Journal of Nuclear Materials 377 (2008) 261–267

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Low cycle fatigue tests of surface modified T91 steel in 10^{-6} wt% oxygen containing $Pb_{45}Bi_{55}$ at 550 °C

A. Weisenburger [a,*], A. Heinzel [a], C. Fazio [b], G. Müller [a], V.G. Markow [c], A.D. Kastanov [c]

[a] Forschungszentrum Karlsruhe GmbH, Institut für Hochleistungsimpuls-und Mikrowellentechnik, (IHM) Postfach 3640, 76021 Karlsruhe, Germany
[b] Forschungszentrum Karlsruhe GmbH, Nukleare Sicherheitsforschung, Postfach 3640, 76021 Karlsruhe, Germany
[c] CRISM PROMETEY, 193015 St. Petersburg, Russia

ABSTRACT

Low cycle fatigue tests in air and LBE containing 10^{-6} wt% dissolved oxygen were conducted with T91 steel at 550 °C. T91 was employed in two modifications, one in the as-received state, and the other after alloying FeCrAlY into the surface by pulsed electron beam treatment (GESA process). Tests were carried out with symmetrical cycling ($R = -1$) with a frequency of 0.5 Hz and a total elongation $\Delta\varepsilon_t/2$ between 0.3% and 2%. No influence from LBE on fatigue could be detected. Results in air and LBE showed similar behaviour. Additionally, no difference was observed between surface treated and none treated T91 specimens.

1. Introduction

Heavy liquid metals like $Pb_{45}Bi_{55}$ – lead bismuth eutectic (LBE) – are foreseen as a coolant and or target of an accelerator driven system (ADS) [1]. One of the main problems in the development of such a system is the compatibility of the steels with LBE.

In 1998 experiments were reported in which oxide scales formed on the surface of austenitic steels protected the steel from dissolution attack by flowing LBE [2]. For stabilization of these oxide scales a controlled concentration of dissolved oxygen in LBE has to be maintained. The concentration applied in the experiments was 10^{-6} wt%. Recent work on corrosion of austenitic and martensitic steels gives an overview on the corrosion effects and processes and its prevention in stagnant and flowing Pb and LBE [3–12]. For higher temperatures and lower oxygen content aluminizing of steels can prevent corrosion attack [13]. In addition to industrial applied aluminizing, surface alloying applying pulsed electron beams was also successfully applied to materials exposed to LBE [3,14].

The work reported so far deals with corrosion resistance of steels at relevant LBE temperatures. However, it is expected that any stress applied to materials will result in a change of corrosion and oxidation behavior and that any oxidation or corrosion scale at the surface might influence the mechanical behavior. At lower temperatures liquid metal embrittlement is of concern for steels in liquid metals [15,16]. Low cycle fatigue (LCF) life of ferritic/martensitic steels like T91 and Manet II in contact with LBE is significant lower at 260 and 300 °C [17,18].

Cladding tubes made from T91 will be operated at higher temperatures around 550 °C. From previous research work it is known that alumina scales growing on top of the surface of aluminized steels protect them against corrosion and extreme oxidation. The aim of this work is to investigate whether LBE or the modified surface layer has any influence on the LCF behavior at test temperature of 550 °C. This work describes the LCF of T91 in original and surface modified state in air and LBE.

2. Experimental

2.1. Materials

The compositions of T91 martensitic steel and of the applied FeCrAlY coating before pulsed electron beam treatment are given in Table 1. T91 is a modified 9Cr steel with additions of Nb and V to increase mechanical strength especially at higher temperatures. Before use T91 is normalized at 1050 °C and air quenched followed by tempering at 770 °C. LCF Specimens were then machined with a gauge length of 18 mm and a gauge diameter of 6 mm. Part of the specimens are coated with FeCrAlY by low pressure plasma spraying (LPPS) and subsequent pulsed electron beam treatment applying the GESA facility [19].

2.2. Methods

2.2.1. LPPS

To achieve a homogenous thin 25 μm coating conventional LPPS processes are not applicable. The particle size usually used in LPPS spraying is about 62 μm. With such coarse grained materials, of course, homogenous coatings having the desired thickness cannot

* Corresponding author. Tel.: +49 7247 82 6238; fax: +49 7247 82 2256.
E-mail address: Alfons.weisenburger@ihm.fzk.de (A. Weisenburger).

