

INFLUENCE OF ALUMINUM DIFFUSION LAYER ON T/P92 STEAM OXIDATION RESISTANCE - A LABORATORY AND FIELD STUDY

J. Allenou,

Vallourec Research Center France, Aulnoye-Aymeries, France

J. Pirón-Abellán,

Vallourec Research Center Germany, Düsseldorf, Germany

E. Hugon,

Vallourec Research Center France, Aulnoye-Aymeries, France

C. Landier,

Vallourec Tubes France, Steam Tubes & Pipes, Boulogne-Billancourt, France

Th. Le Guevel,

Electricité De France, Thermal Engineering Department, Paris, France

ABSTRACT

The steam oxidation behaviour of boiler tubes and steam piping components is a limiting factor for improving the efficiency of the current power plants. Spallation of the oxide scales formed during service can cause a serious damage to the turbine blades. Vallourec has implemented an innovative solution based on an aluminum diffusion coating applied on the inner surface of the T/P92 steel. The functionality of this coating is to protect the tubular components against spallation and increase the actual operating temperature of the metallic components. In the present study, the newly developed VALIORTM T/P92 product was tested at the EDF La Maxe power plant (France) under 167b and 545°C (steam temperature). After 3500h operation, the tubes were removed and characterized by Light Optical Metallography (LOM), Scanning Electron Microscopy (SEM), with Energy Dispersive X-ray spectrometry (EDX) and X-Ray Diffraction (XRD). The results highlight the excellent oxidation resistance of VALIORTM T/P92 product by the formation of a protective aluminum oxide scale. In addition, no enhanced oxidation was observed on the areas close to the welds. These results are compared with the results obtained from laboratory steam oxidation testing performed on a 9%Cr T/P92 steel with and without VALIORTM coating exposed in Ar-50%H₂O at 650°C.

Keywords: Aluminum diffusion coating, ferritic-martensitic steels, steam oxidation, power plant

INTRODUCTION

An increase in efficiency and a reduction in CO₂ emissions in the last generations of Ultra Super Critical (USC) power plants can be achieved by increasing the temperatures and steam pressures. Ferritic-martensitic steels with 9-12% chromium exhibit great interest as material for superheaters (SR) and steam piping due to their high creep resistance and low coefficient of thermal expansion. Nevertheless, oxidation performance of these types of steel needs to be improved in such operating conditions. The oxide layer developed has a high tendency to spall, leading to erosion of valves and turbine blades and/or blockage of heat exchanger tubes [1]. Aluminum diffusion coatings have already proven in several studies to be an effective alternative to prevent or reduce oxidation in steam atmospheres at temperatures up to 650°C [2-10]. However, most of the available works are launched in the laboratory under atmospheric pressure and isothermal conditions [2-9] and not so many are real field tests [10].

Vallourec has developed at industrial scale a new solution mainly dedicated for steam piping application: the VALIOR™ P92 pipes consisting in an aluminum diffusion layer along the inner surface of the P92 pipe. In order to make the test possible in the La Maxe power plant, 1m long VALIOR™ T92 tube was manufactured in our research center according to our standard industrial procedure.

The scope of this study is to characterize the oxidation performance of VALIOR™ T/P92 steel under real power plant operating conditions and compare with laboratory steam oxidation testing. Two T92 tubes with and without inner coating were tested at the EDF La Maxe power plant (France) under 167b at 545°C (steam temperature). These tubes were placed in the last superheater section of the power plant. The microstructure evolution of the Al diffusion layer and the oxide scales formed after exposure in power plant are compared with the results of laboratory steam oxidation testing performed on 9%Cr P92 steel under Ar-50%H₂O at 650°C. The welded parts of the VALIOR™ T92 steel after exposure in power plant are also investigated.

EXPERIMENTAL DETAILS

The ferritic-martensitic steel used in this investigation is X10CrWMoVNb9-2, according to EN10216-2, known also as T92 or P92 according respectively to ASTM A213 and to ASTM A335. The chemical compositions of the materials used in the present study are listed in Table 1.

Table 1. Composition of the T/P92 investigated steels, wt.%

	C	Mn	P	S	Si	Ni	Cr	Mo	V	B	Nb	N	Al	W	Ti	Zr
P92	0.11	0.42	0.009	<0.001	0.23	0.16	8,77	0.48	0.18	0.002	0.044	0.049	0.010	1,58	-	-
T92	0.13	0.51	0.013	<0.001	0.16	0.15	8,65	0.44	0,19	0.001	0.048	0.050	0.008	1,71	-	-

For laboratory testing, specimens of dimensions 20x10x2 mm were machined from a P92 pipe section in 703 mm Outside Diameter (OD) x 85 mm Wall Thickness (WT). The VALIOR™ P92 specimens were produced by manual spraying of aluminum slurry followed by a diffusion heat treatment. Prior to the oxidation tests, the non-coated specimens were grounded by 80 grit SiC paper and degreased in ethanol.

