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# Investigation of LBE embrittlement effects on the fracture properties of T91

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**Abstract**

The susceptibility to liquid metal embrittlement (LME) of the T91 steel was studied by performing 3-point bending tests in liquid lead–bismuth eutectic (LBE), and for comparison, in argon (Ar) atmosphere as well. The specimens of T91 with different heat treatments were tested to access the hardening effect on the fracture toughness of the steel after exposure in LBE. The results showed that the fracture toughness of steel was reduced by contacting with LBE. The susceptibility of T91 to LBE embrittlement increased with the hardening of the steel introduced by heat treatments.

## 1. Introduction

Ferritic/martensitic (FM) steel T91 has been selected as beam window material for liquid metal spallation targets such as megawatt pilot experiment (MEGAPIE) and those foreseen in the future's accelerator driven system (ADS) [1]. Liquid lead–bismuth eutectic (LBE) is one of the candidate target and coolant materials for such systems. Apart from the serous degradation of mechanical properties induced by intensive proton and/or neutron irradiation, liquid metal embrittlement (LME) effect is of great concern for the components made of the T91 steel in contact with LBE, particularly when hardened by irradiation, as reported in literature [2,3].

The LBE embrittlement effect on ferritic/martensitic steels was extensively studied in Europe in last few years with different techniques [4–7] and investigated in our previous work by performing slow-strain-rate tensile (SSRT) tests on T91 and F82H steels [8,9]. It can be concluded that the pre-requests for LME of FM steels are: wetting (intimate contact), surface cracks or flows, and a certain level of stress concentration at crack tips. Meanwhile, our preliminary investigations on irradiated T91 specimens demonstrated that the LBE embrittlement effect might increase with irradiation dose, or with irradiation induced hardening [10,11]. However, due to high costs of irradiation and difficulties in handling radioactive specimens, studies on the LME of irradiated materials are very limited up to now. In order to improve the understanding of irradiation hardening effect on the LME of ferritic/martensitic steels, therefore, some simulations using T91 specimens hardened by tempering at lower temperatures have been carried out [12] that had an essential significance for studying the effect of the strength of materials on LME phenomena.

In our previous work, SSRT tests were conducted in LBE on T91 specimens of different strengths induced by tempering at 500 °C, 600 °C and 750 °C [12]. The results showed that the width and depth of the 'ductility-trough' (namely the degree and the temperature range of the embrittlement induced by LBE) increased with the strength of the material. For a deeper understanding of the LBE embrittlement phenomena, the present work, here described has aimed at studying the fracture behavior of hardened T91 specimens in LBE environment, in particular the LBE effect on the fracture toughness of the steel, which is one of important mechanical data requested for engineering design of facilities like the MEGAPIE target.

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## 2. Experimental

### 2.1. Materials and specimens

The T91 steel was Heat A387 Gr91 produced by the INDUSTEEL Groupe Arcelor, France. The material was purchased for material studies performed in the scientific support program of the MEGAPIE project. The original form of the steel was a plate of 100 mm thickness. The plate was only normalized (at 1040 °C/0.5 h) without tempering. The composition of the steel used was, in wt%: 8.76 Cr, 0.099 Ni, 0.597 Mn, 0.186 V, 0.862 Mo, 0.073 Nb, 0.054 Cu, 0.019 Co, 0.088 C, 0.019 P, 0.317 Si, and balanced by Fe.

For fabricating specimens from the material, small plates of ~100 × 40 × 15 mm were first cut from the big plate using a bend saw, and then tempered at 760 °C for 1 h. The bend bar specimens used in this study were machined from the small plates with an electro-discharge wire saw (EDM) to a dimension of 20 × 4 × 2 mm and with a notch of 1 mm in depth. The outer surfaces of the specimens were finished with fine polishing using #4000 sandpapers. Before performing fatigue pre-cracking, the specimens were heat-treated in two steps: (1) normalized at 1040 °C in high vacuum for 60 min and followed by air cooling and (2) tempered separately at 750 °C, 600 °C and 500 °C in high vacuum for 2 h and followed by air cooling. The treatments resulted in different yield stresses of 510, 905 and 1205 MPa for the three tempering treatments, respectively [12]. According to the different tempering temperatures, the three types of the specimens have been denominated as HT750, HT600 and HT500, respectively.

After heat-treatment, the specimens were fatigue pre-cracked to a ratio of the crack length to specimen width ($a_0/w$) of about 0.5.

