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Slow strain rate tensile tests on T91 in static lead–bismuth eutectic

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Abstract

The embrittlement effect of liquid lead–bismuth eutectic (LBE) on martensitic steel T91 has been studied by performing slow-strain-rate tensile (SSRT) tests in static LBE with about 1 wppm oxygen at temperatures ranging from 250 °C to 425 °C. Two groups of samples were used. Group-I samples with microcracks on the lateral surfaces indicated clearly LBE embrittlement effect at temperatures ⩾300 °C, while Group-II samples without microcracks did not show the effect. The LBE embrittlement effect occurred after the necking of specimens started. The yield and ultimate tensile strengths and uniform elongation were not affected. SEM observations showed the specimens ruptured in a brittle fracture mode when the embrittlement occurred. It is concluded that the requirements for the susceptibility of LBE embrittlement effect on the T91 steel are: surface cracks or flaws, wetting and a certain level of stress concentration at crack tips.

1. Introduction

Liquid lead–bismuth eutectic (LBE) is known for its embrittlement effects on martensitic steels [1–3]. This issue is of great concern in the materials research program for developing Accelerator Driven Systems (ADS) for nuclear waste transmutation, where martensitic steels (e.g. the T91 steel) will be used as structural materials in contact with LBE. In our previous experiments, the LBE embrittlement effect was observed by conducting slow-strain-rate tensile (SSRT) tests on the T91 and F82H steels at 300 °C, as shown in Fig. 1 [1]. As these results were not well understood, the present work aimed to investigate this phenomenon by performing systematic SSRT tests on the T91 steel in a large temperature range.

2. Experimental

2.1. Material and specimen

The material used in the present study is the T91 steel which was supplied by the Ugine (France) company. Its composition is in wt%: 8.63Cr, 0.23Ni, 0.95Mo, 0.31Si, 0.43Mn, 0.1C, 0.21V, 0.02P, 0.09Nb and with Fe in balance. The as-received material was normalized at 1040 °C for 1 h and followed by air cooling, and then tempered at 760 °C for 1 h and followed by air cooling.

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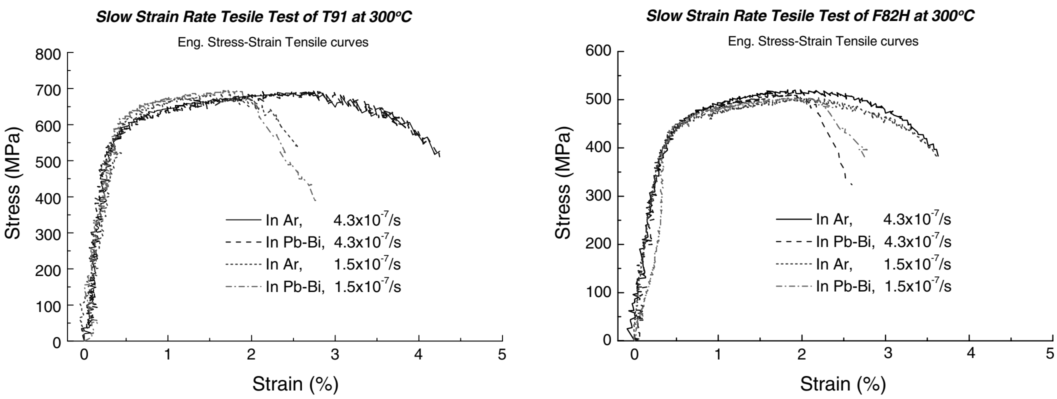

Fig. 1. Results of slow strain tensile tests on T91 (left) and F82H (right) steels in Ar and LBE at 300 °C [1].

Dog-bone shaped flat specimens were cut from the as-received plate (15 mm thick) with an electron discharging machine (EDM). The size of the specimen was $16 \times 4 \times 0.75$ mm with a gauge section of $5 \times 1.5 \times 0.75$ mm. After EDM cutting, the specimens were separated in two groups. Group-I specimens were ground progressively with 400, 600 and 1000 grit abrasive papers, while the lateral surfaces were kept as EDM cut. Group-II specimens were firstly electro-polished to remove about 20 μm from all surfaces and then followed by the same grinding procedures as applied to Group-I specimens. The difference between the two groups of specimens was such that Group-I specimens were with microcracks on the lateral surfaces introduced by EDM cutting, while Group-II ones were without.