0022-3115/$ - see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2008.02.075

Table 1
Composition of T91 steel and coating before and after GESA treatment (wt%)

Element	T91	FeCrAlY orig	FeCrAlY + GESA
C	0.1025		
Si	0.22		
Mn	0.38		
P	0.021		
S	0.0004		
Ni	0.11	<0.1	<0.1
Cu	0.06		
Cr	8.99	14.3	12.8
W	0.01		
Ti	0.0034		
Mo	0.89		
Y	–	<0.5	<0.5
O	–		
Nb	0.06		
N	0.042		
V	0.21		
Al	–	7.4	5.6

be achieved. Therefore, the powders were sieved to a maximum particle size below 38 μm. In addition the spray distance, the powder feeding rate and the number of layers were optimized. Together with an improved pre-treatment all these measures finally lead to a relatively thin and homogenous coating. Like in a conventional LPPS process the coating head swings across the sample resulting in a coating consisting of several layers. The LPPS coating was performed by SULZER METCO.

2.2.2. Pulsed electron beam facility (GESA)

The surface treatment of the coated T91 steel was carried out with the GESA (gepulste Elektronenstrahlanlage) facility [19].

GESA is a pulsed electron beam facility consisting of a high voltage generator with a pulse duration control unit, a multipoint explosive emission cathode, a controlling grid and an anode which form a triode system. The electron kinetic energy can be varied in the range of 50–150 keV with an energy density up to 50 J/cm^2 at the target. The duration of a single pulse is less than 40 μs. This is sufficient to melt metallic materials adiabatically up to a depth of 10–50 μm. The beam diameter is 6–10 cm and this is the area of surface melting by applying one single pulse. Due to the high cooling rate in the order of 10^7 K/s very fine grained or even amorphous structures develop during solidification of the molten surface layer.

The appearance of the coating after treatment is shown in Fig. 1. The layering due to manufacturing process observed in the as-sprayed coating was removed and no gap between coating and substrate is visible. The turbulent nature of the process is depicted

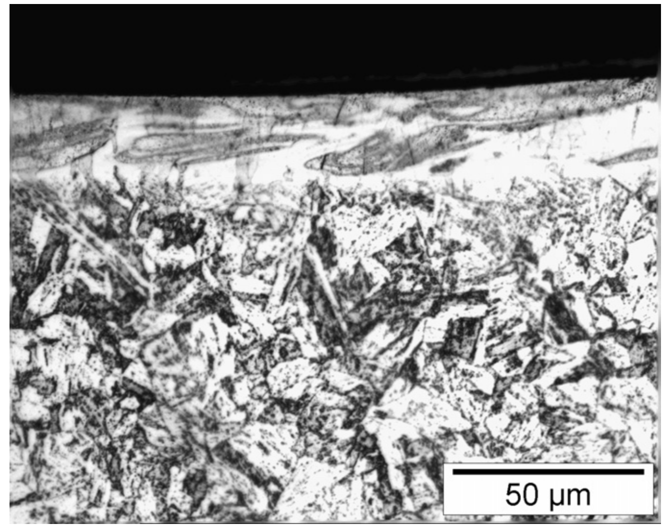

Fig. 1. LOM of etched cross section of T91 with GESA modified FeCrAlY layer showing the perfect bonding of modified coating to substrate and a whirling structure of the modified layer.

in Fig. 1 by the swirl structure in the modified surface layer. After the GESA treatment no visible interface between the modified layer and the substrate could be detected. An alloying of the layer with the substrate led to an almost perfect intermixing especially at the former interface. Therefore, after GESA treatment the term coating is no longer correct. Unfortunately, at a few places not depicted in Fig. 1 some alumina particles from the coating process were still present at the interface. This may result in reduced adhesion of the coating to the substrate.

2.2.3. Low cycle fatigue tests

The low cycle fatigue tests were carried out at PROMETEY in St. Petersburg. Before the LCF tests in LBE start the entire test section (Fig. 2(a)) including the specimen was welded into a loop at PROMETEY (Fig. 2(b)). There it was kept for 100 h at test temperature of 550 °C under controlled oxygen content of 10^{-6} wt%. The flow velocity of about 1.3 m/s was maintained by an electromagnetic pump. The oxygen control was performed via the gas phase by addition of Ar H_2 and H_2O. After 100 h the LBE in the loop was frozen and the test section was cut out and transferred to the LCF machine and implemented. There the test section was heated up again to test temperature of 550 °C and the tests were started. Each test was conducted until rupture of the specimen occurred. For com-