The dimensions of the T92 tubes tested in the power plant were OD 48.3 mm x WT 7.65 mm (length: 1000 mm). The non-coated T92 tube was produced following the typical Vallourec production routes. The VALIOR™ T92 tube prototypes were produced according to the industrial routes and followed by the deposition of the aluminum slurry on the inner surface of

the tube. Finally, the slurry-coated T92 tubes were subjected to an industrial diffusion heat treatment to obtain the final VALIOR™ product.

The T92 and VALIOR™ T92 tubes were welded to TP321H (Fe-17.5Cr-10.5Ni) stainless steel in the last superheater section of the power plant. A picture of the welded tubes after 3500 h power plant operation can be seen on Figure 1.

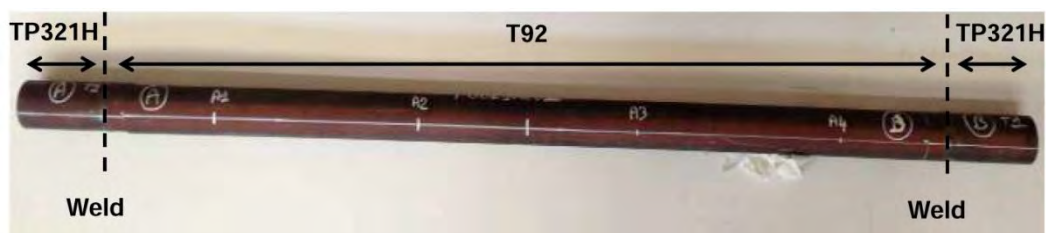


Figure 1. T92 tube welded to TP321H stainless steel tubes after power plant operation.

The aluminum concentration on the coating surface of the specimens prepared for laboratory conditions prior exposure was 15 wt% (low Al-concentration) whereas the aluminum surface concentration on the coating was 30 wt% (high Al-concentration) for the tubes tested in La Maxe power plant.

The oxidation conditions for laboratory and field conditions are summarized in Table 2. The steam conditions in the laboratory are similar to those described in [7]. The specimens were exposed to the model gas Ar-50%H₂O at 650 °C using a horizontal furnace equipped with a silica protective tube. The oxidation experiments were performed for 3000 h. The mass gains of the specimens were measured after periodically interruption of the tests every 250 h, i.e. after specimen cooling to room temperature.

VALIOR™ coated and standard non-coated T92 tubes were placed in the last superheater section of the La Maxe power plant for 3500 h. The number of start-ups from temperatures < 200°C represented 30% of the total cycles, whereas the number of start-ups from a temperature > 200°C represented 70% of the total cycles.

Table 2. Comparison of the steam oxidation conditions under laboratory and real field testing (La Maxe power plant)

Conditions	Laboratory	La Maxe
Steam temperature (°C)	650	545
Metal temperature (°C)	650	570
Time of exposure / operating (h)	3000	3500
Cycle frequency (Number of cycles / 1000 h)	4	10
Pressure (b)	1	167

The specimens after exposure were cross sectioned and prepared following typical metallographic techniques. Prior mounting in an epoxy resin, the specimens were sputtered with a thin gold layer and subsequently electrochemically coated with a nickel layer. The specimens were characterized by Light Optical Microscopy (LOM), scanning electron microscopy (SEM) combined with energy dispersive X-ray (EDX) analysis and X-ray diffraction (XRD).

RESULTS

Steam oxidation under laboratory conditions

Figure 2 shows the mass gain of the two studied P92 samples, a standard non-coated and a VALIOR™ P92 steels during exposure in Ar-50%H₂O at 650°C. After 3000 h exposure the non-coated P92 specimen clearly shows higher mass gain values than those observed for the VALIOR™ P92 testing coupon. The initial mass loss observed for the VALIOR™ specimen can be attributed to the loss of undiffused Al slurry [2].

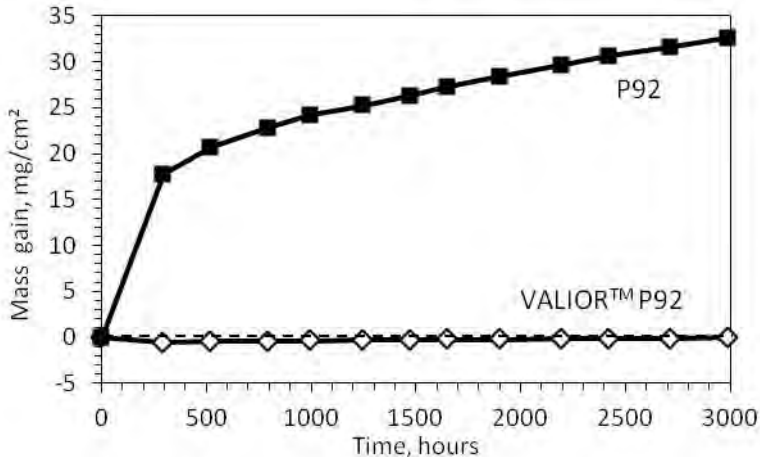


Figure 2. Mass gain of standard P92 and VALIOR™ P92 samples during exposure in Ar-50%H₂O at 650°C.

