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Mechanical properties of martensitic steels after exposure to flowing liquid metals

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Abstract

This paper describes the tensile test results of martensitic steels EUROFER 97 and T91 after exposure to flowing Pb–17Li and Pb–55.5Bi alloy, respectively. The corrosion tests were performed in LIFUS II loop (Pb–17Li) and LECOR loop (Pb–55.5Bi) located in the ENEA Brasimone Center. Tensile test were carried in SYNTECH test machine under Ar atmosphere with an extension rate of 2 mm/min, and test temperature for EUROFER and T91 steels were at 480 and 400 °C respectively. The exposure of EUROFER steel to the liquid Pb–17Li did not affect its mechanical properties, while the ductility of T91 steel was deteriorated after exposed to flowing liquid Pb–55.5Bi.

1. Introduction

EUROFER 97 steel is a reduced activation ferritic–martensitic (RAFM) steel which is the candidates for structural materials in the water-cooled lead lithium (WCLL) concept of fusion reactors [1]. Martensitic steel T91 will be chosen as the container material of a spallation target for accelerator driven systems (ADS), which is proposed for the transmutation of nuclear waste [2]. The compatibility of structural materials with liquid metal must be considered in these two systems. Two dynamic loops were constructed in ENEA Brasimone Centre to perform this kind of studies – the LIFUS II with Pb-17Li and the LECOR with Pb–55.5Bi.

The aims of the experimental studies described in the following text are to investigate the mechanical properties of EUROFER 97 steel and T91 steel after corrosion in flowing Pb–17Li and Pb–55.5Bi, respectively.

2. Experiment

2.1. Materials and specimens

The chemical compositions of T91 and EUROFER 97 steels are shown in Table 1. The heat treatment of T91 is: normalized at 1040 °C for 60 min, and then tempered at 760 °C for 60 min. The EUROFER 97 was heat treated by normalizing at 980 °C for 30 min and tempering at 760 °C for 90 min.

Cylindrical tensile specimens with a 3 mm diameter and a gauge length of 15 mm were machined for both

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Table 1
Chemical compositions of various martensitic steels (wt%, balance Fe)

Element	Cr	Ni	Mo	Mn	W
EUROFER	8.8	–	0.003	0.44	1.15
T91	8.26	0.13	0.95	0.38	–
Element	V	Nb	Si	C	
EUROFER	0.20	0.02	0.05	0.1	
T91	0.20	0.075	0.43	0.105	

steels. There was no additional heat treatment for all the specimens after fabrication.

2.2. Corrosion test

After degreasing and measurement of dimension, the specimens were put into the loops to perform the corrosion test. EUROFER 97 was exposed in the LIFUS II loop (details are described in [3]), and T91 was in LECOR loop (for detailed description see [4]), respectively. Specimens were taken out of the liquid metal about every 1500 h and replaced by fresh ones. The maximum exposure time reached to 4500 h. The test parameters of EUROFER 97 steel and T91 steel in different loops are listed in Table 2.

2.3. Tensile test

After exposure, specimens were taken out from the test sections of the corrosion loops, and the tensile test was performed without cleaning. The tensile tests were carried at constant extension velocity of 2 mm/min corresponding to an initial strain rate of $3 \times 10^{-3}\ s^{-1}$ using an electro-mechanical SYNTECH test machine. For all tests, specimens were placed in a reservoir with Ar atmosphere, the temperature was maintained by a regulated resistor furnace. The specimens without exposure (as-received specimen) were also tested to have a comparison. The test temperature of EUROFER 97 was 480 °C, while T91 was 400 °C. The 0.2% offset yield strength (YS), ultimate tensile strength (UTS), and reduction of area (Z%) were calculated from the engineering load–elongation curves. Scanning electron microscope (SEM) was performed on the fracture surface of tensile specimens.

3. Results and discussion

3.1. Corrosion behaviour

The detailed corrosion properties of EUROFER 97 steel in flowing Pb–17Li and of T91 steel in Pb–55.5Bi are reported in literatures [3,4] respectively. Here the main results of corrosion after exposure are given.

EUROFER 97 steel suffered from typical corrosive attack due to dissolution of the steel elements in liquid Pb–17Li alloys. After 3000 h exposure, a Cr depleted layer was observed which was detached from the surface of material (see Fig. 1).

For T91, the weight measurement showed weight loss after exposure to flowing Pb–55.5Bi eutectic. The metallographic analysis showed a uniform attack around the circumference of specimens after 4500 h exposure. SEM analysis showed that the liquid metal penetrated into the bulk material on the interface of steel–liquid metal (see Fig. 2).

3.2. Mechanical properties

Fig. 3 gives the pictures of specimens of EUROFER 97 steel after tensile test. It shows that the fracture behaviour is the same before and after corrosion. SEM micrograph of the fracture surface exhibits typical ductile fracture characterized by the presence of dimples (see Fig. 4).