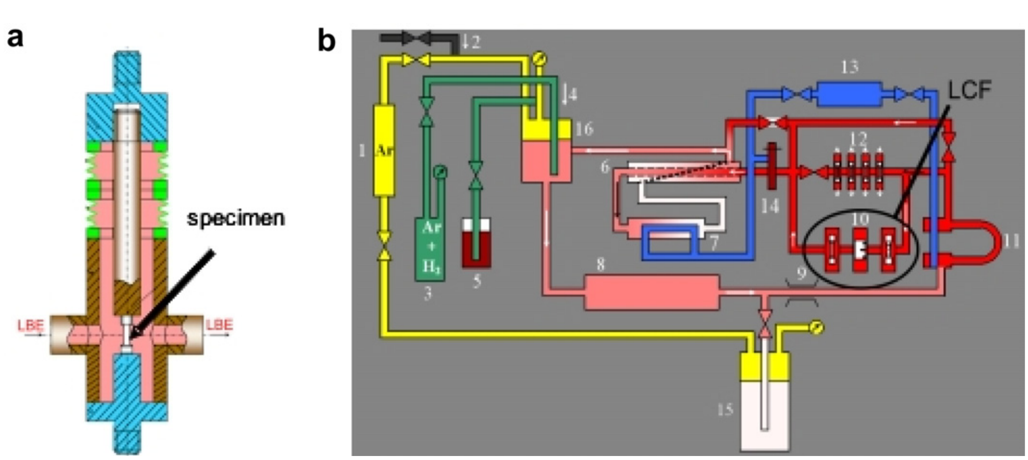

Fig. 2. Scheme of LCF test section (a) and loop (b) at Prometey, St. Petersburg.

parison and to investigate any possible influence of the liquid metal fatigue tests were also performed in air at the same temperature. Such samples were not pre-exposed to LBE.

The fatigue tests were carried out applying a servo hydraulics machine (UME-10T by Russian Electronic Mechanical Device) with a maximum load capacity of 100 kN. The tests were performed under total axial strain control $\Delta\varepsilon_t/2$ from 0.3% to 2%. All tests were conducted under a fully push pull mode ($R = -1$), a triangular waveform and a constant frequency of 0.5 s^{-1}. The oxygen activity was not controlled during the test, but due to the test procedure and duration no change in oxygen content was expected.

2.2.4. Evaluation

Light optical (LOM) and scanning electron (SEM) microscopy (S-800, Hitachi) were employed for observation of the fracture surface and side view of the fracture. Some specimens were cut using a diamond disk saw perpendicular to the surface near the broken area and were grinded and polished for metallographic examinations. To investigate the microstructure polished cross sections were etched using Vilella's reagent with addition of HNO_3. SEM and LOM of cross sections to evaluate the coating performance after the LCF test were additionally performed.

3. Results and discussion

3.1. Influence of air and LBE on cyclic stress response

The influence of LBE on the fatigue behavior of T91 with and without GESA modified surface layer are depicted in Figs. 3 and 4. The evolution of stress amplitude $\Delta\sigma/2$ is plotted versus the number of cycles to failure (N). T91 in original state tested in air showed an almost similar behavior like the specimens tested in

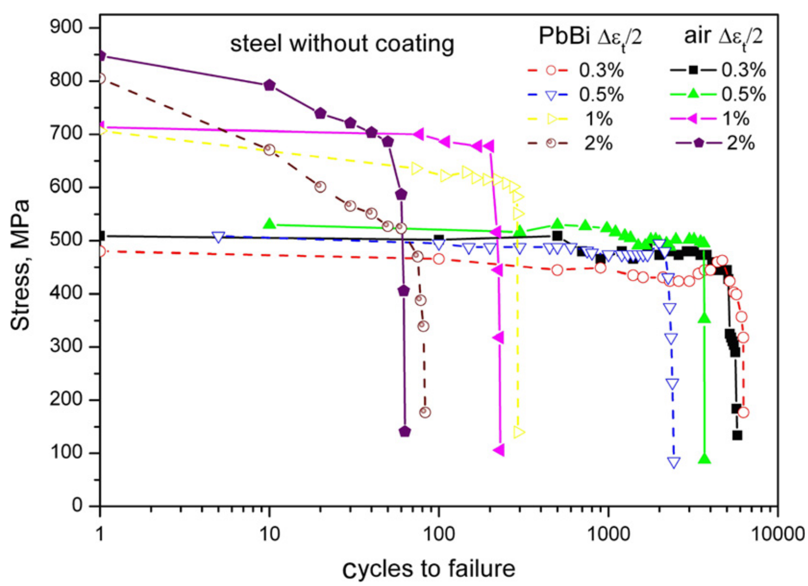

Fig. 3. Stress over cycles to failure, comparison of LCF tests of T91 in as-received state in air and LBE at 550 °C.