Figure 3 shows micrographs of a VALIOR™ P92 sample before exposure (Fig. 3a) and after steam exposure (Fig. 3b) as well as a micrograph of the standard non-coated P92 sample after 3000 hours of steam exposure (Fig. 3c) under laboratory conditions.

The two layers of the coating, i.e. top coat (outer layer) and interdiffusion zone (inner layer) before exposure, as shown in Figure 3a, have been described in detail elsewhere [7]. Porosity is mainly located at the interface between the outer layer and the interdiffusion zone and was reported to be linked to Kirkendall effect [2]. In the outer part of the top coat layer another zone characterized by the presence of porosity was found. After 3000h exposure, we did not observe any modification of the diffusion front and only a thin oxide layer was observed on the top of VALIOR™ P92 sample (Figure 3b). In contrast, a very thick iron-rich oxide scale was observed on the standard non-coated P92 sample. Typical oxide morphology is observed [11]: beneath the original metal surface, an inner layer is formed, consisting of Fe₃O₄ and Fe,Cr-rich spinel. Above the original metal surface, Fe₃O₄ and Fe₂O₃ layers were present. Close to the steel, an internal oxidation zone is observed.

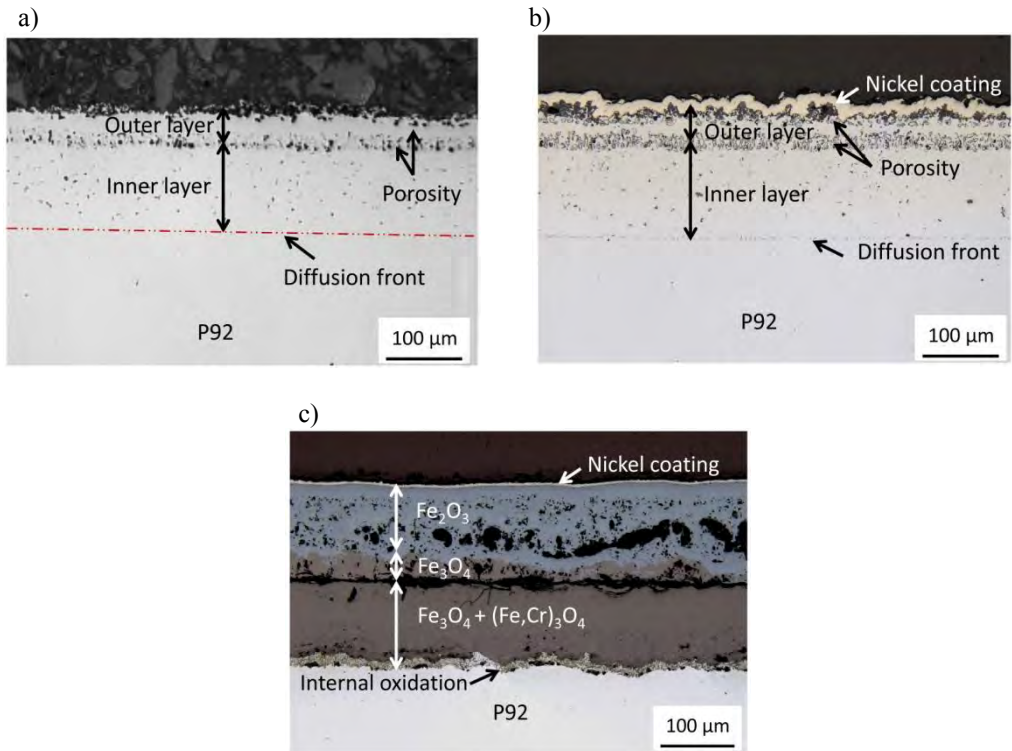


Figure 3. Metallographic cross sections of a) VALIOR™ P92 sample before exposure b) after 3000 hours exposure and c) non-coated P92 sample in Ar-50% H_2O at 650°C.

Figure 4 shows the EDX mapping of the elements in the oxide scale formed on the VALIOR™ P92 sample after 3000 h exposure at 650 °C. The external oxide scale is characterized by the presence of a rather thin aluminum oxide scale. However, iron enrichment is locally identified in the outer part of the scale as presented in Figure 4. Beneath the external oxide scale an area characterized by the presence of porosity was observed. EDX analyses show that the inner surfaces of these pores are covered by a very thin aluminum oxide scale.

