



Surface modification/alloying using intense pulsed electron beam as a tool for improving the corrosion resistance of steels exposed to heavy liquid metals

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ARTICLE INFO

Article history:

Available online 29 April 2011

ABSTRACT

The alloying of steel surface with aluminum (Al) using Microsecond-pulsed Intense Electron Beams (MIEB-Al) was developed and optimized in order to be used for improving the corrosion resistance of the 316, 1.4970 and T91 steels, exposed to liquid Pb and Pb–Bi-eutectic. The procedure consists in two steps: (i) coating the steel surface with Al or an Al-containing alloy layer and (ii) melting the coating layer and the steel surface layer using intense pulsed electron beam. In order to cover the steel surface with an homogeneous and crack-free Al-alloyed layer, the following experimental conditions are required: Al coating thickness range 5–10 μm , electron kinetic energy 120 keV; pulse duration 30 μs ; energy density 40–45 J/cm^2 ; number of pulses 2–3.

Using the mentioned procedure, the corrosion resistance of the 316, T91 and 1.4970 steels, exposed to Pb and Pb–Bi-eutectic with different oxygen concentrations and under different temperatures, was considerably improved due to the formation of a thin alumina layer (which thickness is lower than 1 μm for all the tested temperatures and durations) acting as an anti-corrosion barrier.

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1. Introduction

Heavy liquid metals (HLM), such as lead and lead–bismuth-eutectic alloys (LBE), are considered working fluids for advanced nuclear applications due to their thermal and neutronic properties [1,2]. The reference structural materials, selected for the nuclear systems using HLMs, are ferritic–martensitic (F/M) steels and austenitic steels. Concerns related to the use of HLM arise from the materials compatibility, in terms of corrosion and mechanical resistance. Previous studies showed that the corrosion mechanism of these steels in LBE is affected by several parameters, such as the oxygen activity in the liquid metal and the temperature. With appropriate oxygen activity in HLM, the dissolution of steel elements in the liquid metal can be minimized or suppressed through the formation of a protective oxide scale on the steel surface [3–9]. As far as the temperature effect is concerned, it was shown that austenitic steels suffer from severe corrosion attack in lead or LBE melt at temperatures above 500 °C, while F/M steels form thick oxide scales, which affects the efficiency of the heat transfer. According to [7], 316L, 1.4970 and MANET steels are suitable up

to 550 °C and 7200 h, but lead–bismuth infiltrations are observed in the oxide layer. Destabilization of the oxide layer occurs on 316L steel above 7200 h. Therefore application of both steels should be restricted to temperatures below 500 °C.

In order to improve the corrosion resistance of the steels exposed to oxygen-containing HLM above 500 °C, one possibility is to change the oxidation behavior of the steels. In previous works it was shown that this can be achieved by alloying the steel surface with Al, using a procedure called GESA (Gepulste Elektronstrahl Anlagen–pulsed electron beam installations.) process [5,10,11]. The goal of Al alloying into the steel surface is to form an alumina scale as a result of the interaction with the oxygen dissolved in HLM. This goal has been reached several times [12,13], as the same kind of coatings allowed to increase the corrosion resistance of steels in HLM. The authors concluded that the formation of a thin alumina scale was responsible of this improved behavior [12,13].

The procedure consists in two steps: (i) coating the steel surface with Al or Al-containing alloy and (ii) melting both the coating and the steel surface by irradiation with Microsecond-pulsed Intense Electron Beams (MIEB). Such treatment causes the mixing of the steel elements with the coating elements, finally leading to a modified Al-containing layer on the steel surface. The thickness of the Al-containing layer (10–30 μm) is around the penetration depth

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of electrons into the steel. Therefore, by applying the MIEB-AL procedure, the microstructural properties of the substrate materials (excepting the superficial layer) do not change and it is possible to obtain a surface layer with a uniform distribution of Al, of controlled and uniform thickness, which is crack-free and adherent to the substrate. The procedure is susceptible for industrialization and involves relatively low energy and time consumption.

The development of the MIEB-AL method and the optimization of the procedural parameters in order to obtain an Al-alloyed surface layer, with the above-mentioned characteristics, on flat and tube specimens was one of the goals of two research projects, funded by ISTC and EC (Project No. 2048 and “EUROTRANS” Project No. FI6W-CT-2004-516520).