The composition of the LBE is 44.8 wt% Pb and 55.2 wt% Bi, which was supplied by the Imbag AG, Switzerland. The impurities in the alloy are in wppm: Ag 11.4, Fe 0.78, Ni 0.42, Sn 13.3, Cd 2.89, Al 0.3, Cu 9.8 and Zn 0.2.

2.2. Testing device and procedures

The SSRT tests were performed in a Twick mechanical testing machine with 20 kN load capacity. A special device for tensile testing in LBE was developed, which was composed of three LBE tanks: a pre-melting tank – for melting LBE ingots, a storage tank – for keeping LBE constantly at 250 °C, and a test tank – for performing tensile tests at any temperatures between 150 and 500 °C in Ar + 2% H_2 atmosphere or in LBE. The oxygen content in the LBE in the storage tank was in oxygen saturated condition because an oxide layer maintained on the surface. In the test tank the oxygen concentration should increase slightly after the LBE pressed over from the storage tank because of the little oxygen content in the Ar + 2% H_2 cover gas above the LBE. According to the Ellingham diagram, the oxygen concentration in the LBE at 250 °C was less than 1 wppm in saturate condition. It is believed that the oxygen content in the LBE in the test tank should not exceed 1 wppm in all the tests at different temperatures because the additional oxygen could be obtained from the cover gas was very limited.

Since irradiated specimens will be also studied in the future using this device, the specimen grips were designed so that radioactive specimens could be easily handled with manipulators in a hot cell. After a specimen was mounted in the grips and inserted into the test tank, the tank was vacuumed to about 0.1 mbar and then washed with Ar + 2% H_2 gas. It was repeated four times in order to reduce the oxygen content inside the tank. Afterwards the specimen was heated up to the testing temperature in Ar + 2% H_2 gas and the test tank was filled with LBE. The specimen was immerged in LBE about 5 cm below the free surface and kept in the LBE for 12 h or longer before starting the SSRT test. An SSRT test took few hours. After the SSRT test, the LBE was pressed back to the storage tank. The broken specimen was taken out after the test tank was cooled down to about room temperature.

In the present work, the SSRT tests were conducted at a constant cross-head speed of 3 μm/min which corresponds to a nominal strain rate of $1 \times 10^{-5}\,s^{-1}$. This strain rate was selected as the LBE embrittlement effects were evidently observed in tests performed at 300 °C [4]. After SSRT tests, the fracture surfaces of the specimens were examined with an LEO 440 type scanning electron microscope (SEM). Prior to the SEM examination, some broken specimens were cleaned with a mixture of CH_3COOH, H_2O_2 and C_2H_5OH at a ratio of 1:1:1 to remove the LBE adhered on the fracture surfaces.

3. Results

3.1. Results of Group-I specimens

Group-I specimens were tested at eight temperatures between 250 and 425 °C in a step of 25 °C.