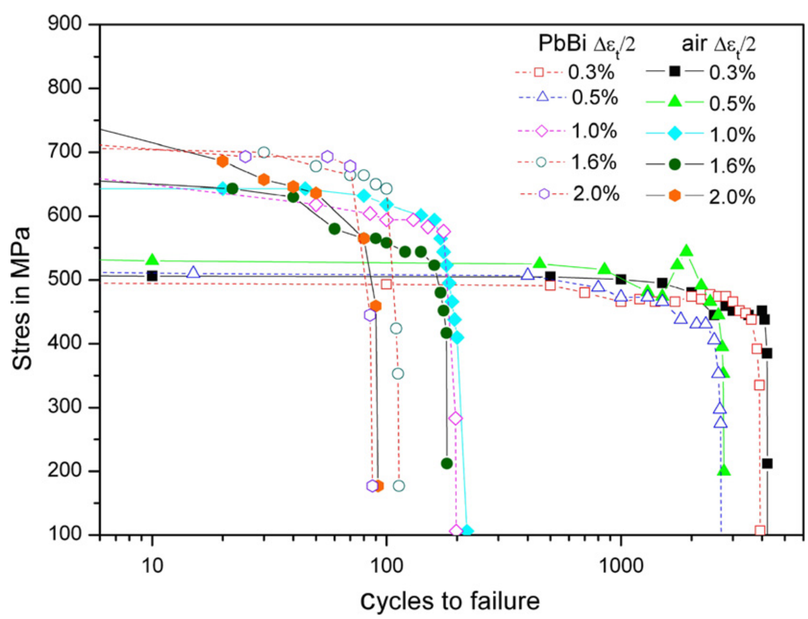

Fig. 4. Stress over cycles to failure, comparison of LCF tests of T91 with GESA modified FeCrAlY coating in air and LBE at 550 °C.

LBE (Fig. 3). The number of cycles to failure is almost identical in LBE and air for each tested strain level. The lower stress amplitude for specimens tested in LBE is mainly due to better temperature homogeneity in LBE compared to tests conducted in air. At all strain levels a slight softening of the materials in air and LBE is observed like in previous experiments performed in air [20]. For higher strain rates $\Delta\varepsilon_t/2$ of 2% this is most obvious. Cyclic softening is a common feature of tempered martensitic steels containing a high density of dislocations. The specimens coated with FeCrAlY and subsequent GESA treatment again show no influence of LBE on the fatigue strength (Fig. 4). The number of cycles to failure and the corresponding stress amplitudes is almost identical in LBE and air for strain levels $\Delta\varepsilon_t/2$ below 1% and above 1.6%. At a strain level of 1.6% the specimens tested in LBE breaks after half the time compared to the specimen tested in air. This is most probably due to some rare imperfections on the surface after coating and GESA treatment.

3.2. The influence of GESA modified FeCrAlY layer on the low cycle fatigue of T91

One major aim of this LCF experiments was to determine whether the GESA modified FeCrAlY layer on top of T91 has any negative influence on the fatigue behavior. The evolutions of stress amplitude $\Delta\sigma/2$ versus N of T91 with and without GESA modified layer are plotted separately for tests in air and LBE to compare the influence of the modified layer (Figs. 5 and 6). In air, at low strain levels <0.5%, the samples with GESA modified layer break slightly earlier, but with very similar stress amplitude. At higher strain level > 1% the stress level is slightly reduced for samples

