



Multiscale investigation of quasi-brittle fracture characteristics in a 9Cr–1Mo ferritic–martensitic steel embrittled by liquid lead–bismuth under low cycle fatigue

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ABSTRACT

Liquid metal embrittlement (LME) induced quasi-brittle fracture characteristics of a 9Cr–1Mo ferritic–martensitic steel (T91) after fatigue cracking in lead–bismuth eutectic (LBE) have been investigated at various length scales. The results show that the LME fracture morphology is primarily characterized by quasi-brittle transverse flat regions partially covered by nanodimples, shallow secondary cracks propagating along the martensitic lath boundaries as well as tear ridges covered by micro dimples. These diverse LME fracture features likely indicate a LME mechanism involving multiple physical processes, such as weakening induced interatomic decohesion at the crack tip and plastic shearing induced nano/micro voiding in the plastic zone.

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1. Introduction

Liquid metal embrittlement (LME) has been reported to occur in a wide variety of solid metal/liquid metal couples, characterized by loss of ductility and fracture toughness of normally ductile metallic materials when stressed in a specific liquid metal environment [1–5]. Since this phenomenon was first discovered about one century ago, extensive investigations have been conducted to study its characteristics and to understand the mechanism, but the essence of LME, particularly on atomic scale, is still in dispute [6–8]. So far, some promising LME models and mechanisms have been proposed, including adsorption induced reduction in surface energy [9], adsorption induced reduction in interatomic cohesion [10,11], adsorption induced dislocation emission [8,12–14], grain boundary penetration [15,16], stress assisted dissolution–condensation [17,18] and adsorption enhanced work hardening [19]. The adsorp-

tion induced reduction in surface energy mechanism postulates that the surface energy of a solid metal is reduced by adsorption of a liquid metal, thereby lowering the stress required to fracture the solid. The adsorption induced reduction in interatomic cohesion model, to some extent, is equivalent to the adsorption induced reduction in surface energy mechanism. This model claims that the adsorption of liquid metal atoms at crack tips weakens the atomic bonds being in contact with the liquid metal so as to cause interatomic brittle decohesion. The adsorption induced dislocation emission mechanism was proposed to account for the formation of micro dimples observed on LME fracture surfaces. The basic assumption of this mechanism is that adsorption of embrittler atoms at crack tips reduces the shear strength of interatomic bonds, which facilitates dislocation emission at the crack tips and then leads to ductile failure by highly localized plasticity. The two grain boundary penetration models report that grain boundary penetration of liquid metal atoms by atomic diffusion is necessary for LME crack initiation and propagation. The stress assisted dissolution–condensation model hypothesizes that dissolution of a solid in a liquid can be enhanced by an applied stress. As a result, the stress distribution around crack tips creates a gradient of chemical

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potential, which causes a diffusion flux directing from the crack tips towards the liquid, accompanied by re-condensation of the dissolved solid at the crack walls behind the crack tips. Similar to the adsorption induced dislocation emission mechanism, the adsorption enhanced work hardening mechanism also agrees that adsorption of embrittling atoms at crack tips promotes dislocation emission. However, the difference is that the consequence of the favored dislocation emission in the adsorption enhanced work hardening mechanism is to enhance work hardening at a surface layer around the crack tips, instead of to create immediate crack propagation as assumed by the adsorption induced dislocation emission mechanism. Then, brittle cracking occurs through the hardened surface layer around the crack tip. Since none of these models and mechanisms is able to provide a unified explanation for all the solid metal/liquid metal embrittling systems discovered so far, to completely figure out the underlying mechanism of LME is still a challenging task [6–8].

Recently, the LME phenomenon of the T91/LBE couple has aroused great interest in the nuclear industry, because of the potential application of T91 steel in the GenIV liquid Pb–Bi cooled nuclear reactors [20–41]. For instance, the excellent swelling resistance under fast neutron flux and superior high temperature mechanical properties enable this steel to be a candidate material to construct the proton beam window of the accelerator driven system (ADS) based MYRRHA nuclear facility, which is being developed at SCK•CEN, Belgium for transmuting long-lived nuclear waste [42]. However, one barrier to qualify this steel for safe use in the MYRRHA reactor is that LME may pose a threat to the structural integrity of the proton beam window by involving premature brittle failure [20–41]. In order to take effective measures to mitigate and prevent the LME risk, the underlying mechanism of this intricate embrittlement phenomenon must be resolved.