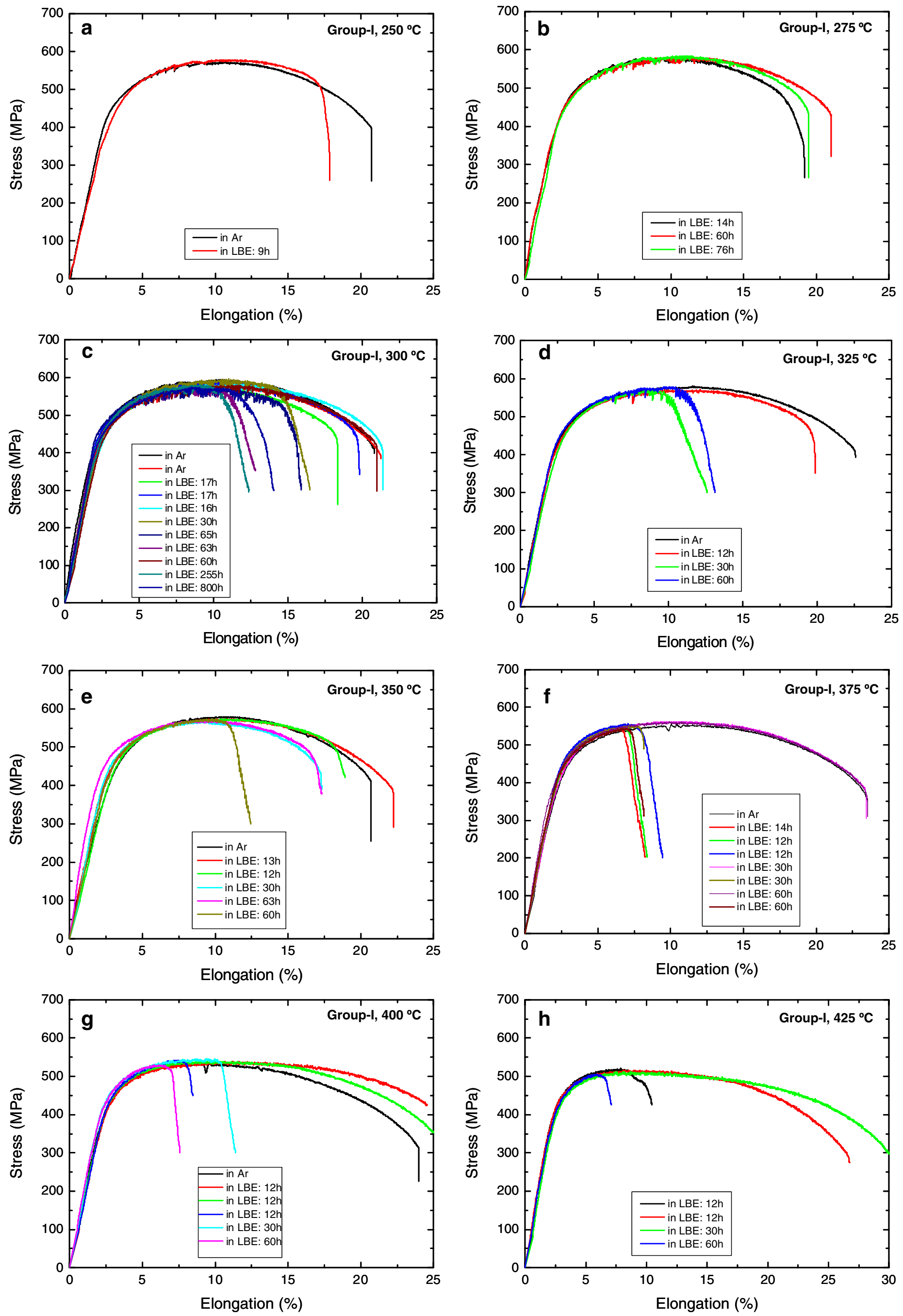

Fig. 2. Tensile stress–strain curves of Group-I specimens tested in Ar and in LBE (with ⩽1 wppm oxygen) at temperatures between 250 and 425 °C and a nominal strain rate of $1 \times 10^{-5}\ s^{-1}$.

Figs. 2a to h show the results of the specimens tested at these temperatures in LBE and in Ar as well. The time exposed to LBE before starting tensile testing is indicated for the corresponding curves, which varies from 12 to 60 h generally, but from 12 to 800 h at 300 °C. As one can see from the figures, the results demonstrate that: (1) at ⩾300 °C, the LBE embrittlement phenomenon was observed at all the temperatures, although it did not always appear; (2) the embrittlement effect started after necking and the yield and ultimate tensile strengths and uniform elongation were not affected; (3) there was no certain rule of the exposure time; (4) a general trend was that the embrittlement effect started earlier with increasing temperature, as shown in Fig. 3 which indicates that the total elongation decreases with temperature; and (5) at 250 and 275 °C the embrittlement effect was not evident.