Two austenitic steels, the 316 (Fe, <0.03 wt.% C, 16–18.5 wt.% Cr, 10–14 wt.% Ni, 2–3 wt.% Mo, <2 wt.% Mn, <1 wt.% Si) and the 1.4970 (Fe, 16 wt.% Cr, 14 wt.% Ni, 2 wt.% Mn, 0.9 wt.% Si, 0.5 wt.% C), and one ferritic–martensitic steel, the T91 (9 wt.% Cr, 1 wt.% Mo, 0.4 wt.% Si, 0.4 wt.% Mn, 0.1 wt.% C), have been considered as candidates for the construction of the core components of advanced nuclear systems.

In the present paper a selection of the most relevant experiments, results and conclusions, related to the MIEB-AL procedure development and parameters optimization, will be presented. Some edifying results, obtained during testing of Al-surface alloyed samples, made out of the above-mentioned steels and exposed to HLMS, under representative conditions, are also reported.

2. Experimental

The modification/alloying of steel surfaces, using the MIEB-AL procedure, is intended to be used for improving the corrosion resistance of fuel elements claddings in nuclear systems using HLM as coolants. The foreseen claddings are tubes with a diameter of 8.5 mm and thickness of 0.5 mm. The experimental work was carried out on tubular samples of the mentioned diameter and thickness. The length of the samples varied in the range of 20–150 mm. Flat samples were also used for the optimization of the MIEB-AL procedure.

As a first step, the specimens made out of the steels were coated with Al or an Al-containing alloy by vacuum-arc deposition.

The irradiation of the coated samples with a microsecond-pulsed intense electron beam was performed using the GESA-1 facility, described in Ref. [14]. The main characteristics of the facility are: accelerating voltage 100–150 keV, beam energy density 20–50 J/cm² and pulse duration 20–35 μ s. The beam has a cylindrical shape with a diameter of 5–6 cm. For these e-beam parameters, the calculated thickness of the modified layer lies in the range of 10–25 μ m.

The corrosion and creep-to-rupture tests were performed using the facilities available at IPPE, Obninsk and CRISM “Prometey”, St. Petersburg, [13,15,16]. These facilities allow specimens testing in lead and LBE, under a wide range of conditions: temperatures from 400 °C to 700 °C, flow velocities from stagnant up to turbulent flow, oxygen concentrations from 10^{−8} wt.% up to saturation level, isothermal and non-isothermal conditions, with and without mechanical load.

After completion of the tests, the evaluation of the specimen microstructure and chemical composition was performed using Light Microscopy (LM), Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray spectroscopy (EDX).

3. Results

3.1. Development and optimization of MIEB process

The Al concentration, the thickness and the homogeneity of the modified superficial layer depend on the MIEB process parameters, such as energy density and number of pulses, as well as on the steel composition, surface preparation and coating thickness.

Homogeneous Al coatings, with the thickness in the range of 5–20 μ m, were deposited on flat and cylindrical specimens using vacuum-arc deposition (Fig. 1a).

The MIEB process, which allows the melting of a large surface area to a depth from a few to several tens of micrometers, was used for mixing the coating with the steel matrix. The flat specimens were irradiated directly, while tubes were irradiated through a diaphragm, with a sample rotation of 30° after each pulse (Fig. 1b).

In all the experiments the accelerating voltage was 120 kV and the energy density at the target was varied from 20 to 50 J/cm². These values correspond to a variation of the pulse duration from 20 to 35 μ s. Fig. 2a shows that the MIEB with the energy density in the range 20–50 J/cm² provides a modified layer with the thickness from 10 to 25 μ m, for an initial 10 μ m Al coating thickness. It should be mentioned that the melting depths depend on the coating thickness. The melting depths were calculated using the ORION code [17]. The coating thickness influences also the Al concentration and distribution in the modified layer as depicted in Fig. 2b: higher coating thickness leads to higher Al content in the melted layer. However, cracks were observed at the surface of the samples coated with more than 10 μ m.