### 2.2. Three point bending test

The fracture toughness tests were conducted according to the American Society for Testing and Materials (ASTM) E1820-99: Standard Test Method for Measurement of Fracture Toughness. The single-specimen technique with the loading–unloading compliance method was used for determining the $J$-integral resistance ($J$–$R$) curves and $J$-values. The difficulty associated with the application of the single-specimen technique is the determination of the crack length of a specimen, which is normally, accessed using the multi-specimen technique. In our study, the multi-specimen technique was used to derive the relationship between the crack length and the bending displacement (the detailed information about it could be seen in our previous work [13]).

The specimens were tested with a Zwick-20 mechanical testing machine in LBE at 200 °C, 300 °C, 400 °C and 500 °C. There was no oxygen meter installed for measuring oxygen content in the LBE. However, as the LBE was always kept in a storage tank at 250 °C, the oxygen content in the LBE should have been below 1 wppm. The experimental procedures were the same as that for SSRT tests [9]. For comparison, the 3-p bending tests were performed in Ar atmosphere, too.

After bending testing, the fracture surfaces of the specimens were observed with an optical microscope (OM) and a scanning electron microscope (SEM) to identify the fracture mode of the specimens.

## 3. Results and discussion

### 3.1. Three point bending tests in Ar

The load–displacement curves obtained at 200 °C for the specimens with different heat treatments are shown in Fig. 1. It can be seen that the HT750 specimen demonstrates a typical load–displacement trace for a stable crack growth. While the HT500 specimen exhibits local ductile instability (it means fast crack propagation in a ductile mode for a constant loading rate) after reaching the maximum load ($F_{\max} \sim 500$ N in this case). As expected, the HT600 specimen shows a case in between that of the HT750 and HT500 specimens. The $J$-values evaluated from these tests are 405, 260 and 200 kJ/m$^2$ for the HT750, HT600 and HT500 specimens, respectively. One can see that the $J$-values decrease with decreasing tempering temperatures.

It should be noted that in this figure the maximum loads of the specimens do not reflect the strengths of the materials because the initial crack lengths are different for different specimens.

### 3.2. Three point bending tests in LBE

Tests carried out on the HT500 specimens in LBE demonstrated very different behaviors from the ones tested in Ar. As an example, Fig. 2(a) shows the load–displacement curves of the tests performed in both LBE and Ar at 200 °C

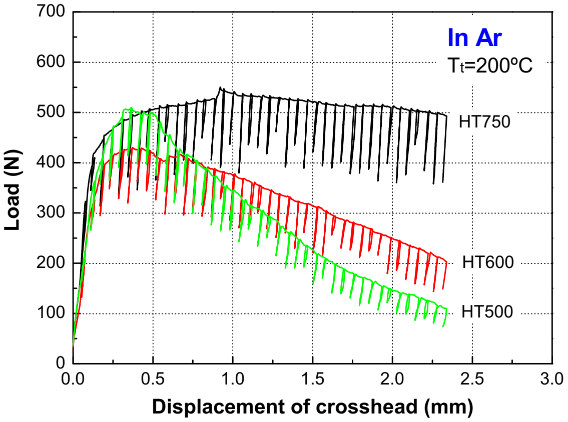

Fig. 1. Load–displacement curves of the HT750, HT600 and HT500 specimens tested in Ar at 200 °C.