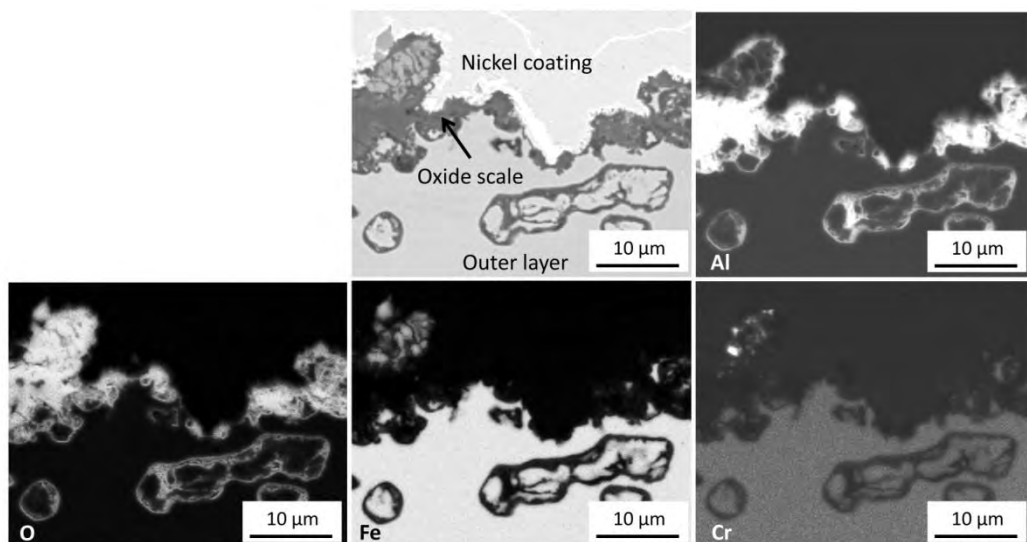


Figure 4. SEM/EDX mapping of the external scale formed on top of VALIOR™ P92 sample after 3000 hours exposure in Ar-50% H_2O at 650°C.

XRD analyses performed on the surface of the VALIOR™ P92 sample confirmed the presence of α - Al_2O_3 and Fe_2O_3 (Figure 5). In addition FeAl and Fe_3Al intermetallic phases were also identified. The external formation of a thin α - Al_2O_3 layer is well reported in many studies to be protective under steam exposure [2-10].

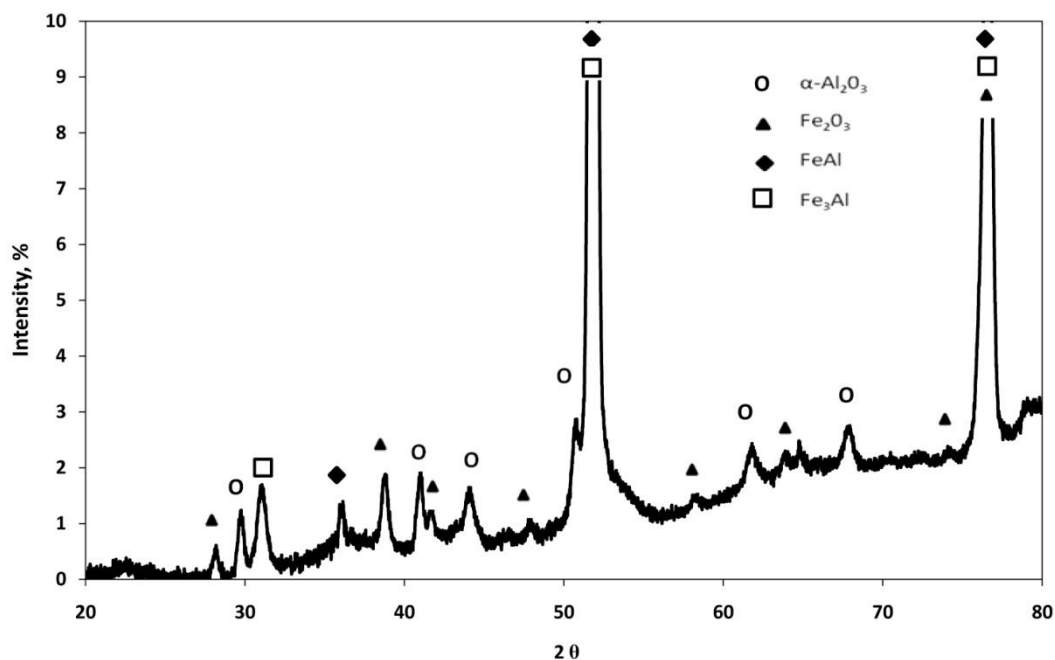


Figure 5. Surface XRD analysis performed on VALIOR™ P92 sample after 3000 hours exposure in Ar-50% H_2O at 650°C.

Steam oxidation in La Maxe power plant

Micrographs of standard non-coated T92 and VALIOR™ T92 tubes after exposure in La Maxe power plant are illustrated in Figure 6. As for laboratory testing, the non-coated T92 tube shows the formation of thick iron-rich oxide scales. The iron-rich oxides formed are similar to those described in e.g. [12]. However the thicknesses of iron scales formed under power plant conditions tends to be lower than those observed after laboratory testing. In the case of the VALIOR™ T92 tube exposed in power plant, the Al diffusion layer remains full without any damage. The only indications are very thin and small cracks occurring not beyond the diffusion front. They are observed in the outer layer and sometimes in the inner layer (Figure 66). Their formation could be related to stress relaxations after diffusion heat treatment or be linked to thermal cycling occurring in the power plant. The presence of small cracks in field conditions into an aluminide coating was also mentioned in other studies [see e.g. 10]. We can observe the Kirkendall porosity located between the outer and interdiffusion layers.