The experiments showed a variation of the Al concentration profiles perpendicular to the surface which depends on the number of MIEB pulses and the initial thickness of the Al coating. The analysis shows that Al penetrates into the steel within the molten surface layer. Fig. 3 depicts the Al concentration profiles, after the exposure of samples, covered with 10 μ m thick Al-coating, to MIEB

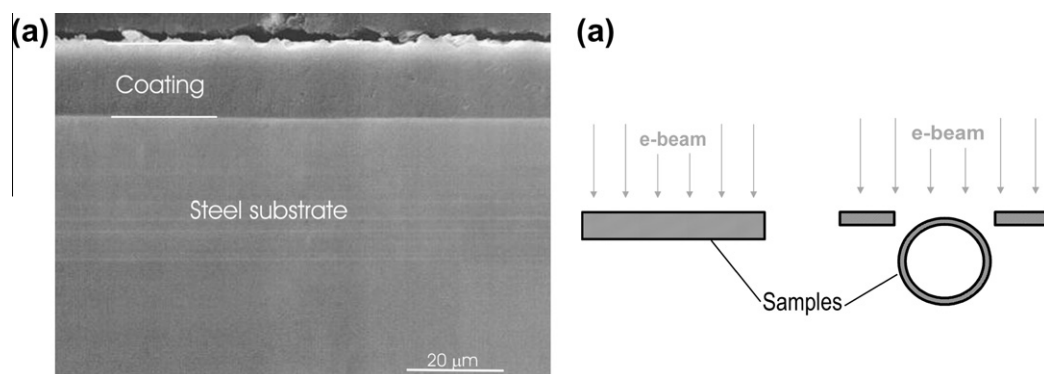


Fig. 1. (a) SEM cross section of a T91 tubular specimen with Al coating, 15 μ m thick, deposited by vacuum-arc method; (b) irradiation methodology scheme of flat and tubular specimens.

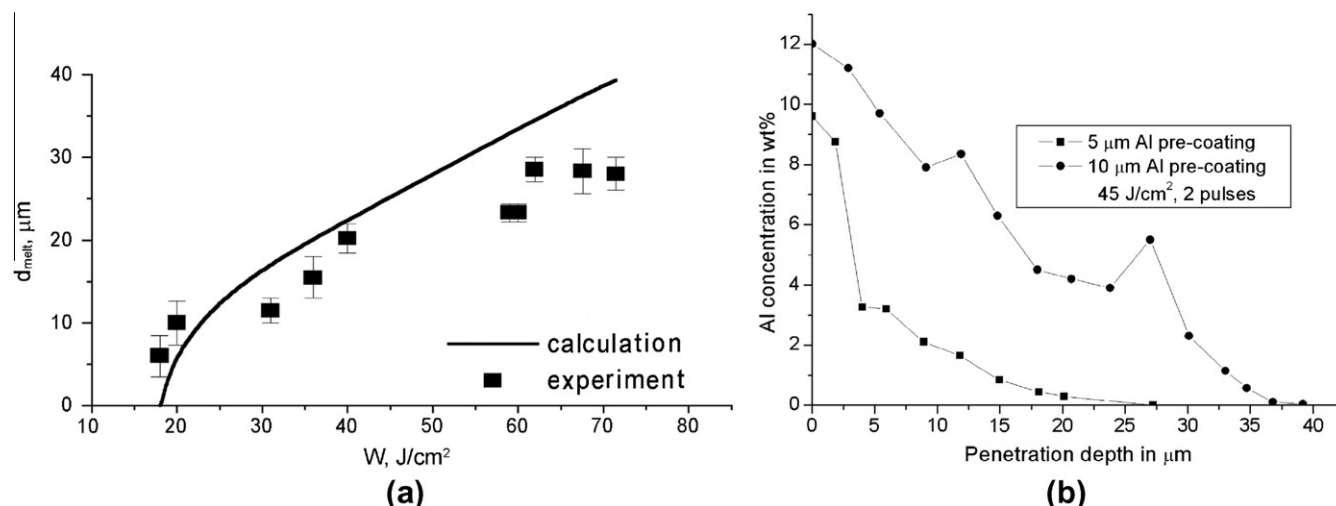


Fig. 2. (a) Measured and calculated melting depth in steel specimen covered with 10 μm Al coating; (b) Al concentration profile perpendicular to the surface, function of the Al coating thickness (energy input 45 J/cm^2 , two pulses).