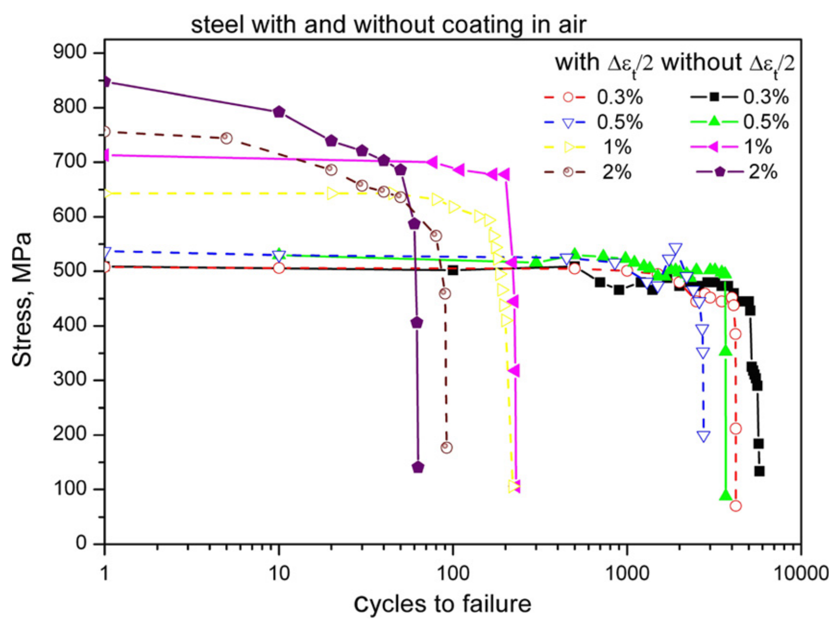

Fig. 5. Stress over cycles to failure, comparison of LCF tests of T91 in as-received state with GESA modified FeCrAlY coating in air at 550 °C.

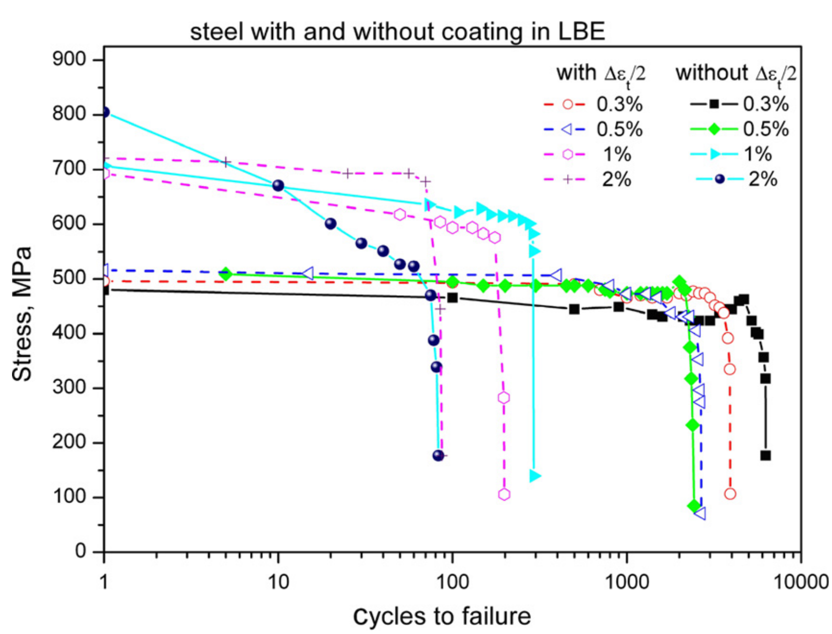

Fig. 6. Stress over cycles to failure, comparison of LCF tests of T91 in as-received state with GESA modified FeCrAlY coating in LBE at 550 °C.

with GESA modified surface layer tested in air. The number of cycles to failure N is almost identical for samples with and without modified surface tested at a strain level of 1%. At the highest strain of 2% specimens without GESA modified surface layer show a slight reduction in lifetime. The same comparison but for tests in LBE is depicted in Fig. 6. Like in test performed in air no significant differences between samples with and without GESA modified layer can be detected.

3.3. Fatigue life of T91 with and without modified coating in air and LBE

The total strain $\Delta\varepsilon_t$ is the sum of the plastic $\Delta\varepsilon_p$ and elastic $\Delta\varepsilon_e$ strain. According to Basquin, Manson and Coffin [21–23] the elastic and plastic portion of the total strain at mid-life can be fitted as follows:

$$\Delta\varepsilon_t = \Delta\varepsilon_e + \Delta\varepsilon_p = K_e(N)^{C_e} + K_p(N)^{C_p},$$

Table 2
Coefficient of elastic and plastic strain of T91 – Coffin–Manson plot

		K_e	C_e	K_p	C_p
Without coating	Air	0.502	−0.09	20.52	−0.61
	LBE	0.47	−0.093	22.9	−0.607
With GESA modified coating	Air	0.47	−0.09	30.84	−0.64
	LBE	0.47	−0.09	27.14	−0.624

where K_e is the elastic coefficient, K_p the plastic coefficient, C_e the elastic exponent and C_p the plastic coefficient, their values are listed in Table 2. The evolution of plastic and elastic strain as a function of number of cycles to failure is depicted in Fig. 7. The elastic part of the samples with GESA modified surface shows no difference between LBE and air. Concerning the elastic part of T91 in original state a slightly higher strain for samples tested in air can be found. This again is mainly due to better temperature homogeneity in LBE compared to air. The plastic strain curve of the GESA modified samples is like for the elastic strain almost identical in LBE and air. T91 specimens without modified layer shows in air a very limited decrease in fatigue resistance compared to LBE. For all the specimens the share of the elastic part becomes dominant at lower total strain amplitudes. Therefore at low total strain levels the fatigue life is practically independent from the plastic deformation.