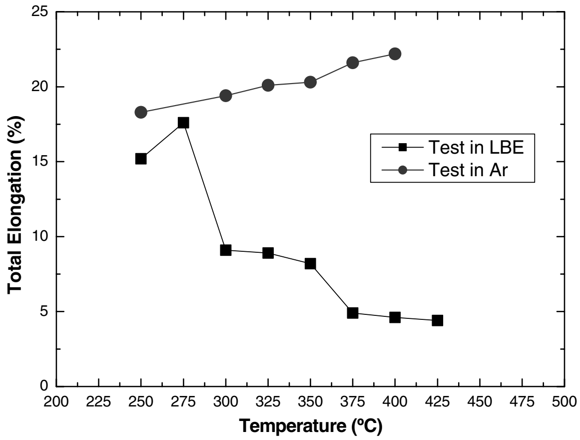

Fig. 3. The total elongation versus testing temperature for Group-I specimens tested in LBE (where the embrittlement occurs) and in Ar.

The SEM observations were performed on some broken specimens to identify the fracture mode. The examination indicated that the fracture mode of specimens tested in Ar at all temperatures was ductile, as an example shown in the left column of Fig. 4. The fracture surfaces of the specimens tested in LBE, which illustrated the embrittlement effect, showed generally brittle fracture, as can be seen from the right column of Fig. 4 for a specimen tested at 300 °C in LBE.

The propagation of microcracks on the lateral surfaces of the specimens was investigated. Fig. 5a and b illustrate the areas close to the fracture surfaces of two specimens tested at 300 °C in Ar and in LBE, respectively. One can see that the microcracks filled with LBE at tips are much deeper than those without LBE or those in the specimen tested in Ar.

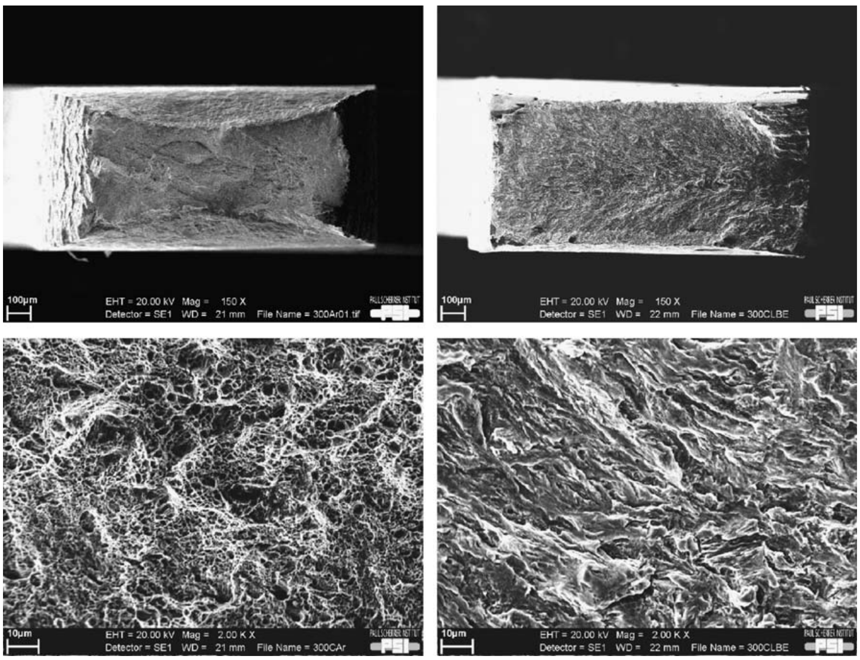

Fig. 4. Fractographs of Group-I specimens tested at 300 °C in Ar (left column) and in LBE (right column).

