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Mechanical behaviour of the T91 martensitic steel under monotonic and cyclic loadings in liquid metals

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Abstract

The paper deals with the mechanical properties in liquid metals of the T91 martensitic steel, a candidate material for the window of an accelerating driven system (ADS). Two main questions are examined, the risk of liquid metal embrittlement and the accelerated fatigue damage by a liquid metal. It is found that the transition from ductile to brittle behaviour induced by a liquid metal is possible as a result of a decrease in surface energy caused by the adsorbed liquid metal. The embrittlement can occur only with a hard microstructure and a nucleation of very sharp defects inside the liquid metal. Under cycling straining, the fatigue resistance of the standard T91 steel is decreased by a factor of about 2 in the liquid metal as compared to air. It is proposed that short crack growth is promoted by the liquid metal which weakens the microstructural grain boundary barriers and skip the microcrack coalescence stage.

1. Introduction

In most of installations working with components under pressure, temperature, stresses, and other aggressive environments, their reliability is always of prime interest and the need to foresee scenarios where a concomitant situation of unexpected factors occurs. These unusual situations are difficult to be included by designers because they are not really well-documented phenomena and are not quantified by a mechanical value. In plants aimed to work for long time periods (power plants, petrochemical reactors, ...), the creep resistance is one of the mechanical characteristics that are needed for design. Creep is also a concern for the ADS but the presence of irradiation and liquid metal adds even more complication. In this paper, we are interested in the study of the mechanical properties of the T91 martensitic steel under monotonic and cyclic loadings. The T91 steel is selected for the window because of its good resistance to creep and swelling under irradiation. For such a structural material, two main questions will be discussed in the following. Firstly, it is necessary to find any limit conditions where the ductility typical for this steel can disappear. Besides irradiation embrittlement, liquid metal embrittlement (LME) is one of the reasons why a ductile to brittle transition could be observed since the window is in contact with a liquid Pb–Bi bath. Secondly, components in service work generally under cyclic stress or strain, e.g. because of temperature variation in required power of the plant or in the present case repeated stops of the proton beam. Fatigue damage is an inherent fact for components in service and therefore the

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second point to be examined is to determine if the liquid metal accelerates the fatigue damage process.

2. Materials

For the experiments under the monotonic loading condition, the T91 steel was supplied by Creusot Loire Industries (CLI) while supplied by Ascometal for the fatigue investigations. Their chemical compositions are given in Table 1.

The recommended heat treatment for the T91 steel consists of an austenisation at 1050 °C and air quenching followed by a tempering at 750 °C for 1 h. But there is a strong effect of the tempering temperature on hardness. While tempering at 750 °C gives the softest microstructure (HV = 220), the hardest one is obtained when the steel is tempered at 500 °C (HV = 400). This point will be considered because it is known that high strength steels are more sensitive to LME than low strength ones. So for the T91 from CLI, the effect of the tempering temperature (500 and 750 °C) will be studied while only tempering at 750 °C will be applied for the steel from Ascometal. For both steels, the microstructure was fully martensitic with an average grain size of 20 μm.

3. Liquid metal embrittlement sensitivity

3.1. Test conditions

The sensitivity to LME was studied using monotonic tensile tests on cylindrical specimens deformed at an equivalent strain rate of $4 \times 10^{-3}\ s^{-1}$ in air and in liquid metals (lead, lead bismuth eutectic, tin). Attention was paid on the effect of the tempering temperature (500 and 750 °C). In addition, stress triaxiality has also been considered by using smooth and notched specimens. The test temperature depended on the liquid metal (see Table 2).

3.2. Results and discussion

In its standard heat treatment (tempering at 750 °C) condition, the T91 steel exhibits a yield stress of 360–400 MPa (UTS: 900 MPa) with an elongation to fracture of about 22% in air and in liquid lead at 350 °C. Tempering the T91 steel at 500 °C considerably increases its yield stress up to 800–900 MPa (UTS: 1200–1300 MPa) with appreciable domain plasticity up to 15% at fracture after test at 350 °C in air and in liquid lead. Tests in air at 350 °C on notched specimens led to strong effects of strain localization, increasing the macroscopic load and reducing the elongation to fracture by a factor of two but the T91 steel remains ductile as indicated by the fracture surfaces (Fig. 1). The transition from ductile to brittle fracture occurred after testing in liquid lead at 350 °C on the notched specimen of the T91 steel tempered at 500 °C (Fig. 2). Such a transition can also occur at 260 °C in liquid lead–bismuth or in liquid tin but requires a notched specimen with a hard microstructure.