3.4. Fracture surface observation and microstructure

The morphology of broken specimens is not different for specimens tested in air and LBE (Fig. 8). T91 specimens without GESA modified surface layer examined in air and LBE showed a flat fracture surface almost perpendicular to the loading axis (Fig. 8 left). The strain level seems to have no major influence on the appearance of the broken areas; only at strain rates above $\Delta\varepsilon_t/2 = 1.6\%$ a slightly inclined fracture is visible. A macro- and microscopic view on top of the fracture is depicted in Fig. 9. The typical striations

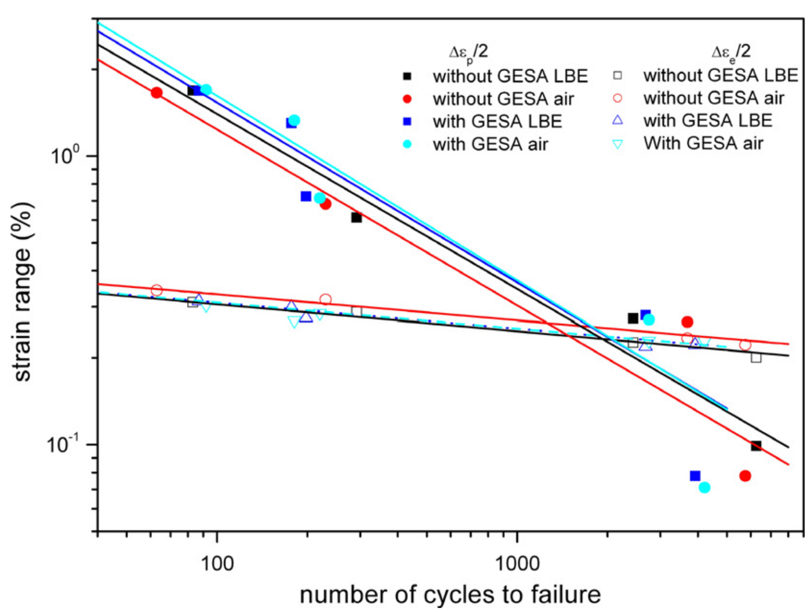

Fig. 7. Fatigue life (Coffin–Manson plot) as a function of elastic and plastic strain; dots are measured values, solid lines are fitting lines to data points.

Fig. 8. Morphology of surfaces after tested to failure, strain level for all specimens $\Delta\varepsilon_t/2 = 0.8\%$.