Table 2
The parameters of the corrosion tests

	LECOR loop	LIFUS II loop
Temperature in the test section (°C)	400	480
Temperature in the cold part (°C)	300	400
Test time (h)	1500, 4500 h	1500, 3000, 4500
Velocity in the test section (m/s)	1	10^{-2}
Liquid metal alloys	Pb–55.5Bi	Pb–17Li
Oxygen concentration (wt%)	3.1×10^{-10}–7.3×10^{-8}	Un-controlled

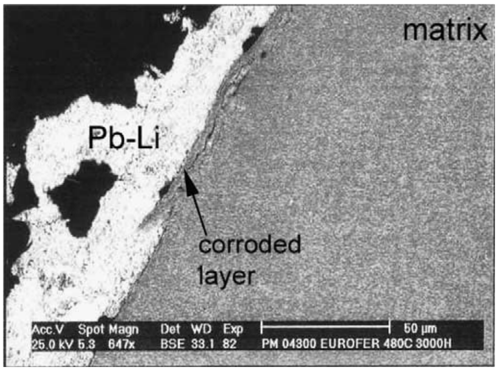

Fig. 1. SEM micrograph of EUROFER 97 after exposure to flowing Pb–17Li (T: 480 °C, t: 3000 h).

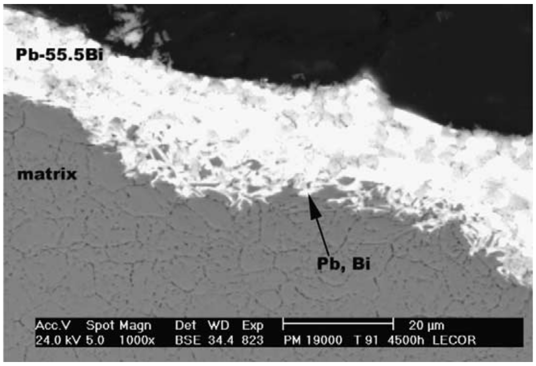

Fig. 2. SEM micrograph of T91 after exposure to flowing Pb–55.5Bi (T: 400 °C, t: 4500 h).

The main results of the tensile tests are listed in Table 3, where the average values of yield strength (YS), ultimate tensile strength (UTS) and reduction of area (Z%) are given. It can be seen that the exposure of steel to the liquid Pb–17Li did not affect its mechanical properties. Also the reduction of area of EUROFER 97 did not show degradation by liquid metal attack. This agrees with other previous results [5], from tensile tests on martensitic steel F82H-mod and OPTIFER IVb after exposure to static Pb–17Li, which also revealed there was no degradation on mechanical properties at 250 and 300 °C.

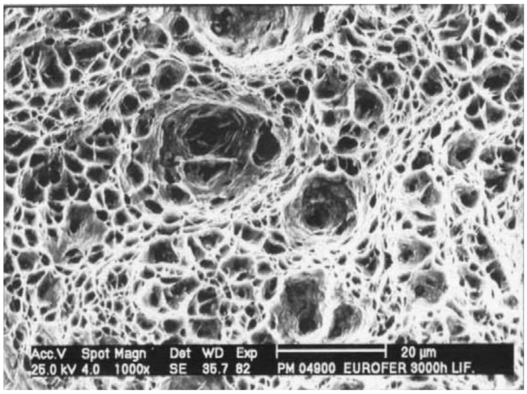

Fig. 4. SEM micrograph of fracture surface of EUROFER 97 steel (T: 480 °C, t: 3000 h).

The tensile tests of T91 steel were performed under the same conditions as those of EUROFER 97 steel except that test temperature was at 400 °C, as reported in Section 2.3. Comparison of the stress–strain curves after exposure to liquid Pb–55.5Bi to the curves of as-received specimens showed that the ductility of T91 was reduced due to the contact with liquid metal. The morphology of specimens after tensile test is showed in Fig. 5. It is very clear that the as-received specimen shows ductile fracture, while the corroded specimens exhibits some degradation in ductility. Fig. 6 gives the fracture surfaces of corroded specimens, showing a loss of ductility at the peripheral part of the specimens that exhibited a flat morphology with slip steps appearing as wavy lines. On the contrary, the central part shows ductile fracture, i.e. T91 shows a mixed brittle and ductile fracture after exposure to Pb–55.5Bi alloy.

The tensile test data of T91 are summarized in Table 4. Comparing the data on reduction of area of as-received specimens with that of corroded specimens shows that the reduction of area percent of the corroded

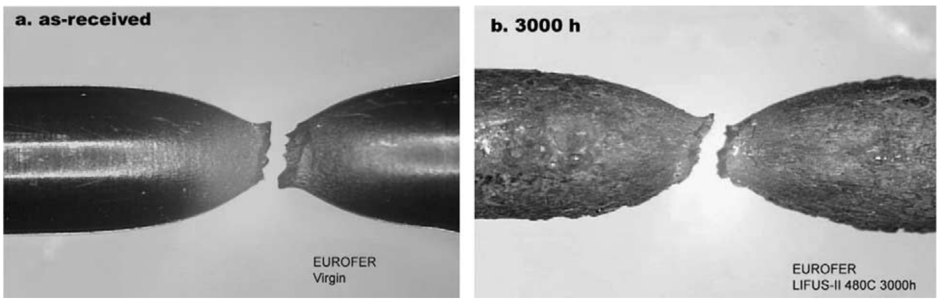

Fig. 3. The morphology of EUROFER 97 specimens after tensile test (T: 480 °C).