The different combinations of liquid metals and types of specimens resulting in brittle or ductile fracture are summarized in Table 2 and the typical SEM images of the ductile and brittle fracture surfaces are shown in Figs. 1 and 2 respectively. Additional results and data can be found in [1,2]. It is important to note that no intergranular fracture was observed excluding thus any effect of dissolution or liquid metal penetration at grain boundary.

The experimental conditions and mechanism for LME of the T91 steel can be summarized as follows. After immersion of the steel in the liquid metal, no intimate contact exists between the liquid metal and the iron matrix because of an oxide layer at the surface specimen. To obtain a good wetting, fresh surfaces must be generated in the liquid metal which can be achieved by

Table 1
Chemical compositions of the T91 steels according to the suppliers

Element wt%	Cr	Mo	V	Mn	Si	Ni	C	Nb	Fe
CLI	8.80	1.00	0.25	0.38	0.41	0.17	0.11	0.07	Bal.
Ascometal	8.50	0.95	0.21	0.47	0.22	0.12	0.10	0.06	Bal.

Table 2
Occurrence of ductile and brittle fracture according to the liquid metal and the type of specimen of T91 steel

	Pb 350 °C	Pb 400 °C	Pb 425 °C	Pb–Bi 260 °C	Sn 260 °C
750 °C/smooth	Ductile	Ductile	Ductile	Not tested	Not tested
500 °C/smooth	Ductile	Ductile	Ductile	Ductile	Ductile
750 °C/notched	Ductile	Ductile	Ductile	Not tested	Not tested
500 °C/notched	Brittle	Brittle	Mixed	Brittle	Brittle

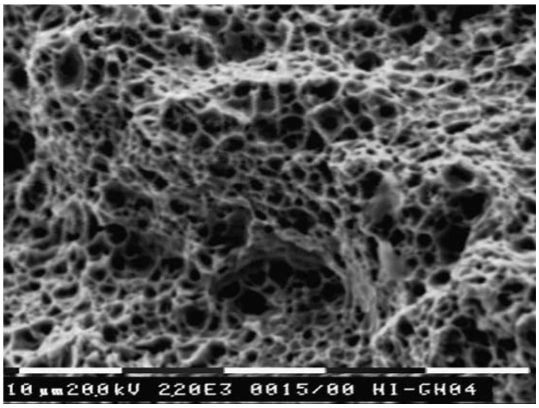

Fig. 1. Ductile fracture after test in air at 350 °C of the T91 steel tempered at 750 °C.

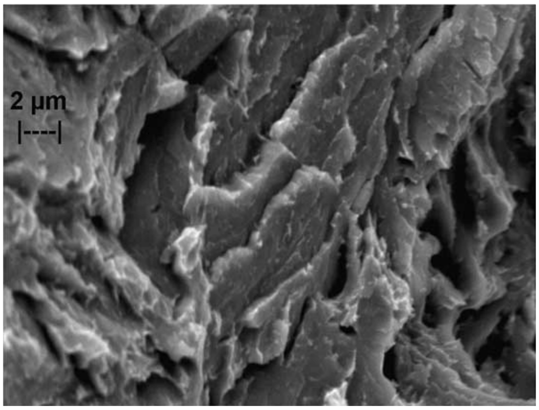

Fig. 2. Brittle fracture after test in liquid Pb at 350 °C of the T91 steel tempered at 500 °C.

microcracking at the specimen surface. At the tip of the microcrack, a strong stress triaxiality occurs provided the tip is sharp. Because of the high yield stress, plasticity in the bulk is not promoted and much less ahead the tip. Therefore, the stress intensity factor approaches the toughness which is decreased by the adsorption of liquid metal. The proposed mechanism is based on a reduction of the surface energy by liquid metals adsorption. Ab initio atomic scale simulations have allowed estimating the amount of this reduction in surface energy reduction by liquid metal atoms. More details about the calculation conditions can be found in [3]. Considering several crystallographic orientations and several adsorbed chemical species, it has been found that the variation of the reduction in the surface energy from liquid metal adsorption was between 13% and 28%.