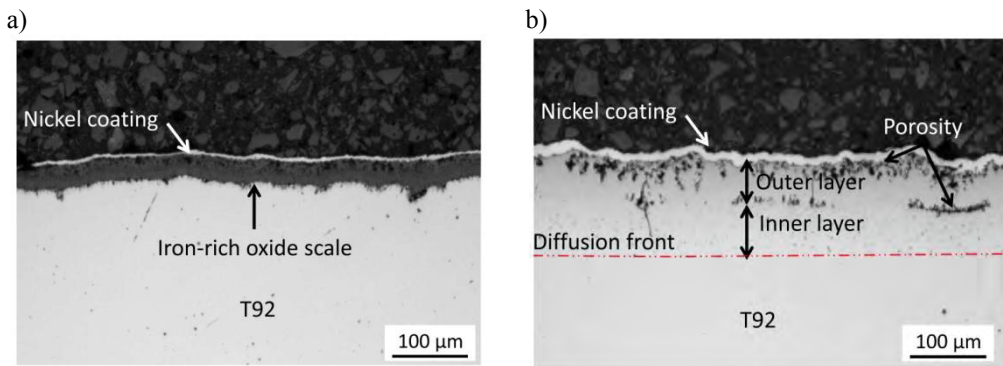


Figure 6. Metallographic cross sections of a) uncoated T92 tube and b) VALIOR™ T92 tube after 3500 hours operation in power plant at 570°C.

Figure 7 shows the EDX mapping of the elements at the surface performed on the top of the aluminum diffusion coating after operation in the power plant. The external oxide scale seems to be enriched in iron, aluminum and in minor amounts of chromium. The porosity located close to the surface shows also the formation of aluminum rich oxides on the inner surface of the pores.

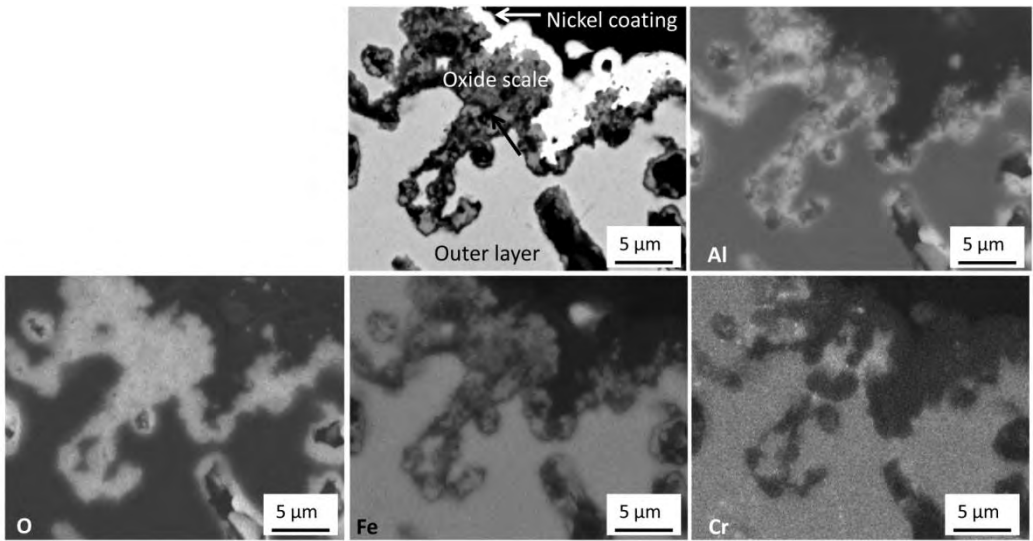


Figure 7. SEM/EDX mapping of the elements at the surface of the Al diffusion coating after 3500 hours exposure in power plant at 570°C.

XRD analyses performed on the surface of the VALIOR™ T92 tube confirmed also the presence of α - Al_2O_3 and Fe_2O_3 (Figure 8). The FeAl and Fe_3Al intermetallic phases are also detected. The presence of protective layer of α - Al_2O_3 was also identified under field conditions at 640°C at 180b [10].

