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Investigating liquid-metal embrittlement of T91 steel by fracture toughness tests

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A B S T R A C T

Heavy liquid metals such as lead bismuth eutectic (LBE) are chosen as the coolant to innovative Generation IV (Gen IV) reactors where ferritic/martensitic T91 steel is a candidate material for high temperature applications. It is known that LBE has a degrading effect on the mechanical properties of this steel. This degrading effect, which is known as liquid metal embrittlement (LME), has been screened by several tests such as tensile and small punch tests, and was most severe in the temperature range from 300 °C to 425 °C. To meet the design needs, mechanical properties such as fracture toughness should be addressed by corresponding tests. For this reason liquid-metal embrittlement of T91 steel was investigated by fracture toughness tests at 350 °C.

Tests were conducted in Ar-5%H_2 and LBE under the same experimental conditions Tests in Ar-5%H_2 were used as reference. The basic procedure in the ASTM E 1820 standard was followed to perform tests and the normalization data reduction (NDR) method was used for the analysis. Comparison of the tests demonstrated that the elastic–plastic fracture toughness (J_{1C}) of the material was reduced by a factor in LBE and the fracture mode changed from ductile to quasi-cleavage. It was also shown that the pre-cracking environment played an important role in observing LME of the material since it impacts the contact conditions between LBE and steel at the crack tip. It was demonstrated that when specimens were pre-cracked in air and tested in LBE, wetting of the crack surface by LBE could not be achieved. When specimens were pre-cracked in LBE though, they showed a significant reduction in fracture toughness.

1. Introduction

In order to produce energy for a time span beyond the operational life of current reactors in an efficient and environmentally friendly way, a new generation of reactors (Generation IV reactors) are now under development. These require high temperatures to reach the appropriate efficiency [1,2]. The need for high temperatures has made ferritic-martensitic T91 steel candidate materials for structural and functional components of Gen IV reactors due to their better resistance to increased temperatures, lower thermal expansion coefficient and higher swelling resistance as compared to austenitic stainless steels [3,4]. One of the main challenges in the development of many innovative reactor systems is compatibility of the selected candidate materials with the coolant. For systems with heavy liquid metal coolant as lead-bismuth eutectic (LBE) the major materials degradation effects to be investigated are liquid metal corrosion and the effect of LBE on mechanical properties of steels. LBE is indeed known to have a deteriorating effect on the mechanical properties of T91 which has been demonstrated by tensile [5,6], bending [7] and small punch tests [8]. For instance, a reduction in ductility in LBE was reported by many authors by tensile tests at temperatures between 200 and 400 °C [5,6,9]. In these tests, the comparison of total elongation of the specimens tested in inert and LBE environments was used as the liquid-metal embrittlement (LME) susceptibility criterion. At 300 °C Di Gabriele et al. [5] observed a reduction of about 8–17% in total elongation. Dai et al. [6] on the other hand showed that specimens with micro-cracks on their surfaces showed the highest reduction in total elongation in the 300–425 °C temperature range. This ductility loss, referred to as liquid-metal embrittlement (LME), has been a

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subject of investigation since the early 1900's [10,11]. Many factors play a role when investigating LME and evaluating its severity; such as the steel microstructure, temperature, chemistry of LBE, stresses and the wetting behaviour between the steel and LBE [10,12,13]. The results of the above mentioned tests clearly indicated the susceptibility of T91 steel to LME in LBE environment. To assess the potential effect of LME on the material performance in reactors, an investigation on the mechanical properties which are used in the mechanical design is a clear prerequisite. Fracture toughness is probably the most important property of the materials to be assessed especially in view of potential material degradation effects, which results in brittleness.

Since multiple factors affect the LME appearance and not all of them are thoroughly investigated, it is quite challenging to perform tests to provide representative fracture toughness data for operational conditions of nuclear systems with LBE coolant. An additional challenge is to demonstrate that the obtained data assess fracture toughness properties in a conservative way. Technical difficulties for testing arise from the necessity to use elevated testing temperatures, the conductive nature of LBE and LBE's degrading effect on the set-up itself. For instance, the potential drop method, which is conventionally used for fracture toughness testing in air, cannot be applied in LBE because the electrically conductivity of LBE interferes with the measurement. Additionally, the unloading compliance method requires the use of a clip gage extensometer which can withstand the high temperatures in LBE, but it is not readily available for current set-ups. In addition, the selection of steel parts for the test set-up is crucial as well, as LBE can embrittle these parts which are loaded under stresses. These challenges force us to use alternative testing methods and find the most suitable materials for the parts of the set-up that work under stresses; such as pins and grips. Moreover, an additional challenge is to ensure contact between the crack tip and LBE environment throughout the test as the wetting of the fracture surface by the liquid metal is an absolute prerequisite in observing LME. The thin oxide films formed at the sides of the initial crack during pre-cracking in oxygen containing environments might prevent this contact.