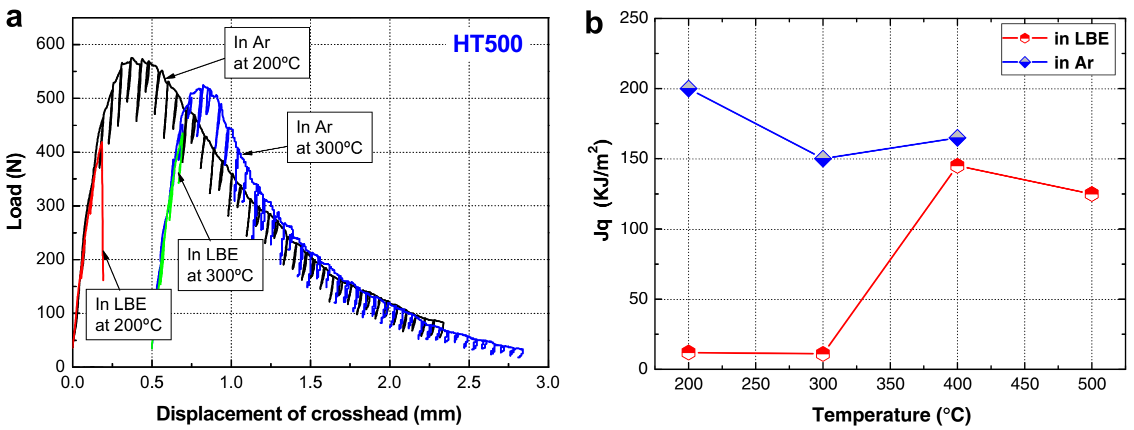

Fig. 2. (a) Load–displacement curves of the HT500 specimens tested in Ar and in LBE at 200 °C and 300 °C (shifted by 0.5 mm in *X*-axis); (b) *Jq*-values derived from tests at 200–500 °C in Ar (up to 400 °C) and LBE.

and 300 °C. It can be seen that both specimens failed very early (in elastic regime) by sudden, unstable crack extension when they were exposed to LBE. The curves indicate a typical brittle fracture behavior. The *J*-values evaluated from the curves are about 12 kJ/m$^2$ for both specimens tested in LBE. Comparing to that of tests in Ar, 200 and 150 kJ/m$^2$, one can see that the presence of LBE results in a significant decrease in the fracture toughness of HT500 specimens. Fig. 2(b) presents the fracture toughness values of the HT500 specimens tested in Ar at 200 °C, 300 °C and 400 °C, and in LBE at 200 °C, 300 °C, 400 °C and 500 °C. The results indicate that the LME effect on the HT500 specimens is detected at all temperatures between 200 °C and 500 °C, with a stronger influence at lower temperatures ($\leqslant$300 °C). At higher temperatures above 400 °C, the effect turns to be greatly reduced.

The behavior of the HT600 specimens is similar to that of the HT500 specimens. Fig. 3(a) shows the results of 3-point bending tests performed in Ar and in LBE at 200 °C and 300 °C. Similar to the HT500 specimen tested at 300 °C in LBE, the HT600 specimen tested in LBE at 300 °C failed very early (with just slight plastic deformation) by quite fast crack propagation. For the specimen tested at 200 °C in LBE, it also failed much earlier as compared to that tested in Ar and with fast crack propagation as well. The *J*-values of these two specimens tested at 200 °C and 300 °C are 135 and 42 kJ/m$^2$, respectively, which are much lower than the values obtained in the Ar tests, 260 and 270 kJ/m$^2$. This indicates the HT600 specimens are also sensitive to LBE embrittlement. However, compared to the HT500 specimens, the HT600 specimens show a less pronounced embrittlement effect, which is indicated by less reduction of *J*-value at 200–400 °C and fully recovery at 500 °C, as shown in Fig. 3(b).

Fig. 4(a) illustrates the load–displacement curves of the HT750 specimens tested at 200 °C and 300 °C in both LBE and Ar environments and Fig. 4(b) shows the *J*-values derived from the tests. From the load–displacement curves one can see that for specimens tested in LBE, the load decreased faster after it reached the maximum value. This means that the cracks of the specimens tested in LBE propagated still faster than those tested in Ar, although no sudden crack extension appeared. The *J*-values were reduced 20–30% in the presence of LBE.