It can be concluded that the risk of LME is possible but requires special conditions (hard microstructure and formation of a sharp defect in the liquid metal) for it to occur simultaneously.

4. Accelerated fatigue damage

4.1. Test conditions

To study the effect of a liquid metal on the fatigue properties, low cycle fatigue experiments have been carried out on the T91 steel after the standard heat treatment (i.e. tempering at 750 °C). The sensitivity was evaluated at 300 °C only with the liquid Pb–Bi (56 at.%Pb–44 at.%Bi) alloy and with tests in air. The specimens were smooth and cylindrical with a gage length of 10 mm and a gage diameter of 10 mm. The specimen surface was carefully electropolished in order to avoid any effect of the surface roughness on the fatigue life.

Tests were carried out in a fully push pull mode ($R_\varepsilon = -1$) at three strain variations $\Delta\varepsilon_t$ (2.2%, 1.6%, 0.6%). An extensometer for the strain control, a triangular wave form and a constant strain rate of 4×10^{-3} s^{-1} (thus the frequencies for each test were 0.09, 0.125 and 0.33 Hz) were used. During cycling, hysteresis loops were periodically recorded from which the stress variation $\Delta\sigma$ could be measured.

4.2. Results and discussion

The evolution of the stress amplitude $\Delta\sigma/2$ versus the number of cycles N is reported in Fig. 3 for the tests conducted in air at 300 °C. As can be seen, the T91 steel exhibits a cyclic softening very early in the fatigue life. Then a marked decrease of the stress amplitude occurs and is related with the propagation of a macroscopic crack into the bulk before final failure. This is typical for martensitic or ferrito-bainitic steels even if the adequate tempering heat treatment has been performed [4]. The same behaviour was observed when cycling in

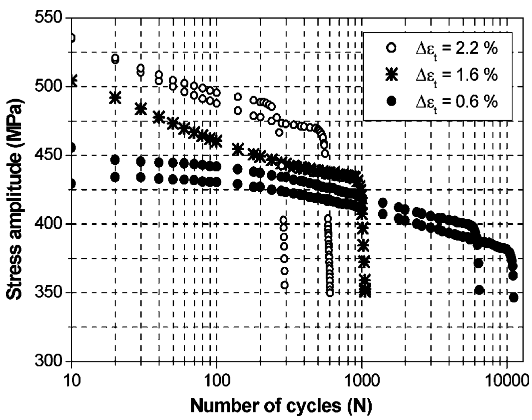

Fig. 3. Evolution of the stress amplitude versus the number of cycles in air at 300 °C at different strain amplitudes of the T91 steel tempered at 750 °C.

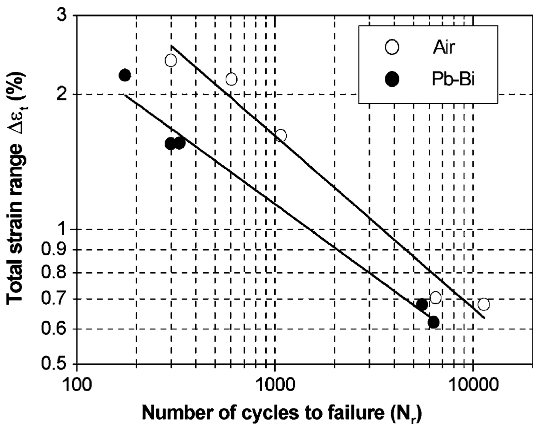

Fig. 4. Fatigue resistance diagram of the T91 steel tempered at 750 °C tested at 300 °C in air and liquid Pb–Bi alloy.