Several attempts to measure the LBE effect on the fracture toughness of T91 steel have been made [14–16]. The reduction in fracture toughness due to LBE was reported in most cases. However, a significant difference between the results of the tests exists, mainly due to the differences in testing and analysis methods. For instance, Auger et al. [14] performed fracture toughness tests using center cracked in tension (CCT) geometry specimens and although they observed a slight reduction in elastic–plastic fracture toughness parameter (J), it was difficult to quantify the difference. This was because they could not measure a full reference J–R curve due to extended shear band localization which occurred almost after the crack initiation in reference tests. It should also be kept in mind that even though the CCT geometry is used to assure the plane-stress conditions for assessment of thin components behaviour, it is very important to also investigate the plane-strain conditions for thicker components. Van den Bosch et al. [15] carried out fracture toughness tests by using disc shaped compact tension (DCT) geometry specimens to study the plane-strain conditions. They observed around 20–30% reduction in J-values and fracture surface analysis revealed a mixture of ductile and quasi-cleavage features at the crack. However, pre-cracking and testing was performed in oxygen saturated conditions since the vessel with LBE was open to air which could have led to formation of protective oxide layers on metal surface and have an impact on the wetting of the crack surface by LBE. Hojna et al. [16] also used the DCT geometry and at 300 °C they observed higher crack growth rate in LBE than in air All these observations clearly demonstrate the detrimental effect of LBE on the fracture toughness of T91 steel. However, the reliable quantitative estimation of the effect is still missing. In this paper, an attempt is made to develop a procedure for quantitative assessment of fracture toughness properties of T91 steel in LBE environment. As tensile tests showed the most severe embrittlement in a temperature range of 300–425 °C, the temperature of 350 °C has been selected for the present tests. Fracture surfaces and elastic–plastic fracture toughness parameters (J_{1C}) obtained from tests in Ar-5%H_2 were used as reference. The changes in fracture mode and the reduction in fracture toughness in LBE environment compared to the reference values were used to determine the severity of embrittlement. It was also demonstrated that when specimens which were pre-cracked in air were used for testing in LBE, the intimate contact between the crack surface and LBE could not be achieved and the obtained results underestimate the effect of LME of T91 steel. A method was developed for pre-cracking specimens in LBE and it was shown that the intimate contact between the crack surface and LBE could be assured this way. These specimens showed severe embrittlement when tested in LBE. These efforts are part of a collective exercise on the development of guidelines for fracture toughness tests in liquid metal environment performed in the framework of EUROATOM FP7 MATTER project.

2. Experimental

The ferritic-martensitic T91 steel used in this work was provided by Industeel as a 15 mm hot rolled and heat treated plate. The as-received T91 plate underwent a two-step heat treatment; normalization and tempering. The steel was heated to austenite phase (1050 °C) for 1 h and then cooled rapidly to room temperature to form martensite. It was then tempered at 770 °C for 45 min. The composition of ferritic-martensitic T91 steel is given in Table 1.

½ compact tension (CT) geometry specimens (W = 25 mm, B = 12.5 mm) were chosen for the fracture toughness tests discussed in this work to meet the plane-strain condition (Fig. 1). All specimens were machined in T-L orientation (T: direction of loading, L: direction of crack propagation).

Based on the tensile data performed on the T91 steel at 350 °C and reported elsewhere [17], the effective yield strength (σ_Y) to be used in the fracture toughness analysis was calculated as 545.6 MPa from the equation;

$$\sigma_Y = \frac{(\sigma_{YS} + \sigma_{UTS})}{2} \tag{1}$$

All tests were done in liquid-metal embrittlement testing set-up (LIMETS-4) which was manufactured at SCK-CEN to conduct tensile and fracture toughness tests in LBE. LBE is stored in the dump tank where it is conditioned at 300 °C. Specimens are loaded in the autoclave where the test is performed. For tests in LBE, LBE is transferred from dump tank to the autoclave at 300 °C. Before LBE is transferred to autoclave, the autoclave is vacuum purged and gas cleaned. Consequently it is ensured that the dissolved oxygen concentration in LBE does not suddenly increase when LBE is transferred from dump tank to autoclave.

The specimens were monotonically loaded following the basic procedure in the ASTM E 1820 standard [18] and the normalization data reduction (NDR) method was applied to analyse test results. In

Table 1
Composition of T91 ferritic-martensitic steel (wt%).

C	N	Al	Si	P	S	Ti	V	Cr
0.10	0.11	0.015	0.22	0.021	0.0004	0.003	0.21	8.99
Mn	Ni	Cu	As	Nb	Mo	Sn	W	
0.38	0.11	0.06	0.008	0.06	0.89	0.004	0.01	