Over the past decade, extensive work has been performed to investigate the LME characteristics of T91 steel in LBE environment under different conditions, with special attention to the impact of various parameters on the embrittlement susceptibility, including temperature [21–23,26,43,44], strain rate/loading rate [21,24,33], metallurgical state [26,43], oxygen concentration in LBE [29], pre-exposure treatment [21,45], surface oxides [45,46], surface flaws or notches [22], etc. Some important results have been obtained. For example, LME occurred only in a certain temperature domain, called “ductility trough” [22,26,33,43,47]. A slow strain rate in conjunction with a low oxygen concentration of LBE were shown to enhance the LME susceptibility of T91 steel [29]. Tempering at a relatively low temperature to harden the steel was also reported to intensify the LME severity [26,43]. The effect of pre-exposure treatment was demonstrated to rely on oxygen concentration of LBE. Pre-exposure to an oxygen-saturated LBE resulted in the formation of double-layer protective oxides (magnetite and Fe–Cr spinel [48–50]) on the steel surface, thereby reducing the LME effect due to the fact that the crack initiation process was inhibited by the oxide films [45,51]. On the other hand, pre-exposure to an oxygen-depleted LBE led to dissolution of the protective oxides, which improves the wettability and facilitates the crack initiation [45,51]. In addition, the presence of stress concentrators such as surface cracks, flaws and notches was observed to greatly favor the occurrence of LME in the T91/LBE system [22]. Unfortunately, a clear explanation of these experimental observations is missing. This can partly be attributed to the great difficulty in clarifying the correlation between the complex microstructure of T91 steel and the LME fracture morphology.

After T91 is cracked in LBE under monotonic loading, transgranular “flat” and “featureless” fracture surfaces have been observed by conventional scanning electron microscopy (SEM) in most of the previous investigations [30,31,34,41,51]. These observed fractographic features are usually considered as typical manifestations

of LME for the T91/LBE system. To describe these complex fracture modes formed under the influence of LBE, the terminology “quasi-cleavage” has been widely used to indicate likely a mixture of prevalent transgranular (or translath) brittle features and limited ductile features. However, using focused-ion beam (FIB) to extract thin foils beneath the fracture surface for subsequent transmission electron microscopy (TEM) observations, Martin et al. [52] and Hémerly et al. [53] have recently obtained different results in T91 steel tested in liquid LBE, liquid indium and liquid sodium environments under monotonic loading. They found that the fracture mode of the secondary cracks emanating from the fracture surfaces was mainly intergranular cracking either at the prior-austenite grain boundaries or at the martensite lath boundaries, rather than transgranular or translath “quasi-cleavage”. Based on this interesting finding, they finally proposed not to use “quasi-cleavage” to describe the LME fracture morphology of T91 steel. Hémerly et al. [54] even observed intergranular failure in a Fe18Cr9Ni1Mn stainless steel/liquid sodium system. Their observations seem to be supported by a series of LME cases involving evident intergranular penetration of liquid metal atoms, such as polycrystalline Al/liquid Ga [55–58], Ni/liquid Bi [59,60], Cu/liquid Bi [61] and ferrite iron/liquid zinc [62]. Nevertheless, in our recent work [41], using high resolution electron backscatter diffraction (EBSD), transgranular cracking by cutting either through the prior-austenite grain boundaries or through the martensite lath boundaries was observed to dominate the propagation behavior of the main crack of T91 steel under low cycle fatigue in LBE at 350 °C. This means that the main fatigue crack has a different propagation behavior from the secondary cracks. In this regard, how to properly depict the LME fracture features of T91 steel and especially to interpret the underlying mechanism of their formation is still an open issue. One of the key approaches to deal with this issue could be to examine the LME fracture surface and the microstructure immediately beneath it under very high magnification, considering that the fracture morphology represents the history of the crack tips and LME is a surface phenomenon involving direct microstructural changes only within a very thin layer of material in contact with the liquid metal. Fatigue is the most important origin of engineering failure, therefore, the main focus of this study is on fatigue fracture surfaces after testing in LBE.

In order to get deeper insights into the nature of the LME phenomenon in the T91/LBE couple, a systematic multiscale microstructural investigation has been performed to characterize the fracture surface of T91 steel cracked in low oxygen LBE under low cycle fatigue and the deformation microstructure immediately beneath it by means of multiple advanced characterization techniques. These techniques include high resolution SEM, atomic force microscopy (AFM), high resolution EBSD and FIB sampling technique for subsequent TEM observations. The observed results are discussed in the framework of the previous literature models and mechanisms. Some new viewpoints are also proposed for a better understanding of the LME mechanism of the T91/LBE couple.

2. Experimental

2.1. Material

The material used in this study was T91 steel. This steel was austenitized at 1050 °C for 15 min and then water quenched, followed by tempering at 770 °C for 45 min and then air cooling. The chemical composition of the steel is C: 0.097, Cr: 8.87, Ni: 0.12, Mo: 0.87, Mn: 0.39, V: 0.19, Si: 0.22, Nb: 0.08, N: 0.044 and Fe in balance (wt.%).

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