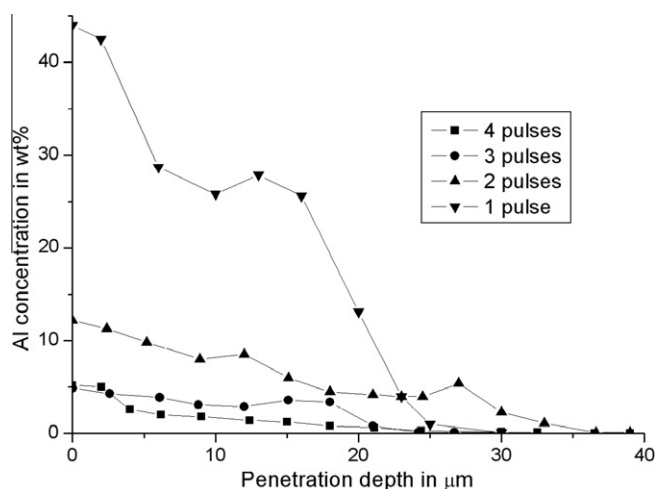


Fig. 3. Al concentration profile function of the number of applied pulses (10 μm of Al coating and energy density of the electron beam of 45 J/cm^2).

with 45 J/cm^2 and a number of pulses between one and four. After three pulses the profile of the Al concentration is almost constant up to 20 μm . One should note that (i) the concentration profiles are not typical for a diffusion process, but for the distribution by turbulent mixing in the melt [18] and (ii) the Al content decreases, by increasing the pulse number, mainly due to evaporation but also to redistribution deeper inside the substrate.

The amount of Al, which can be introduced in the superficial layer, is a function not only of the coating thickness (as can be observed in Fig. 2b), but also of the energy input. In previous works it was shown that the energy input in the range 30–50 J/cm^2 allows for up to 30% of the original Al amount to be alloyed [10].

In order to obtain a surface layer with an uniform Al distribution, of controlled and uniform thickness, which is crack-free and adherent to the substrate, the following procedural steps and parameter values were found as optimal:

- Preparation of the steel specimen surfaces (before Al-coating deposition) by:
 - electropolishing of the austenitic steels surface and mechanical polishing of the ferritic–martensitic steels;

- ultrasonic cleaning and ion beam etching of the steel surface;

- Al-coating deposition using vacuum arc process (coating thickness 5–10 μm);
- Thermal treatment of coated steel specimens (200 $^{\circ}\text{C}$, 2 h, argon);
- Application of the MIEB process: melting the Al-coated steel surfaces using intense pulsed electron beams (GES-1 facility: accelerating voltage 120 kV; energy density 40–45 J/cm^2 ; pulse duration 30 μs ; number of pulses: 2–3).

The general aspect of the alloyed surface layer (parameters: 10 μm , 45 J/cm^2 , 30 μs , 2 pulses) together with the Al concentration measured in different regions are shown in Fig. 4. The Al concentration in the alloyed layer varies from 15 to 20 wt.%, near the surface, and 5 to 10 wt.%, at the interior. The observed concentration oscillation along the thickness of the layer suggests again a turbulent mixing process.

3.2. Testing of the modified/alloyed surface steels

For nuclear applications, a homogeneous Al distribution of defined concentration has to be guaranteed for the entire modified surface, especially in the case of cladding tubes. The main goal of the experimental evaluation was to verify that the MIEB-Al procedure, applied to different steel grades envisaged for advanced nuclear systems, can provide protection in different HLMs.

Steel specimens, in original polished state and after Al surface alloying by electron beam melting, were exposed to liquid lead and LBE under different normal and abnormal conditions related to nuclear applications:

- oxygen concentration: 10^{−4}, 10^{−6} and 10^{−8} wt.%;
- exposure time: hundreds and up to more than 15,000 h;
- with and without mechanical load;
- temperature range: 450–650 $^{\circ}\text{C}$;
- stagnant and flowing conditions;
- different coatings: Al, Al–Fe, Al–Cr–Fe.

The tubular specimens were machined as shown in Fig. 5a, upper part. Fig. 5a lower part shows, as an example, the pictures of a specimen made out of T91 steel with no Al-coating, before and after exposure to LBE for 2500 h (550 $^{\circ}\text{C}$, 1–4 $\times 10^{-6}$ wt.%