Table 3
The tensile test results of EUROFER'97 after exposure to liquid Pb–17Li

	YS ($R_{0.2}$) MPa	UTS (σ_b) MPa	RAZ%
As-received	435 ± 16	480 ± 28	81 ± 2
1500 h	425 ± 22	455 ± 24	79 ± 3
3000 h	400 ± 11	440 ± 16	83 ± 2

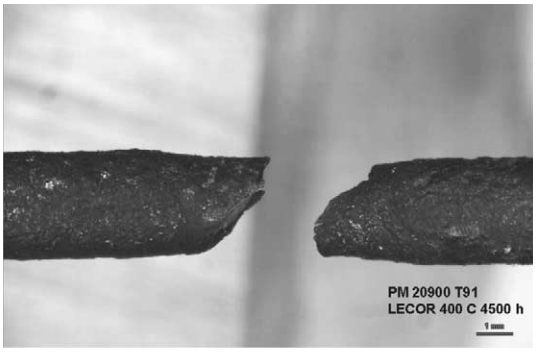

Fig. 5. The morphology of T91 specimens after tensile test (*T*: 400 °C, *t*: 4500 h).

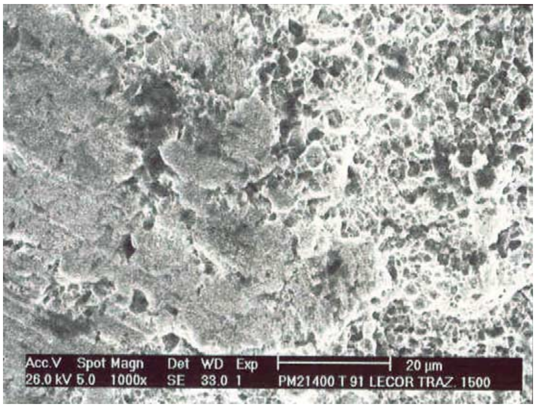

Fig. 6. SEM micrograph of fracture surface at the peripheral part of T91 (*T*: 400 °C, *t*: 4500 h).

specimens ($Z = 32\%$) is significant lower than that of un-corroded ones ($Z = 72\%$). This result is consistent with fractographic analysis.

Liquid metal embrittlement (LBE) is the decrease in ductility of a metal caused by contact with a specific liquid metal. The decrease in ductility can result in brittle failure of a normally ductile material. The specimens of T91 exhibited lose of ductility after wetting in flowing Pb–55.5Bi. It can be seen from Fig. 2 that liquid metal penetrated into the matrix on the interface of steel–liquid metal. The penetration of lead and bismuth may be one of the reasons that induced the reduction of ductility of T91. But the mechanism of liquid metal penetration is still un-clear and more experiments are needed to verify the degradation effect on mechanical properties. In most cases, the LME effect occurs just above the melting point of the embrittling liquid metal ($T_{Pb–55.5Bi} = 125$ °C), but our test results are un-consistent with this common rule. The report presented by Glasbrenner et al. [6] showed similar results for martensitic steel MANET II, where LME occurred at 250 and 300 °C. Legris et al. [7] indicated that the martensitic steel T91 was prone to LME and supposed that LME was induced by the reduction of the surface energies of the steel by the liquid metal.

The further studies are in progress in order to reveal the mechanism of liquid metal embrittlement effect on T91. The RAFM EUROFER'97 is tested now in the LECOR loop (Pb–55.5Bi) for comparison.

4. Conclusion

The results of these tests show that the EUROFER 97 steel and the T91 steel were corroded by flowing liquid Pb–17Li and Pb–55.5Bi, respectively, after they were exposed to liquid metals.

For RAFM steel EUROFER 97, the tensile test results show there is no degradation of mechanical properties after exposed to liquid Pb–17Li, with the fracture surface after tensile test exhibiting dimple–ductile fracture.

For martensitic steel T91, the tensile test results shows some degradation of ductility after exposure to

Table 4
The tensile test results of T91 after exposure to liquid Pb–55.5Bi

	YS ($R_{0.2}$) MPa	UTS (σ_b) MPa	RAZ%
As-received	535 ± 42	645 ± 26	72 ± 2
1500 h	535 ± 10	640 ± 14	42 ± 8
4500 h	515 ± 7	625 ± 15	32 ± 12

liquid Pb–55.5Bi eutectic, with the fracture surface exhibiting a mixed ductile and brittle morphology. The penetration of liquid metal element may be one of the reasons of the degradation of the mechanical properties.

Further works are on the way to verify and understand the degradation effect on mechanical property of T91 induced by liquid Pb–55.5Bi alloy.

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