Journal of Nuclear Materials 511 (2018) 459–472

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Initiation of LME crack in ferritic martensitic steel in liquid lead-bismuth

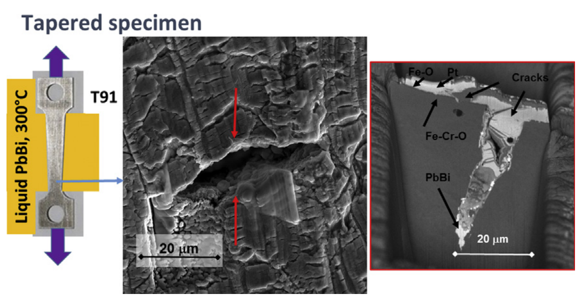
Anna Hojna*, Fosca Di Gabriele, Michal Chocholousek, Zbynek Spirit, Patricie Halodova, Jan Lorincik

Centrum Vyzkumu Rez (CVR), Rez 130, Husinec, 25068, Czech Republic

HIGHLIGHTS

- In air and in LBE (10^{-6} wt % O) at 300 °C, on the ground and polished surfaces, testing resulted in various crack initiation.
- Five types of crack morphologies were observed; most cracks in the region of plastic strain.
- Fracture path and morphology of only several cracks indicated LME/EAC degradation mechanism.
- The conditions of LME/EAC cracks initiation were more than 1.3 % plastic strain and higher than 645 MPa.
- The shallow cracks opening in the protective oxide were observed as the precursor for the cracking.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 29 June 2018
Received in revised form
11 September 2018
Accepted 18 September 2018
Available online 20 September 2018

Keywords:
Ferritic-martensitic steel
Lead-bismuth eutectic
Environmentally assisted cracking
Tapered specimen
Crack initiation

ABSTRACT

This work is aimed at fundamental understanding of the T91 steel cracking mechanisms in liquid PbBi eutectic. In particular, the crack initiation was studied by means of Constant Extension Rate Tensile (CERT) tests with flat tapered specimens in PbBi at 300 °C with oxygen content 3×10^{-7} - 6×10^{-6} wt. % and in air, up to rupture and maximum load. The tapered specimens were meant to create a uniform variation of stress along the gauge length and the threshold stress of the crack initiation was determined. The performance of the steel was affected by the environment. The threshold stress decreased and cracking character changed in PbBi when compared to the testing in air. Post-test examinations carrying out with SEM equipped with FIB found shallow cracks from broken oxide having ductile propagation and a grain-size-deep crack of inter-lath martensite path in the plastic strain region. The cracking stress-strain conditions and the role of oxide rupture causing wetting, as the cracking precursors, are discussed.

1. Introduction

Materials interaction with Heavy Liquid Metals (HLM) has been a topic of wide interest for about twenty years [1] because of worldwide interest in developing HLM-cooled nuclear reactors. The

* Corresponding author.
E-mail address: anna.hojna@cvrez.cz (A. Hojna).

https://doi.org/10.1016/j.jnucmat.2018.09.033

9%Cr ferritic martensitic steel T91 has been among the best candidate materials for internal structural components of future nuclear reactors. The position of T91 on the list of the candidate materials is now being questioned due to the evidence of its sensitivity to cracking in liquid metals, so called Liquid Metal Embrittlement (LME). This phenomenon is typically associated with a change from ductile to cleavage-like fracture mode [2].

The phenomenon of LME used to be characterized by the tendency of structural materials to low energy fracture under stress in contact with liquid metals [1] based on ductility-loss experiments. The alternative, that the low ductility could be explained by crack initiation and propagation before final fracture, was not considered. Over the past few years, it has been shown that the term was not accurate and even misleading. It has been proven that LME crack propagation is much faster than diffusion [3] and that liquid metals did not diffuse into the bulk but interact only with close-to-surface layers [4]. Moreover, LME did not occur if the melt applied for long time had been removed [5]. For all those reasons, the LME is better to consider as a special case of Environmentally Assisted Cracking (EAC).

It is well known, that EAC appears to be a multiple stage process, namely initiation - a crack embryo development, short crack growth, and long crack growth [6,7], in which the crack initiation, including all precursors, preconditioning and incubation, usually constitutes the longest part of the whole process. Extensive investigations have been conducted to study LME/EAC characteristics and to understand the mechanism [1,8–11], but there are still many open questions on the initiation and mechanisms of propagation. It was well-demonstrated, that any direct contact between liquid metals and steels, defined as wetting of the material surface by liquid metal, is a pre-condition for susceptibility to LME [12]. In this context, the wetting is the precursor of LME/EAC cracking.