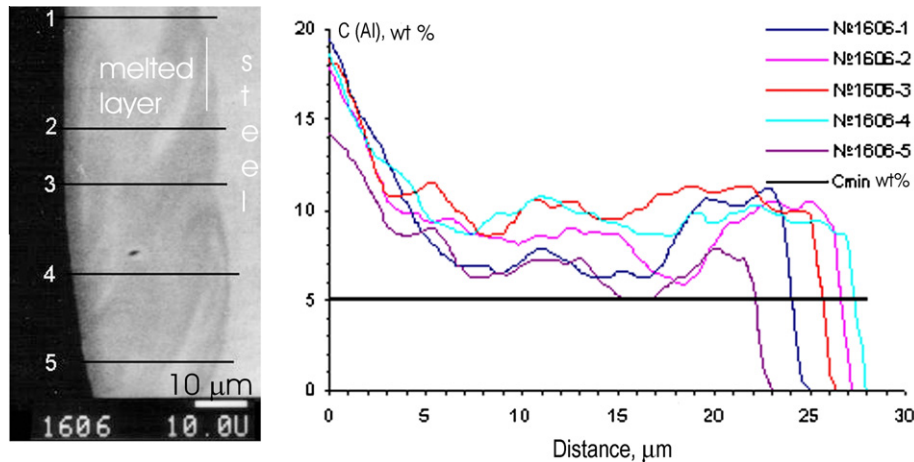


Fig. 4. SEM of the cross section of the 10 μm Al-coated T91 tube specimen and the profiles of Al concentration after GESA treatment (energy density of 45 J/cm²; two pulses).

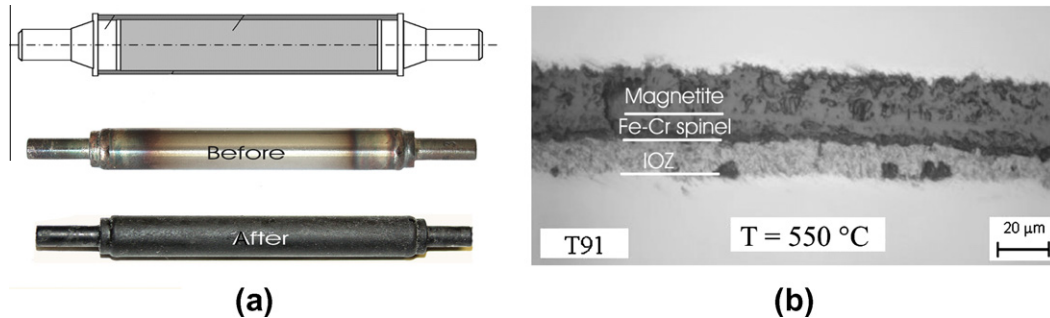


Fig. 5. (a) Scheme and photographs of tubular specimens made out of T91 steel without Al-coating before and after exposure to LBE for 2500 h (550 °C, $1\text{--}4 \times 10^{-6}$ wt.% oxygen content); (b) LM of cross section of T91 steel specimens after exposure to LBE (2500 h, 550 °C, $1\text{--}4 \times 10^{-6}$ wt.% oxygen content, static conditions).

oxygen content). After the corrosion test, a dark oxide film appears over the whole sample surface. The experiments performed at three different temperatures showed that the oxidation is intensive even at low temperature. After 2500 h of exposure to LBE the thickness of the oxide scale varies from approximately 15 μm , for exposure at 476 °C, to 20 μm , at 490 °C and 25 μm at 550 °C. As known from literature the oxide scale has two sub-layers which consist of magnetite on top followed by the Fe–Cr-spinel oxide [5,8]. An internal oxidation zone (IOZ) exists after exposure at 490 °C and 550 °C. The LM picture of the cross section of the T91 steel specimen, after exposure at 550 °C (2500 h, 10^{-6} wt.% oxygen), is shown in Fig. 5b.

Up to now, the maximum exposure time to LBE at 550 °C was 16,547 h. After this time interval the original non-alloyed T91 specimens have shown a duplex oxide scale (Fe–Cr spinel with magnetite on top) with a thickness ranging from 30 to 300 μm . The outer layer, consisting of porous magnetite, broke off frequently. Local dissolution attacks were observed on a few areas, where also the Fe–Cr-spinel oxide sub-layer broke off.

Fig. 6 depicts tubes, made out of T91 and 1.4970 steels, with surface alloyed with Al by the MIEB process, showing a shiny metallic surface with only a few “dark” places after exposure at 550 °C to LBE ($1\text{--}4 \times 10^{-6}$ wt.% oxygen content). In the dark regions, where the modification was not fully successful, the Al concentration is too low (less than 3 wt.%), but the steel specimens are protected against dissolution attack by a dense Al-containing Fe–Cr spinel oxide scale, which shows a lower growing rate compared with non-modified specimens [19].