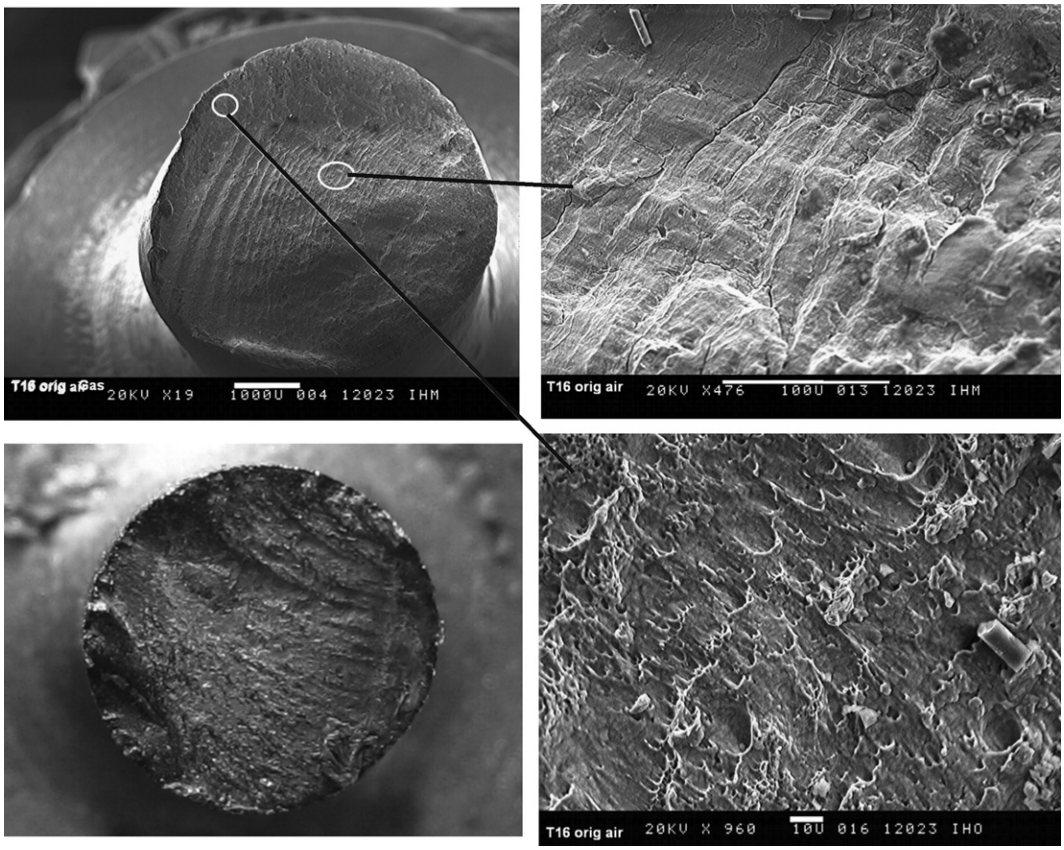

Fig. 9. SEM of fracture surface at different levels; upper left: macroscopic view of T91 in as-received state tested with 1.6% strain; lower left T91 with modified layer tested with 0.8% strain. Right: magnification of in areas indicated in upper left figure. Upper showing cracks parallel to the striations, lower showing dimples typical for ductile rupture.

resulting from the low cycle fatigue test are shown in the upper left part of the figure. Only one initiation point for cracks, starting from the surface, perpendicular to the loading axis can be determined. The two SEM pictures on the right show higher magnification of the areas indicated in the macro view. The area with smaller fatigue striations shows in the magnification many fine cracks parallel to the striations. The area of forced rupture, depicted in the lower right picture, clearly displays the dimples that are typical for ductile ruptures. As expected the spacing between the striations increases with increasing strain levels. The specimens with GESA modified surface showed especially at lower strain levels, $\Delta\varepsilon_t/2 < 1.6\%$, either in air or in LBE, a rough-textured surface that is inclined to the loading axis (Fig. 8, right) indicating multiple initiation sides for crack propagation (Fig. 9 lower left). This is obviously a small difference between samples with and without GESA modified surface layer. At a strain level of 2% the fracture surface of T91 with and without GESA treatment is practically identical. The overall microscopic appearance of the fracture surface of GESA modified samples is not significantly different from the T91 in original state.

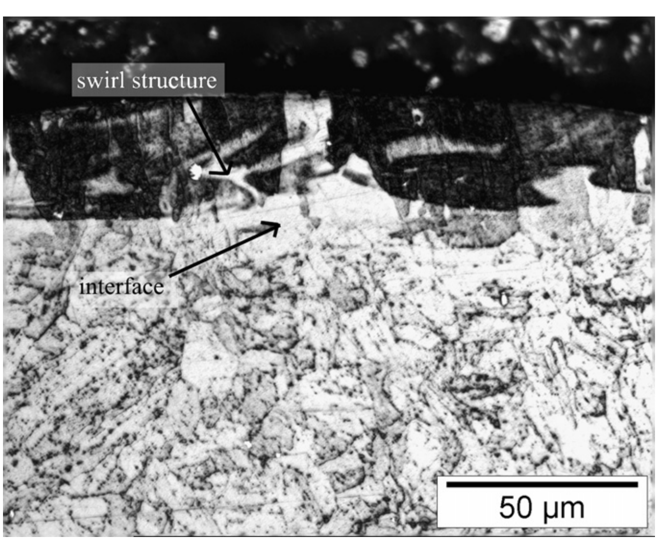

Fig. 10. LOM of etched cross section of T91 with GESA modified FeCrAlY layer after LCF test with strain of 2% tested in LBE showing the still perfect bonding of modified coating to substrate and a whirling structure of the modified layer.

All examined data so far do not indicate any significant influence of the GESA modified FeCrAlY layer on top of the T91. To prove finally whether the modified surface layer undergo any change in microstructure or detach somehow from the bulk a cross section near the rupture was investigated using SEM (Fig. 10). The unchanged modified layer and the still perfect interface between bulk and surface layer are clearly depicted in Fig. 10. No alumina particles and no detaching at the interface could be observed.