Investigation of T91 performance in liquid PbBi/Pb at 300–450 °C using accelerated testing (accelerated by applied mechanical straining) showed sensitivity to LME/EAC [10,11]. In the case of medium and high oxygen contents in the PbBi/Pb, when protective oxide layers were built up, only very high stress-strain condition over the tensile strength lead to LME/EAC crack occurrence. A rapid crack propagation followed the crack initiation induced by plastic strain. Accordingly, in three-point-bend specimens pre-strained to the yield strength and exposed to the same environment, no cracks were observed after 2000 h [13]. On the other hand, in low oxygen PbBi, when no oxide is preventing the steel/liquid metal contact, cracks initiated at lower stress & strain, but always above yielding and with contribution of plastic strain [14].

The main goal of this work was to provide an insight into the crack initiation phenomena in T91 in liquid PbBi eutectic and the evaluation of possible mechanisms initiating and propagating the cracks.

2. Experimental

2.1. Material

The ferritic-martensitic steel T91 (Grade 91 Class 2/S50460) of nominal composition, given in Table 1, was produced by Industeel, Arcelor Mittal group. The material was normalized at 1150 °C for 15 min with subsequent water cooling to room temperature and finally annealed at 770 °C for 45 min, then slow cooled in the air. A typical microstructure formed by this heat treatment consists of lath martensite in original austenitic grains (Fig. 1a). The average prior-austenite grain size was 20 μm [15]. Several kinds of precipitates were observed at both the prior-austenite grain boundary and the sub-grain boundaries. The larger precipitates are of the $\mathbf{M_{23}C_6}$ type; these ones form the majority. But also $\mathbf{M_6C}$ type precipitates and small coherent vanadium carbo-nitrides VN(C) are present at the grain boundaries or rather inside the martensitic laths [15]. Fig. 1b shows one example of frequent particles, Al-O. Mechanical properties of the test material is given in Table 2.

2.2. Specimens

Flat tapered specimens (Fig. 2b) of thickness 3 mm, width from 4 mm in minimum to 6.4 mm in the widest size and gauge length 23 mm, were fabricated by wire cutting using electrical discharge machining (EDM). Later, one of the two flat parallel surfaces was ground to 500-grid finish in direction of 25 ± 10° to the load axis (Fig. 2d) and the other one was polished to 1 μm finish (Fig. 2e). The specimen sides were not treated (Fig. 2f).

The specimen surfaces were inspected before testing using SEM. Particles (Fig. 1b) often appeared on the polished surface; flaps and machining grooves dominated on the ground one (Fig. 2d). The surface roughness was measured along the specimen longitudinal axis by means of DektakXT stylus profiler (Bruker). The arithmetical mean roughness (Ra) of the polished, resp. ground surface was 0.005 μm, resp. 0.047 μm (length of scan 20 mm, evaluation length 0.25 mm). The hardness of the polished, resp. ground surface was measured 226 HV30, resp. 230 HV30.

2.3. Experimental procedure

Tests in air and argon were performed using the electromechanical testing machine, Z250 (Zwick/Roell). Once test temperature was reached and stabilized for about 1 hour, the loading was started.

Tests in liquid LBE were performed in the CALLISTO cell built on the electromechanical creep testing machine, Kappa 50DS (Zwick/Roell) (Fig. 2a). CALLISTO is based on two-vessel concept, where the first vessel serves for preparation of the liquid metal (melting and oxygen dosing) and the second one for testing of a specimen (Fig. 2c). The oxygen content was measured in liquid PbBi with oxygen sensors (Bi/Bi_2O_3). Single specimen was fixed into grips inside the CALLISTO cell (Fig. 2c) and the cell was filled with Ar/H. LBE was melted in the first tank with Ar/H gas bubbled as long as low oxygen was achieved, then filled to the second tank. In there, LBE was further conditioned by gases bubbling until achieved the requested oxygen concentration (measured by the sensors), usually for 1–7 days. Then, the specimen was monotonic tensile loaded up to a target load.

Constant Extension Rate Tensile (CERT) testing was applied. The specimens were tested at one temperature (300 °C) in different environments, air/argon and PbBi eutectic (LBE) by three strain rates (SR1 < SR2 < SR3). Table 3 contains the test matrix.

Table 1
Composition of the steel T91 as provided by the producer.

Fe	C	Cr	Mo	Mn	Si	Ni	V	Cu	Nb	P	Al	Ti	S	N
Bal.	0.102	8.895	0.889	0.401	0.235	0.121	0.202	0.080	0.079	0.019	0.010	0.004	0.0007	0.048