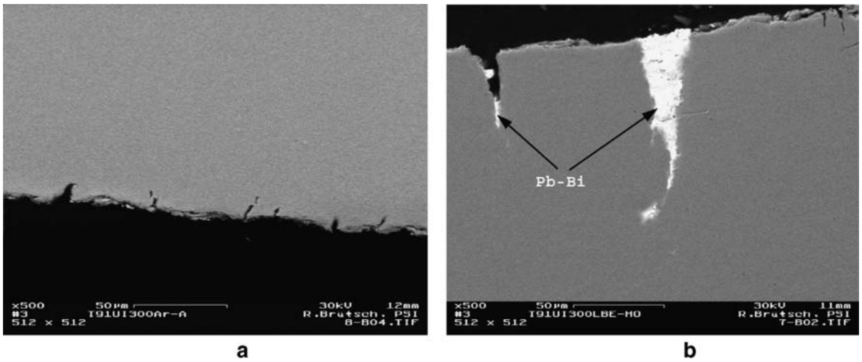

Fig. 5. Microcracks on the surfaces of Group-I specimens after tensile tests in Ar (a) and in LBE (b) at 300 °C.

3.2. Results of Group-II specimens

Group-II specimens were only tested at 300 and 375 °C. The results are presented in Figs. 6a and b, respectively. At these temperature it is clear that the specimens tested in LBE do not indicate any embrittlement effect as observed from the tests of Group-I specimens.

4. Discussion

Liquid metal embrittlement (LME) effects on solid metals have been studied for many decades. The phenomenon described earlier by Rostoker et al. is such that [5]: LME is the decrease in ductility of a metal caused by contact with a liquid metal. LME may be considered as a special case of brittle fracture. The effects of mechanical, metallurgical, physical and chemical factors on embrittlement may be explained rationally in term of the principle of brittle fracture. The present and other observations [2,3,6–8] demonstrate that the LBE embrittlement effect on martensitic steels will result in brittle fracture.

Although a lot of experiments have been performed, the mechanism of LBE embrittlement effects on martensitic steels is not yet very clear. The reason is that the embrittlement phenomenon is complicated. It may depend on many experimental parameters such as temperature, strain rate, surface condition, and perhaps oxygen concentration in LBE as well. Nevertheless, the present authors consider one of well accepted LME model which is based on the "weakening inter-atomic bond" mechanism [9,10] as a proper one. This model states that the liquid metal atoms at a crack tip in a solid metal would reduce the surface energy of the solid metal and, consequently, the strength of the inter-atomic bonds. Applying this model to the present results, it can be interpreted as: once a crack tip is filled with LBE, the Pb and Bi atoms will interact with atoms of the steel around the crack tip. This will reduce the effective energy required for crack propagation and enable the crack to propagate at a much higher speed, which results in a brittle cleavage fracture. The necessary conditions are considered to be: (1) Surface cracks or flaws are required, no matter produced in which ways

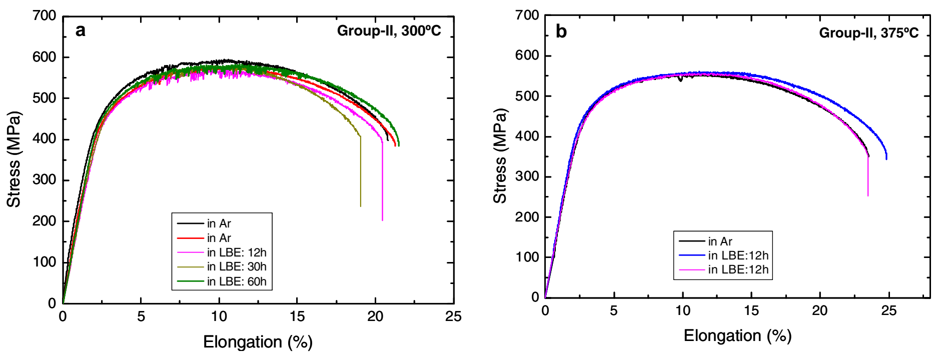

Fig. 6. Stress–strain curves of Group-II specimens tested in Ar and LBE (with ⩽1 wppm oxygen) at (a) 300 °C and (b) 375 °C and a nominal strain rate of $1 \times 10^{-5}\ s^{-1}$.