The cross section through the shiny area of the surface-alloyed T91 specimens after 16547 h exposure to 550 °C shows a com-

pletely different aspect (Fig. 7a): a very thin oxide scale (<1 μm) on top of the surface. It is less visible on the SEM pictures, but it can be well recognized in the concentration profile perpendicular to the surface of the main elements (Fig. 7b). Since the solubility of pure aluminum is much higher than that of iron and chromium in LBE, the Al peak indicates an alumina layer like it was analyzed in [13,20]. Surface-alloyed 316 stainless steel showed the same corrosion resistance after 5000 h exposure to LBE (600 °C, 10^{-6} wt.%) due to the formation of the same assumed alumina layer (Fig. 7c). This layer protects the steel not only from LBE attack but also from oxygen diffusion into the coating and bulk material. The diffusion of Fe through the surface, to form a magnetite layer on top, is also prevented by the thin alumina scale. In order to form alumina as a protective scale, the minimum Al concentration in the modified layer should be significantly higher than 3 wt.%. The upper limit of Al content should be approximately 10 wt.% to avoid cracks in the modified layer during GESA treatment.

Generally, the corrosion tests have confirmed that the MIEB-Al procedure leads to an improved corrosion resistance of the steels exposed to HLMS, by changing the type of the oxide formed on the surface. However, the occurrence of the “dark” area, covered by a spinel-type oxide, requires further optimization of the procedure.

Currently, three directions for the optimization and industrialization of the MIEB-Al procedure are under evaluation: (i) multi-stage modification, (ii) usage of composite coatings and (iii) irradiation of tubular samples by radially converging electron beams.

In the multi-stage modification the MIEB-Al procedure is repeated several times. Experiments with two-stage modification showed a significant improvement of the modified layer homoge-

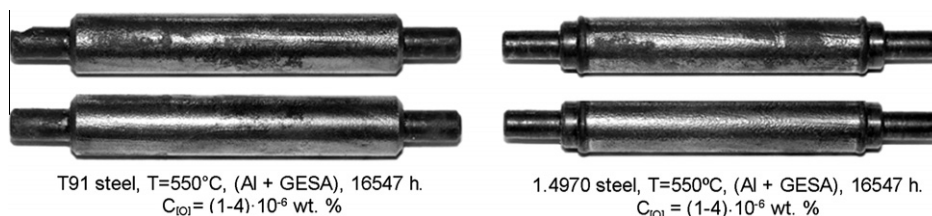


Fig. 6. Photographs of tubes made out of T91 and 1.4970 steels with surface alloyed with Al by GESA-process showing a shiny metallic surface with only few “dark” places after exposure at 550 °C to LBE ($1\text{--}4 \times 10^{-6}$ wt.% oxygen content) for 16,547 h in static conditions.