4. Conclusions and summary

The influence of a corrosion resistant layer modified with a pulsed electron beam (GESA) and of LBE on the low cycle fatigue behavior of T91 at 550 °C was examined. The liquid metal at test temperature showed, contrary to tests at 300 °C, no influence at all on the fatigue properties, even the duration of the tests and the LBE specimen contact time were comparable. The fatigue life and also the macroscopic and microscopic appearance are basically identical for specimens tested in both environments. That clearly indicates that the pre-exposure of specimens for 100 h in LBE prior to testing in LBE did not modify the low cycle fatigue properties under test conditions. The second important issue of these tests was to determine the influence of the GESA modified layer on the fatigue behavior. Concerning fatigue life no difference compared to specimens in as-received state could be detected. Only the fracture morphology at lower strain levels indicates some slight differences, but again without affecting the fatigue properties. Whether this is a general behavior of all GESA modified layers or only effect of surface or interface imperfections cannot be concluded after these experiments.

Summarizing the results, at test conditions the GESA modified layer and the test medium (LBE or air) did not change the low cycle fatigue behavior of T91.

Acknowledgement

The authors acknowledge the financial support of this work by the European Union under the IP project EUROTRANS FI6W-CT2005-516520.

References

[1] C. Rubbia, J.A. Rubio, S. Buono, F. Carminati, Conceptual Design of a fast Neutron Operated High Power Energy Amplifier, CERN/AT/95-44 (ET), 29 September 1995.
[2] I.V. Gorynin, G.P. Karzov, V.G. Markov, V.S. Lavrukhin, V.A. Yakovlev, in: Proceedings of the Conference HLMC 98, Obninsk, SSC RF-IPPE, vol. 1, 1999, p. 120.
[3] G. Müller, G. Schumacher, F. Zimmermann, J. Nucl. Mater. 278 (2000) 85.
[4] H. Glasbrenner, J. Konys, G. Mueller, A. Rusanov, J. Nucl. Mater. 296 (2001) 237.
[5] C. Fazio, G. Benamati, C. Martini, G. Palombarini, J. Nucl. Mater. 296 (2001) 243.
[6] G. Benamati, C. Fazio, H. Piankova, A. Rusanov, J. Nucl. Mater. 301 (2002) 23.
[7] Ph. Deloffre, A. Terlain, F. Barbier, J. Nucl. Mater. 301 (2002) 35.
[8] G. Müller, A. Heinzel, J. Konys, G. Schumacher, A. Weisenburger, F. Zimmermann, V. Engelko, A. Rusanov, V. Markov, J. Nucl. Mater. 301 (2002) 40.
[9] R. Ballinger, J. Lim, E. Loewen, The effect of silicon on the corrosion of iron on lead–bismuth eutectic, in: Proceedings of the 11th International Conference on Nuclear Engineering, Tokyo, 2003, No. ICONE11-36531.
[10] M. Takahashi, H. Sekimoto, K. Ishikawa, N. Sawada, T. Suzuki, K. Hata, S.Yoshida, S. Qiu, T. Yano, M. Imai, Experimental study on flow technology and steel corrosion of Lead–Bismuth, in: Proceedings of the11th International Conference on Nuclear Engineering, Arlington, Virginia, 2002, No. ICONE10-22226.
[11] Y. Kurata, M. Futakawa, K. Kikuchi, S. Saito, T. Osugi, J. Nucl. Mater. 301 (2002) 28.
[12] T. Furukawa, K. Aoto, G. Müller, G. Schumacher, A. Weisenburger, A. Heinzel, F. Zimmermann, J. Nucl. Sci. Technol. 41 (3) (2004) 265.
[13] Ph. Deloffre, F. Balbaud-Célérier, A. Terlain, J. Nucl. Mater. 335 (2004) 180.
[14] G. Mueller, V. Engelko, A. Weisenburger, A. Heinzel, Vacuum 77 (2005) 469.
[15] A. Legris, G. Nicaise, J.-B. Vogt, J. Foct, J. Nucl. Mater. 301 (2002) 70.
[16] J.-B. Vogt, G. Nicaise, A. Legris, J. Foct, J. Phys. IV France 12 (2002) 8.
[17] A. Verleen, J.-B. Vogt, I. Serre, A. Legris, Int. J. Fatigue 28 (8) (2006) 843.
[18] D. Kalkof, M. Grosse, J. Nucl. Mater. 318 (2003) 143.
[19] V. Engelko, B. Yatsenko, G. Mueller, H. Bluhm, Vacuum 62 (2001) 211.
[20] A. Nagesha, M. Valsan, R. Kannan, K. Bhanu Sankara Rao, S.L. Mannan, Int. J. Fatigue 24 (12) (2002) 1285.
[21] O.H. Basquin, Proc. Am. Soc. Test. Mater. 10 (1910) 625.
[22] S.S. Manson, Exp. Mech. 5 (1965) 193.
[23] L.F. Coffin, Jr., Transactions of ASME, vol. 76, 1954, pp. 931–950.