(pre-existing or induced by corrosion etc.); (2) LBE wetting at the crack tips enables direct atomic reactions between Pb/Bi and the steel; and (3) A certain stress concentration at crack tips is necessary. In what follows we try to discuss these points in more detail.

4.1. The first condition: surface cracks or flaws

The present results of Group-I and Group-II specimens demonstrate clearly that surface cracks are important for the appearance of the LBE embrittlement. Except for cracks like those observed in Group-I specimens, flaws such as pre-existing inclusions, corrosion pits (especially at grain boundaries) on surfaces may also induce LBE embrittlement when the other conditions are fulfilled. For example significant reduction in total elongation was observed in the tensile tests on T91 specimens which had transgranular and intergranular attacks of a few microns deep on the surfaces after they were exposed in LBE with low oxygen content at 400 °C [6,7].

4.2. The second condition: LBE wetting at crack tips

LME can only take place when the atoms of the liquid metal interact directly with those of the solid metal in the system, which reduces the binding strength of the atoms of the solid metal. Therefore, it is essential that the crack tips are wet by LBE. LBE wetting can be significantly affected by oxide layers on the surfaces of martensitic steels at temperatures $\leqslant\sim$450 °C. As a consequence, the LBE embrittlement phenomenon disappears or from time to time is not evident. Meanwhile, it enables the exposure time of a specimen in LBE less important, as demonstrated by the present results, because an initial thin oxide layer on the surface of the specimen can prevent wetting, particularly in stagnant LBE at lower temperatures. On the other hand, good wetting will promote the embrittlement, as shown in [6–8].

4.3. The third condition: stress concentration at crack tips

Generally, a brittle fracture requires sharp cracks with high stress concentration at the crack tips. If crack tips are blunted and the stress concentration at crack tips is relaxed, a brittle fracture is hardly to occur. For some Group-I specimens, the surface cracks might be relatively small and the LBE could not fill in (due to the surface tension of the LBE) in the first period of the tensile tests. In some specimens the cracks might be blunted before the LBE filled in them. In these cases, the specimens may not show any obvious embrittlement, as shown in Fig. 2. While for those specimens with relatively large cracks, the LBE could fill into these tips after the cracks opened slightly before the blunting occurs. The interaction of the Pb/Bi atoms with the atoms of the steel will reduce the surface energy, which will efficiently prevent the crack blunting and keep the high stress concentration at the crack tips to enable fast crack propagation, and finally induce a brittle fracture. The work is on going to establish a quantitative model to describe the stress concentration requirement for a brittle fracture induce by LBE embrittlement effect under certain conditions in respect to materials, crack geometry (size, shape and tip radius), and temperature etc.

The temperature effects, namely the embrittlement starts earlier with increasing temperature as shown in Figs. 2 and 3, can be attributed to two reasons. Firstly, the fact that the uniform elongation (or strain-to-necking) of martensitic steels decreases with increasing temperature at $\leqslant\sim$600 °C [11,12] reflects necking starts earlier at a higher temperature. As crack opening or propagating takes place mainly after necking, one can expect that the embrittlement may also start earlier at high temperatures. Secondly, the surface tension of LBE decreases with increasing temperature [13], which allows LBE easier or earlier to fill into small cracks and leads to an earlier start of embrittlement at higher temperatures. Further metallographic investigations will be performed to prove these explanations.

5. Conclusion

Slow strain rate tensile tests have been conducted on two groups of T91specimens with and without surface cracks at temperatures between 250 and 425 °C in Ar and LBE. The results demonstrate that:

(1) In the temperature range of 300 to 425 °C, the T91 steel is susceptible to LBE embrittlement. Outside this temperature range it is not clear and further studies are underway.
(2) The LBE embrittlement effect appears after necking of specimens starts. The yield and ultimate tensile strengths and uniform elongation are not affected.

(3) There is no certain rule of exposure time effects on the embrittlement observed in the present experimental conditions.
(4) The preliminary understanding is such that the essential conditions for the observed LBE embrittlement effect on the T91 steel are: surface cracks or flaws, wetting and a certain level of stress concentration at crack tips.

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