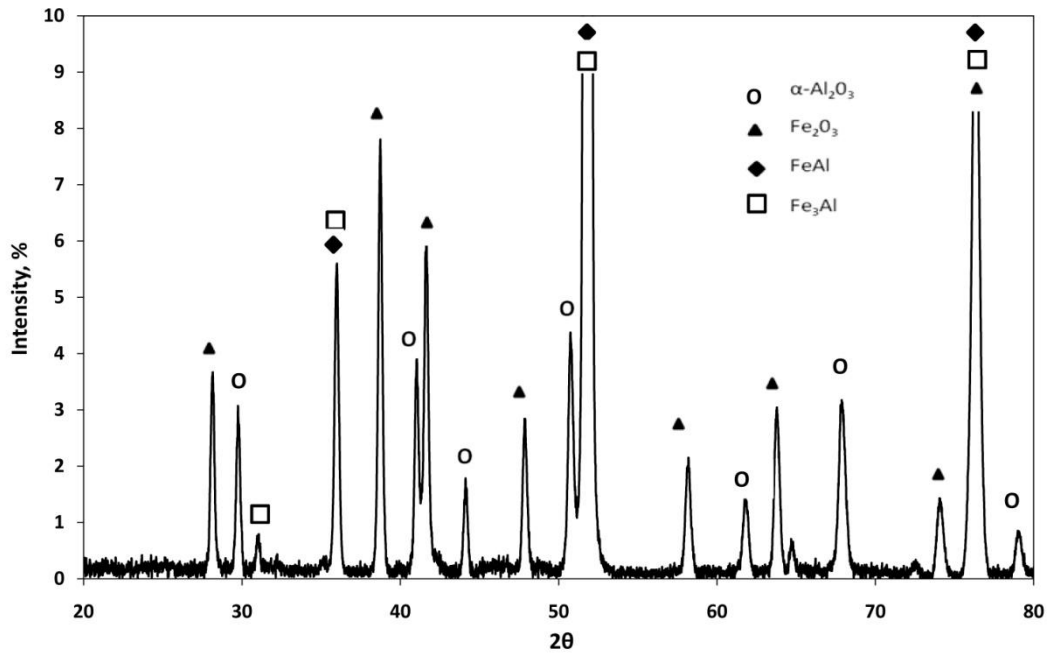


Figure 8. Surface XRD analysis performed on a VALIOR™ T92 tube after 3500 hours exposure in power plant.

The variation in the aluminum profiles of the VALIOR™ T92 tube before and after 3500 h exposure in power plant is illustrated in Figure 9. The aluminum content is similar before and after 3500 h exposure, whereas approximately 20 μm inward aluminum diffusion was observed.

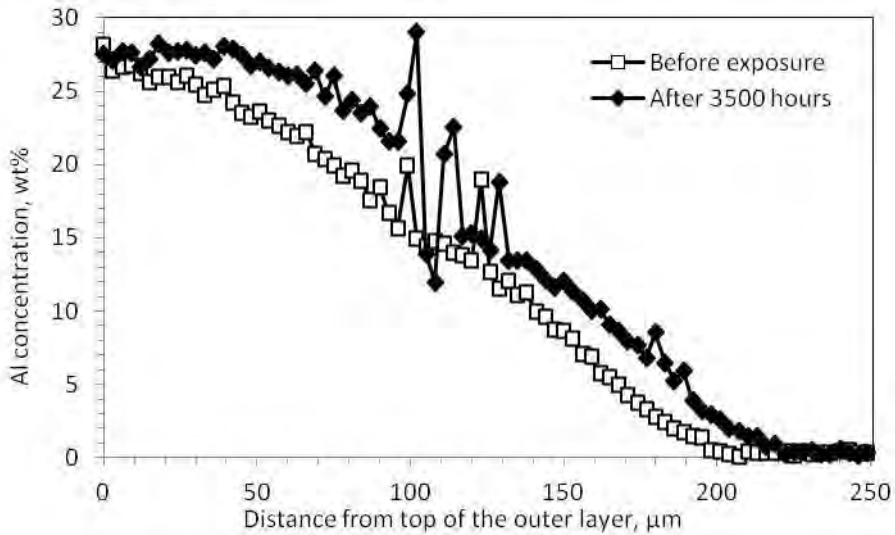


Figure 9. Aluminum concentration profile measured by EDX on a VALIOR™ T92 tube before and after 3500 hours operating in power plant.

Analysis of welds after exposure in La Maxe power plant

VALIOR™ T92 tube was installed in La Maxe power plant in the superheater section and welded to a TP321H (Fe-17.5Cr-10.5Ni) stainless steel tube. The welding procedure for VALIOR™ T92 tubes is typical for 9-12% Cr steels. Prior to the welding process, a chamfer was machined at the ends on the VALIOR™ T92 tube. During this process, the aluminum diffusion coating was completely removed in the chamfer area. The welded parts close to the weld bead and close to the Al diffusion coating were characterized after 3500 h exposure (Figure 10).

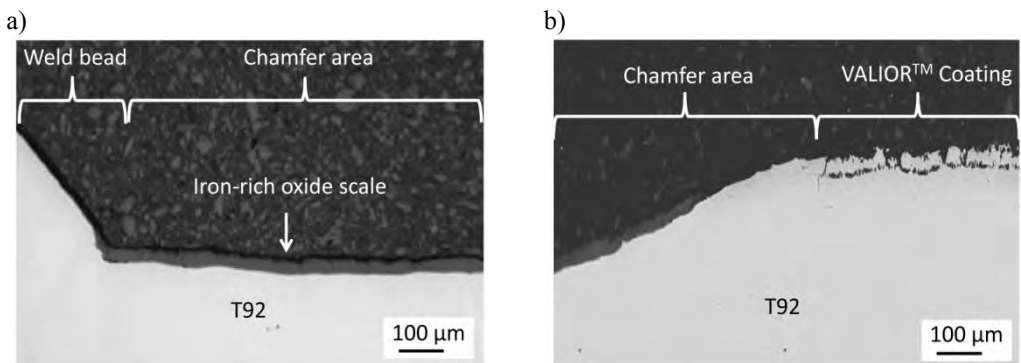


Figure 10. Optical micrographs of the welding parts of the VALIOR™ T92 tube after 3500 hours exposure in power plant: a) area close to the weld bead and b) zone close to the VALIOR™ coating.