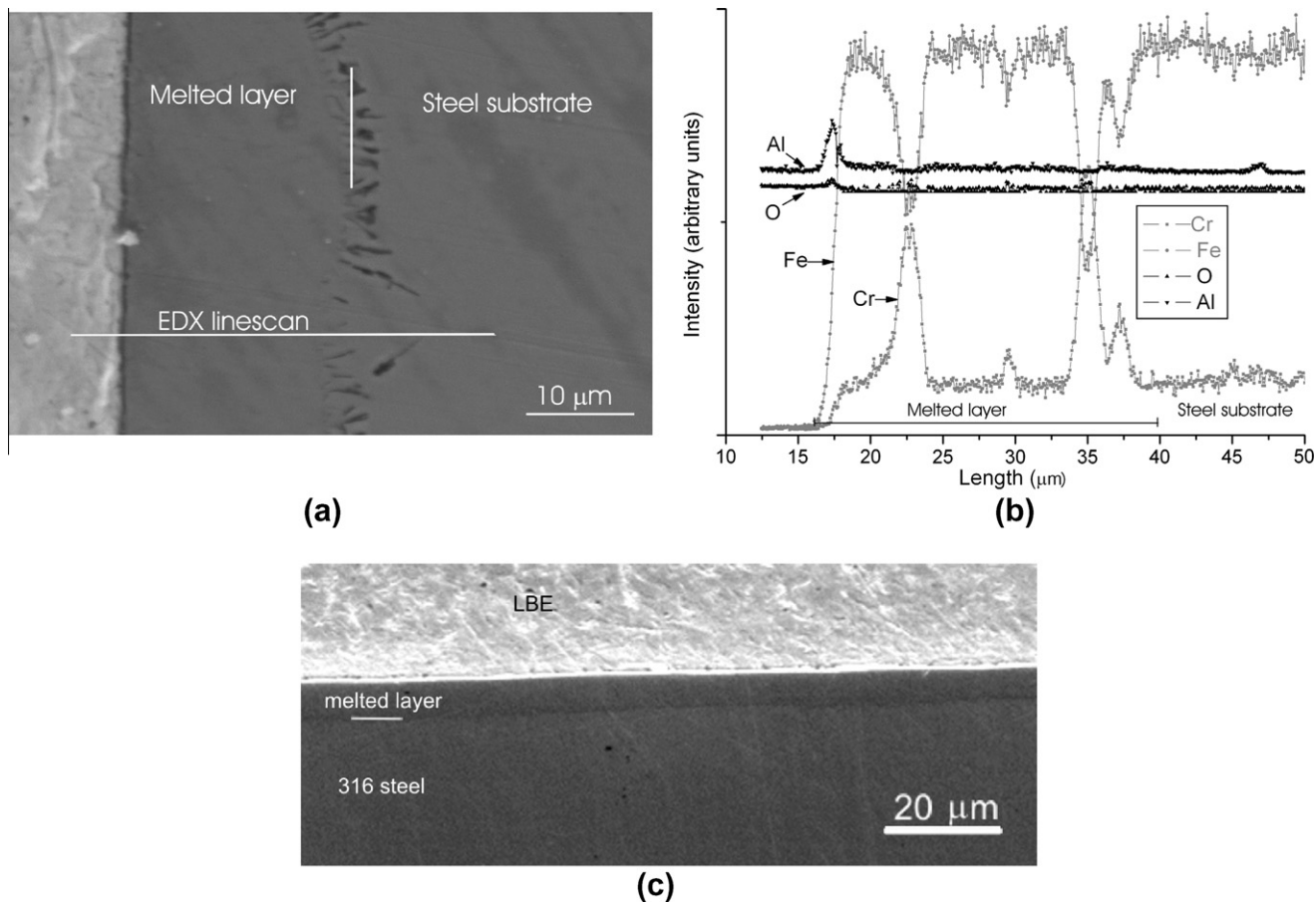


Fig. 7. (a) SEM of the cross section of the Al-coated and GESA-treated T91 tube specimen after 16,547 h exposure to LBE ($1\text{--}4 \times 10^{-6}$ wt.% oxygen) at 550 °C and (b) concentration profiles of the main elements, perpendicular to the surface, indicating the formation of a very thin Al-oxide scale on the specimen surface; (c) SEM of the cross section of Al surface-alloyed 316 stainless steel sample, after 5000 h exposure to LBE (10^{-6} wt.% oxygen, 1–1.2 m/s, 600 °C).

neity. It should be also noted that mechanical tests, performed under creep conditions with thermal cycling, showed that the two-stage modification of the T91 steel promotes an appreciable increase of the creep resistance (Fig. 8). During creep tests, 10 thermal cycles (cooling from 550 °C up to 300 °C with subsequent heating up to 550 °C) have been carried out. Duration of each cycle was about 30 s.

As already mentioned, the protective effect of the GESA modification of steels in oxygen-containing HLM is based on the formation of a thin alumina film on top of the modified layer. The addition of Cr in binary Fe–Al alloys has been shown to reduce the level of the Al content needed to form a protective Al_2O_3 layer at high temperature (1000 °C roughly) (third element effect) [18,19]. This behavior could be extrapolated to lower temperatures (550–600 °C). Therefore, in particular, the quality of the protective

layer on the austenitic steel containing a high amount of chromium is better than the quality of the protective layer on the F/M steels. Based on experimental data obtained during oxidation experiments performed at high temperature [21–23], the $\text{Fe}_{100-x-y}\text{Cr}_x\text{Al}_y$ alloy system ($x = 10\text{--}20$, $y = 6\text{--}20$) was chosen for coating the steel surfaces. After the deposition of alloys, the GESA process was used to melt and bond the coating on the steel surface.