the liquid Pb–Bi alloy, that is the cyclic accommodation and the stress values do not differ from air to liquid Pb–Bi alloy. This is not surprising because the effect of the liquid metal will probably modify the surface properties as occurs in corrosion-fatigue [5] and thus does not affect the stress as would be observed in a bulk effect. However, for a given strain amplitude, the fatigue life was much smaller in liquid Pb–Bi than in air. As can be seen from Fig. 4, the fatigue resistance is decreased by at least a factor of 2. This suggests a role of the liquid metallic alloy on crack initiation and propagation. Such effect in fatigue life reduction seems typical for martensitic steels as shown by Kalkhof et al. [6] on the 10.5Cr-steel Manet-II tested in Pb–Bi at 260 °C.

The different steps in the formation of the main crack are as follows. Cyclic plasticity leads to the formation of fatigue extrusions-intrusions at the surface of the specimen. Intrusion is a favourable site for a microcrack to nucleate transgranularly. The growth of such a grain-sized crack is limited by the grain boundary which can be overcome after a given number of cycles [7]. Crack extension therefore occurs by crystallographic growth which again is limited by other grain boundaries but the length reaches now three or four grain sizes. Longer microcracks (up to 10 grain sizes) can form by coalescence of the smaller microcracks. Finally, only a very few of them propagate in a plane perpendicular to the stress axis into the bulk. Such a description is consistent with the fatigue damage mechanism observed in air. As can be observed from a transversal cut of the specimens, a high density of microcracks whose lengths are ranging from one grain size to 10 grain sizes are present near the specimen surface (Fig. 5). This means that the grain is an efficient barrier for temporary stopping the microcrack growth. However, these different steps in the damage mechanism do not reflect the behaviour in liquid Pb–Bi. Indeed, near the surface of the transversal cuts (Fig. 6), very scarce short microcracks can be observed beside the long macroscopic crack. This decrease in small crack density from air to liquid metal is in agreement with Klakhof's results [6]. This suggests that once a microcrack forms inside a grain, the grain boundary resistance to crystallographic growth vanishes when the liquid metallic alloy is in contact and allows easy extension of the crack towards the neighbouring grains. In addition, the coalescence stage no longer controls the formation of the main crack.

5. Conclusions

The monotonic and cyclic behaviours of the T91 martensitic steel in liquid metals have been investigated in order to increase the reliability of the window in an ADS. The main results are as follows:

1. In general, the T91 steel is a ductile alloy over a large range of temperatures even in contact with liquid Pb, Pb–Bi and Sn.
2. The microstructure combined with sharp crack nucleation in a liquid metal plays an important role on the occurrence of LME.
3. The main cause of LME appears to be the result of the decrease in surface energy caused by adsorbed atoms.

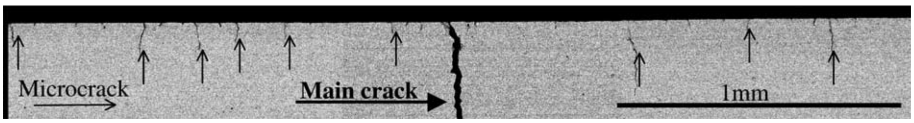

Fig. 5. Transversal cut of the T91 specimen tempered at 750 °C after fatigue failure in air at 300 °C, $\Delta\varepsilon_t = 2.2\%$.

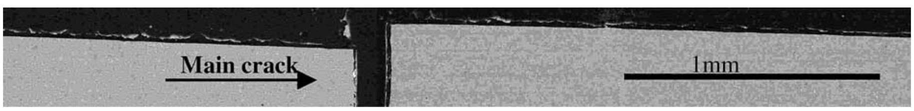

Fig. 6. Transversal cut of the T91 specimen tempered at 750 °C after fatigue failure in liquid Pb–Bi alloy at 300 °C, $\Delta\varepsilon_t = 2.2\%$.

4. The cyclic accommodation of the T91 steel consists of a softening which does not depend on the presence of liquid metal.
5. The low fatigue resistance is decreased by at least a factor 2 when cycling in liquid Pb–Bi alloy instead of air at 300 °C.
6. The liquid metal seems to enhance the short crack growth and skip the microcrack coalescence effect.

Acknowledgments

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