No indication of enhanced fast growing oxide scales was observed in the chamfer area. The thickness of the iron oxide formed close to the weld bead (Fig. 10 a) and close to the Al diffusion coating (Fig. 10b) are very similar to those formed on the non-coated T92 tube. The aluminum diffusion coating which was partially removed close to the chamfer zone does not show any indication of accelerated oxidation.

DISCUSSION

Steam oxidation under laboratory conditions at 650°C

Many studies have pointed out that the morphology of the scales formed in steam atmospheres may differ depending on the exposure laboratory parameters, such as surface condition, flow rate and pressure [13]. However, large differences in the scale growth rates can also be found for the same material class. This observation is based on the so called “batch-to-batch” effect [11]. Figure 1 illustrates the mass gain values obtained when exposing the non-coated and VALIOR™ P92 steels for 3000 h in Ar-50%H₂O at 650°C. The mass gain is significantly higher in case of the non-coated specimen. Typical 9% Cr ferritic-martensitic steels tend to exhibit high scale growth rates when exposed to steam containing atmospheres due to the formation of non-protective multi-layered iron rich oxide scales. For these specimens the oxide scale formed consists of an inner layer which is formed beneath the original metal surface, consisting of Fe₃O₄ and Fe,Cr-rich spinel. Above the original metal surface Fe₃O₄ and Fe₂O₃ layers were present (Fig. 2). The kinetics and mechanism of scale growth under exposure in water vapor containing gases have been presented in a large number of publications [1;11] and therefore it falls outside the description of this investigation. Contrary to the non-coated specimens, the VALIOR™ specimen (low Al-concentration coating, $\alpha_1 = 15$ wt%) has formed a very thin oxide scale after 3000 h exposure. The scales consist mainly in α -Al₂O₃ (Fig. 5) but minor amounts of Fe and Cr were locally found on the scale (Fig. 4). Many authors have found that the oxide scales present in this type of coatings consist in Al₂O₃ and after very long exposure times and subsequent rapid oxidation rates, i.e. breakaway oxidation, iron-rich oxides and/or Fe,Al-mixed oxides are formed. A possible reason for the presence of Fe-rich oxides on top of the oxide scales can be related to the large roughness of the coating surface. The VALIOR™ specimen was exposed in as-coated surface finish. The surface of the coating which exhibits areas with larger surface-to-volume ratios than when mechanical surface finishing is applied, and subsequently faster aluminum consumption takes locally place resulting in the formation of Fe-rich oxides.

Steam oxidation in La Maxe power plant at 570°C

Typical high creep strength 9% Cr steels tend to exhibit high oxidation rates in water vapor containing atmospheres (Fig. 3c and Fig. 6a). During power plant operation, the presence of thick scales lead to an increase of the metal surface temperature of 30-40°C [14-15] and as consequence, a reduction of the heat transfer across the wall thickness of the component. The oxide scale compositions formed on the tubes evaluated in the power plant are similar to those typically found during laboratory testing, however several authors have found that the scales formed in steam atmospheres at high pressures can substantially differ in morphology and composition compared to those formed in laboratory testing [10, 12]. This difference is evident when comparing the scales formed under laboratory conditions (Fig. 3c) with those formed under power plant exposure (Fig. 6a): the scales formed at high steam pressure exhibit substantially lower amounts of in-scale voids/gaps formation compared to those formed at lower (atmospheric) pressure. Undoubtedly, the exposure at high pressure must substantially affect the Fe-oxide defect

structure and thus, reducing the extension of in-scale voidage. However, the oxidation rate of the non-coated T92 specimens significantly differ from those with a well defined surface finish. The standard test laboratory specimens were prepared by using a defined surface treatment (e.g. 80 SiC), whereas the inner surface of the non-coated T92 tubes exposed in the power plant has a larger roughness which is typical of the as-manufactured condition. These surfaces are characterized by the presence of an internal oxidation zone, consisting mainly of Cr-rich oxides. The penetration of this internal oxidation zone into the substrate can reach several tenths of micrometers (between 20-50 μm). Therefore, scale growth rates and the development of voids/gaps typically found in defined surface finished coupons might substantially differ from those where the initial internal oxidation zone is present. However, upon prolonged exposure times, the scales formed in power plant exposure will tend to show larger extend of void/gap formation similar to those found under laboratory conditions. An interesting observation is that in contrast to the laboratory conditions, hematite is locally formed within the scale under real power plant exposure and more precisely, related to cracks formed in the outer magnetite layer (Fig. 3c and Fig. 6a).

