracture did not extend to the steel matrix.

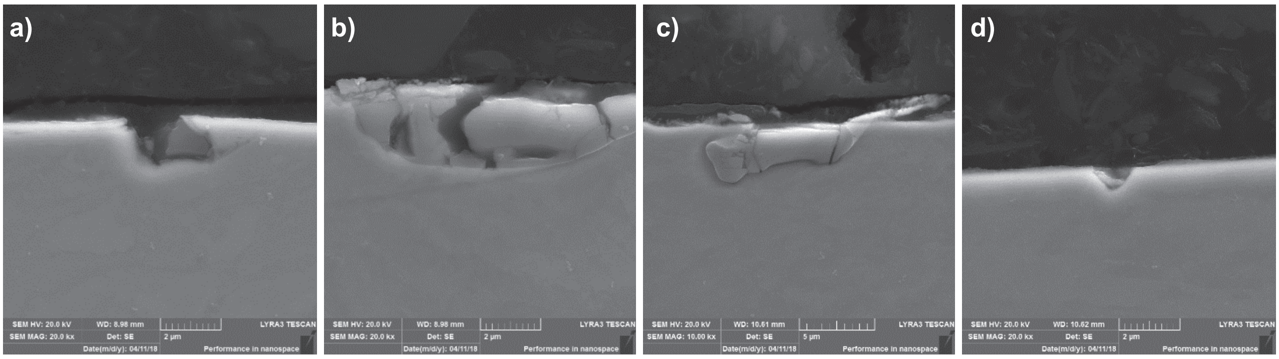

**Figure 5** Cross-section observation of ground (a,b) and polished (c,d)15-15Ti specimen after PbBi exposure

Moreover, the EBSD technique was used to underline the grain orientation around the cracks. Since the material is very deformed, the grain boundaries are not well visible and the whole material is oriented primarily in the direction of stress.

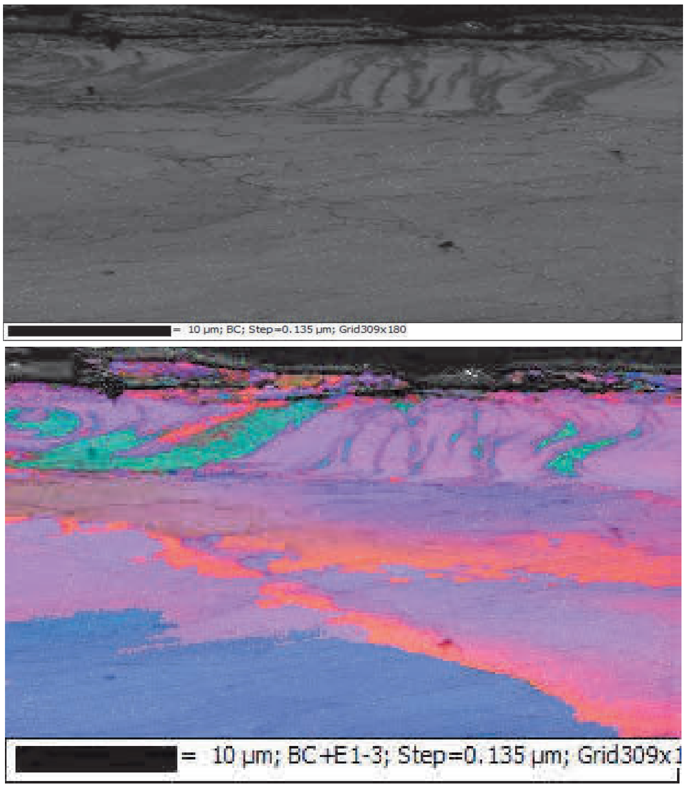

**Figure 6** EBSD pattern of 15-15Ti specimen after PbBi exposure, from the ground edge

## 4. DISCUSSION

In this work, the austenitic steel 15-15Ti was mechanically loaded, in LBE and air for comparison, up to the UTS at 300 °C. The stress-deformation curves highlighted that there is no effect of the environment on the mechanical properties of the steel. This effect is very different from the reduction of plastid displacement observed for instance for the ferritic-martensitic steel T91, which is known to be susceptible to LME [11-12].

The main differences observed where not related to the testing environment, but to the surface finish of the specimens. In particular, no cracks were observed on the polished surfaces in the steel matrix. However, the large Ti-rich precipitates were broken and did not affect the surrounding matrix.

On the other hand, on the ground surfaces, the shallow ductile cracks were more developed around the Ti-rich precipitates, indicating any rough grinding effect. It is proposed that the effect of the grinding is to create a localised condition (refined grain size, strain, tensile residual stress), which has a mechanical resistance different (cracking earlier) than the undisturbed (polished) system.

However, in general the cross-sections confirmed that cracks are shallow and have a ductile character. Moreover, the cracks through the Ti-rich precipitates were limited to the precipitates and did not affect the surrounding matrix.

There was no evidence of LME for this steel in these experimental conditions.

## 5. CONCLUSION

- No effect of the environment (air vs LBE).
- Cracks have the same characteristics in air and LBE, but slightly differ from ground to polished surface.
- Cross-section highlighted that cracks are very shallow.
- Crack initiation does not imply a cleavage-like growth. All cracks have a ductile character.

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