Corrosion tests of steel samples, coated with FeCrAl-alloys of different compositions, were performed in HLM in a wide range of parameters values. As an example, Fig. 9 shows two specimens made out of the T91 steel, initially coated with $\text{Fe}_{78}\text{Cr}_{14}\text{Al}_8$ alloy (thickness: 10 μm) and subsequently MIEB-treated (energy density 45 J/cm², pulse duration 30 μs; number of pulses 2), which were exposed to LBE melt (time 5000 h, temperature 600 °C, oxygen content 10^{-6} wt.%, flowing speed 1.2 m/s). As one can see there is

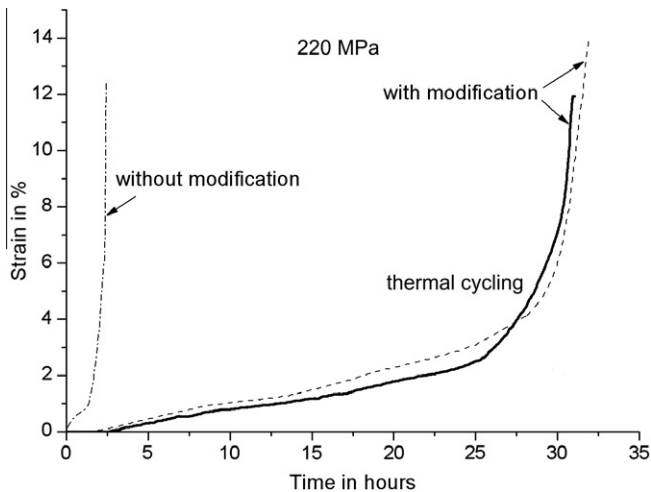


Fig. 8. Comparison of the creep-to rupture experiments (220 MPa) of original and two-stage modified T91 steel in Pb–Bi flow (2 m/s, 10^{-6} wt.% oxygen) at 550 °C with and without thermal cycling.

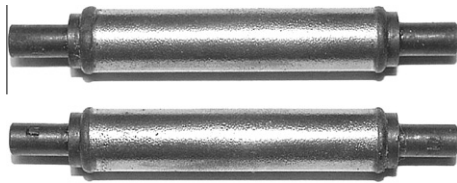


Fig. 9. Surface coated with $\text{Fe}_{78}\text{Cr}_{14}\text{Al}_8$ alloy and subsequently GESA treated test tubes made out of T91 steel showing a shiny aspect after 5000 h exposure to LBE (10^{-6} wt.% oxygen) flowing with 1–1.2 m/s at 600 °C.

no dissolution attack and no dark area on the whole surface of the samples. The investigation of the cross sections to evaluate the nature and the thickness of the protective layer is underway, but one can conclude that the application of the Fe–Cr–Al coating is rather promising.

As already mentioned in Section 3.1, the irradiation of the cylindrical samples was done with MIEB through a diaphragm with a sample rotation after each pulse. It is obvious that this procedure is not optimal since the chemical composition and, implicitly, the properties of the modified layer in overlapping zones can differ from that in the main part of the layer. In order to solve this issue, it would be better to irradiate the samples by radially converging pulsed electron beams. A new facility called GESA IV, able to generate radially converging pulsed electron beams, was designed and manufactured [24]. Currently, the irradiation procedure, using this facility, is under development.

4. Conclusions

The steel surface alloying with aluminum (Al) using Microsecond-pulsed Intense Electron Beams (MIEB–Al) was developed and optimized in order to be used for improving the corrosion resistance of the 316, 1.4970 and T91 steels, exposed to liquid Pb and Pb–Bi-eutectic. The procedure consists in two steps: (i) coating the steel surface with Al or an Al-containing alloy layer and (ii)

melting the coating layer together with the steel surface layer using intense pulsed electron beam. In order to cover the steel surface with an homogeneous and crack-free Al-alloyed layer, the following experimental conditions are required: Al coating thickness range 5–10 μm , electron kinetic energy 120 keV; pulse duration 30 μs ; energy density 40–45 J/cm^2 ; number of pulses 2–3.

Using the mentioned procedure, the corrosion resistance of the 316, T91 and 1.4970 steels, exposed to Pb and Pb–Bi-eutectic with different oxygen concentrations and under different temperatures, was considerably improved due to the formation of a thin alumina layer (which thickness is lower than 1 μm for all the tested temperatures and durations) acting as an anti-corrosion barrier.

Further development of the MIEB–Al technology shall be directed to developing the procedure of coating deposition on full-length fuel claddings and the procedure of irradiation by radially converging MIEB.

Acknowledgements

The authors would like to thank the ISTC and the EC for funding the work presented in this paper under Project No. 2048 and 'EUROTRANS' Project No. FI6W-CT-2004-516520, respectively.

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