The presence of the diffusion aluminide coating and the formation of very thin scales on top of the coating (Fig. 6b) do not seem to significantly affect the heat transfer, avoiding overheating of the metallic component [16] and providing a most homogeneous temperature distribution throughout the wall thickness of the tube. Additionally, the application of slurry coatings to high creep strength materials, such as T/P92 steel, has demonstrated to be an effective method to significantly reduce the oxidation rates in water vapor containing atmospheres at temperatures up to 650 $^{\circ}\text{C}$ [10]. Under power plant exposure at 570 $^{\circ}\text{C}$, VALIORTM coating system also exhibit an excellent oxidation resistance compared to the 9%Cr T92 steel after 3500 h (Fig. 6a). The low scale growth rates observed in both cases are achieved by the formation of an external thin oxide layer (Fig. 4 and Fig. 7). XRD analyses performed to the surfaces show that the oxide scales consists mainly of Fe_2O_3 and $\alpha\text{-Al}_2\text{O}_3$ (Fig. 8). As found after laboratory exposure, Fe_2O_3 was found to be locally formed on the oxide scale (Fig. 7).

In the case of the coating tested under real power plant operating conditions ($C_{\text{Al}}^0 = 30 \text{ wt}\%$), the decrease in the aluminum content at the coating surface is negligible after exposure for 3500 h at 570 $^{\circ}\text{C}$ and a marginal inward diffusion of aluminum into the T92 substrate was observed (Fig. 9). No inward diffusion of aluminum was observed on samples at laboratory conditions. This slight difference between laboratory sample and La Maxe one might be related to morphological differences of the top coat layers, linked to a large extent to the porosity characteristic for each layer which was found to be more significant for the low Al-concentration specimens (Fig. 3b): the porosity and its condensation in form of gaps might act as a barrier, hampering the inward diffusion of aluminum to the substrate in the case of the low Al-concentration coating.

It is frequently found that the consumption of aluminum is mainly related to the interdiffusion with the substrate, being the most critical degradation mechanisms for aluminide coatings [8]. In addition to interdiffusion, the aluminum consumption is also affected by the formation of the external oxide scale and the formation and growth of AlN precipitates within the interdiffusion zone. None of these degradation mechanisms was observed.

Figure 10 shows the micrographs of a welded area between the austenitic TP321H steel and the VALIORTM T92 tube. In the non-coated area, Fe-rich oxides with similar morphologies and thicknesses as those found in Fig. 6c are formed. At the interface region between the chamfer area and the partially removed coating (Fig. 10) the coating still preserves its protective behaviour. The presence of iron oxide on top of the interdiffusion zone is related to the lateral growth commonly found for the iron-rich oxide scales grown in steam containing atmospheres [17]. The partially coated area must be considered as critical because of the lower aluminum concentration with respect to that of the top coat layer.

It is commonly known that the coating reaches the end of its lifetime when the surface aluminum concentration drops to a critical level where the protective aluminum oxide scale cannot be any longer sustained. Below this critical concentration, breakdown of the protective scales tends to occur. Several authors have found that the minimum aluminum concentration before breakdown occurs must be close to 7 wt% [10]. Velraj et al. [18] found that the critical aluminum concentration that leads to the formation of non-protective iron-rich oxide scales is much lower than the above mentioned value. The determination of the critical aluminum concentration to breakaway falls outside of the scope of this study. However, during this study no breakdown of the α -Al₂O₃ scale was observed. Similar aluminum contents were measured in a non-affected area of the coating at the same depth far from the chamfer area, indicating that this concentration is still above to the critical aluminum content required to form and sustain the protective aluminum oxide scale. The reason for the absence of breakdown of the protective scale, notwithstanding its low concentration, might be related to the high chromium content (8-10 wt%) present in the interdiffusion zone, a third element effect which is known to lower the critical aluminum content needed for breakdown to occur [19].

CONCLUSIONS

The oxidation performance of the new VALIOR™ T/P92 product was successfully tested at the EDF La Maxe power plant (France) under 167 b and 545°C (steam temperature). Two T92 tubes with and without inner coating were installed in the last superheater section of the power plant and welded to a TP321H (Fe-17.5Cr-10.5Ni) stainless steel tube. After 3500 h operation exposure, VALIOR™ coating exhibits an excellent oxidation resistance compared to the 9%Cr T92 steel. The reduction of the oxidation rates is achieved by the formation of a thin aluminum oxide layer on top of the coating. The efficiency of the VALIOR™ coating has also been demonstrated in laboratory testing. The mass gain when exposing the non-coated and VALIOR™ P92 steels for 3000 h in Ar-50%H₂O at 650°C is significantly higher in case of the non-coated specimen. The main difference between laboratory and power plant results for VALIOR™ coating is the inward diffusion of aluminum into the T/P92 substrate, which is likely linked to morphological differences of the top coat layers. Finally, an investigation close to the welds highlights no indication of enhanced fast growing oxide scales in the chamfer area when the standard T/P92 welding procedure is used.

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