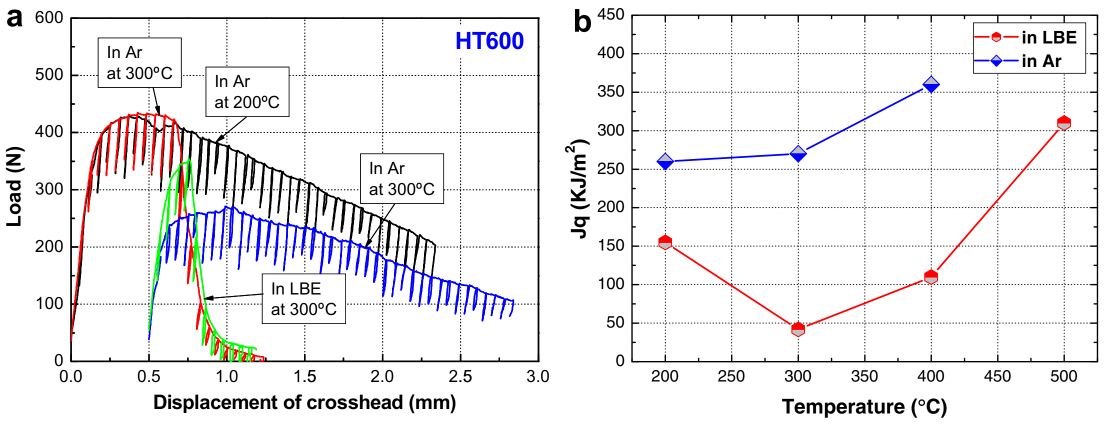

Fig. 3. (a) Load–displacement curves of the HT600 specimens tested in Ar and in LBE at 200 °C and 300 °C (shifted by 0.5 mm in *X*-axis); (b) *Jq*-values derived from tests at 200–500 °C in Ar (up to 400 °C) and LBE.

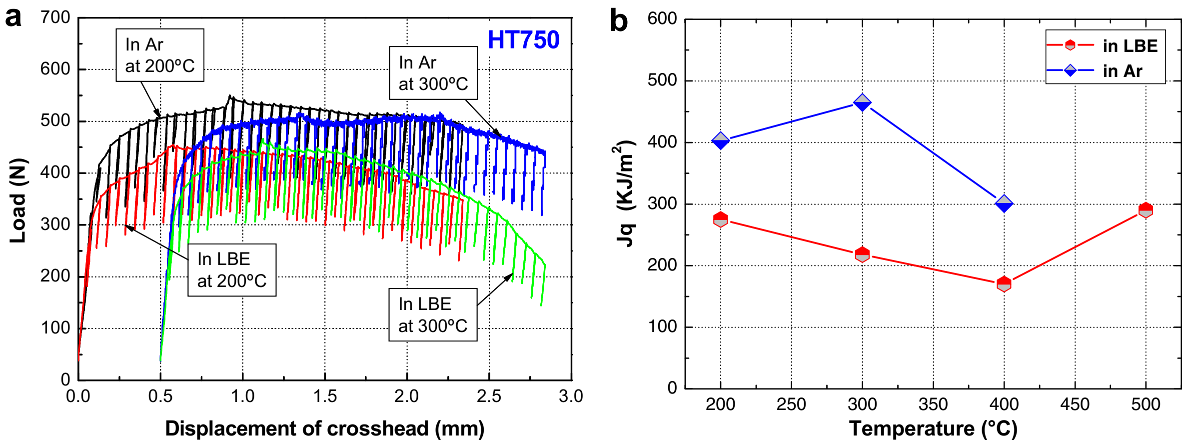

Fig. 4. (a) Load–displacement curves of the HT750 specimens tested in Ar and in LBE at 200 and 300 °C (shifted by 0.5 mm in *X*-axis); (b) *Jq*-values derived from tests at 200–500 °C in Ar (up to 400 °C) and LBE.

### 3.3. OM and SEM observations

After bending testing, most of specimens were observed with OM and SEM to identify the fracture mode. As an example, Fig. 5 shows OM pictures of the HT500 specimens tested in Ar at 300 °C and in LBE at 200–500 °C. It can be seen that the specimen tested in Ar was experienced very large bending deformation before rupture, while all the specimens tested in LBE were bent just slightly, particularly for the specimen tested at 300 °C. This illustrates a significant difference between the fracture behaviors of the specimens tested in Ar and LBE: ductile fracture in Ar but brittle fracture in LBE, which is in agreement with the conclusion given by the bending tests. Fig. 6 shows micrographs taken from the fracture surface of the HT500 specimen tested in LBE at 300 °C: (a) is the fracture surface with adherent LBE; (b) is the fracture surface after removing the adherent LBE; and (c) is a high magnification SEM micrograph on the crack propagation part. From pictures (a) and (b) one can see that the specimen was broken without any lateral expansion, which indicates a very brittle fracture mode. The SEM micrograph also demonstrates brittle cleavage fracture feature.

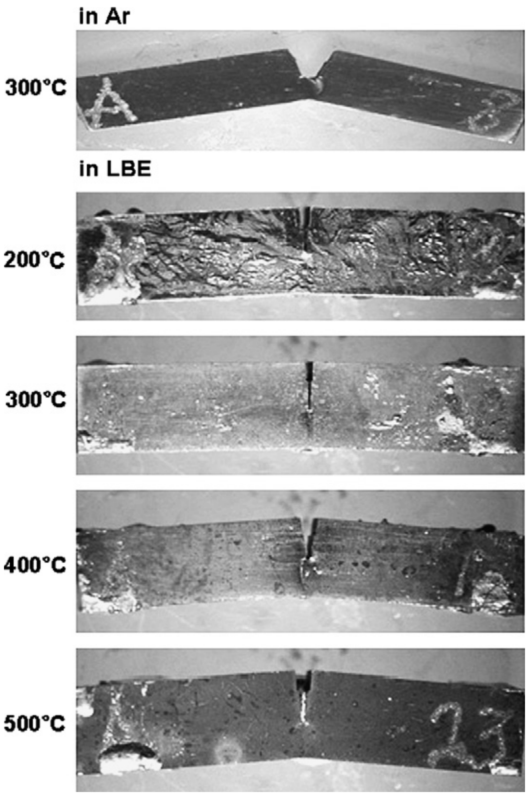

Fig. 5. Pictures of the HT500 specimens after the bending testing in Ar at 300 °C and in LBE at different temperatures.

The SEM observation illustrated that almost all specimens tested in Ar and some the HT760 specimen tested in LBE ruptured in ductile fracture mode. Most of the HT600 specimens tested in LBE ruptured in a mixed ductile-brittle fracture mode. Only the HT 500 specimens tested at 200 °C and 300 °C in LBE showed nearly pure cleavage fracture.

In the previous work, SSRT tests demonstrated that the temperature range of the ‘ductility-trough’ was broadened and the degree of the embrittlement was enhanced with increasing the strength of the steel through heat-treatment [12]. The results of the present work are in agreement with the previous observation, showing that higher is the strength of the steel, higher is the sensitivity to LBE embrittlement. This implies that the susceptibility of irradiated T91 steel increases with hardening induced by irradiation.

The *J*-value can be taken as a measure of effective surface energy of a material in a certain environment required for creating a fracture. The results of the present work will be further analyzed to evaluate the surface energy reduced by contacting with LBE. In addition, microstructure around crack tips will be investigated to reveal the mechanism of crack propagation in different situations. The results will be reported in the near future.

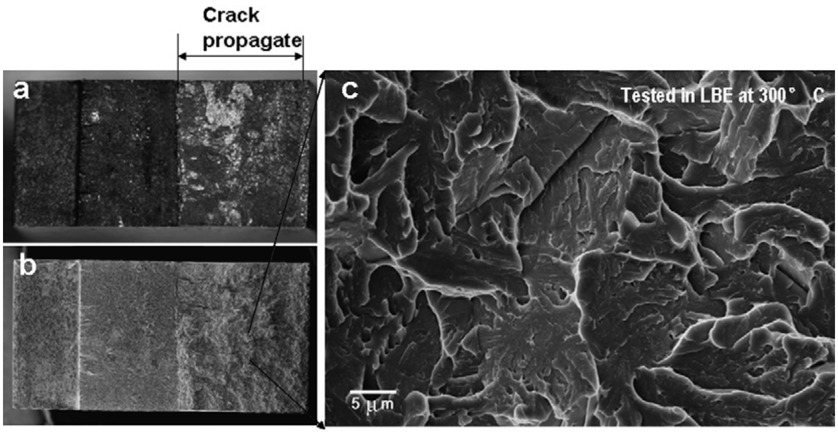

Fig. 6. Micrographs of the fracture surface of the HT500 specimen tested in LBE at 300 °C: (a) the fracture surface with adherent LBE; (b) the fracture surface after removing the adherent LBE and (c) a high magnification SEM micrograph from the crack propagation part.

## 4. Conclusions

In the present work, 3-point bending tests were conducted on specimens of the T91 steel with different strengths in LBE at temperatures between 200 °C and 500 °C. The results demonstrate that the hardening of the T91 steel has significant influence on its susceptibility to LBE induced embrittlement. The 500 °C tempered specimens with highest hardening were the most sensitive to LBE embrittlement, which resulted in brittle fracture at lower temperatures and substantial reduction of fracture toughness up to 400 °C. The 600 °C tempered specimens with medium hardening appeared quite sensitive to LBE embrittlement as well. The *J*-values reduced more than 50% in a temperature range of 200–400 °C. The specimens of non-hardened normal T91 steel illustrated also embrittlement effect. The *J*-values reduced 20–30% in presence of LBE.

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