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Creep behavior of pack cementation aluminide coatings on Grade 91 ferritic–martensitic alloy

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A B S T R A C T

The creep behavior of various pack cementation aluminide coatings on Grade 91 ferritic–martensitic steel was investigated at 650 °C in laboratory air. The coatings were fabricated in two temperature regimes, i.e., 650 or 700 °C (low temperature) and 1050 °C (high temperature), and consisted of a range of Al levels and thicknesses. For comparison, uncoated specimens heat-treated at 1050 °C to simulate the high temperature coating cycle also were included in the creep test. All coated specimens showed a reduction in creep resistance, with 16–51% decrease in rupture life compared to the as-received bare substrate alloy. However, the specimens heat-treated at 1050 °C exhibited the lowest creep resistance among all tested samples, with a surprisingly short rupture time of <25 h, much shorter than the specimen coated at 1050 °C. Factors responsible for the reduction in creep resistance of both coated and heat-treated specimens were discussed.

1. Introduction

The demand for increased energy efficiencies and decreased emissions has been the driving force for development of coal-fired power plants with higher steam temperature and pressure [1]. Under these operating conditions, the class of 9–12% Cr ferritic–martensitic (FM) steels which, may be creep resistant to 650 °C, can suffer extensive steam-side oxidation [2–4] and thus protective coatings need to be considered. Al-rich coatings are of particular interest because of the slow growth of alumina and its stability in steam and exhaust gas environments, as compared to the coatings that form chromia or silica-rich scales [5–8].

One particular concern for the use of Al-rich coatings on FM steels is the effect of coatings on the mechanical integrity of coated alloys, especially their creep resistance. Studies on Ni-based superalloys [9,10] have suggested that several factors are responsible for the reduction of creep resistance by a coating application, including: (1) changing the microstructure of the substrate material during the thermal cycle of the coating process; (2) decreasing the load-bearing cross-section of the component owing to the weak mechanical strength of the coating; and (3) enabling premature crack initiation in the coating layer. For FM steels, although some of these factors have also been noticed to cause a decrease in creep resistance of coated alloys (e.g., the reduction of load-bearing cross-section) [11,12], in general, the creep performance of coated FM alloys has not been studied to the same degree as coated Ni-base alloys.

This study focused on the effect of pack cementation coatings with various Al levels and thicknesses on the creep behavior of Grade 91 (Gr. 91) FM steel. The standard heat treatment procedure for commercial Gr. 91 alloys involves two steps [13]: (i) austenitizing at 1000–1150 °C for 10 min to 2 h, followed by rapid cooling to form martensite; and (ii) tempering at 730–780 °C for 1–2 h to promote carbide precipitation in the tempered martensite. Several European and US coating programs [7,8,14–16] have focused on synthesis of diffusion aluminide coatings at temperatures below the tempering temperature of the FM steel to preserve the tempered martensitic structure. However, in contrast to a typical aluminizing process at 900–1100 °C that produces phases such as FeAl, Fe_3Al or ferritic Fe(Al) in the coating, the reduced coating temperature often leads to the formation of more brittle Al-rich intermetallic phases like Fe_2Al_5 or even $FeAl_3$. Two temperature regimes were selected for coating fabrication in the present study. The low temperature (650 or 700 °C) was below the tempering temperature of Gr. 91, while the high temperature (1050 °C) was in the temperature range of its austenitizing treatment.

2. Experimental procedure

The commercial Gr. 91 alloy (Fe–8.8Cr–1.0Mo–0.5Mn–0.3V–0.2Ni–0.28Si–0.11C–0.06N–0.003S, in wt.%) was used as the substrate material. Small dog-bone specimens, as illustrated schematically in Fig. 1, were cut via electric-discharge machining (EDM). The gage length was ~7.6 mm, with a cross section of 2×2 mm^2. The specimens were ground to a 600-grit finish and ultrasonically cleaned in acetone prior to pack cementation or heat treatment.

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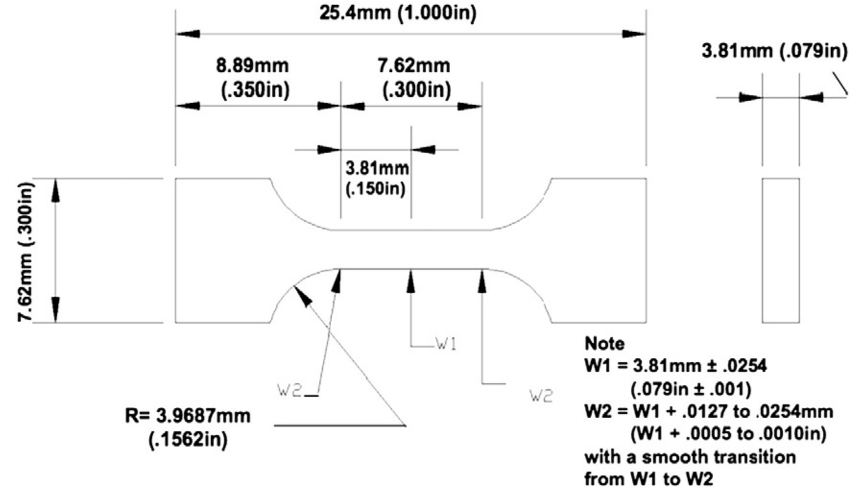

Fig. 1. Schematic showing the dimensions of the dog-bone creep specimen.

The pack mixture consisted of 1–2 wt.% NH_4Cl activator, 10–20 wt.% masteralloy, and the balance inert Al_2O_3 filler. Masteralloys of pure Al, Cr–25Al, and Cr–15Al (in wt.%) were employed to vary the Al activity in the pack cementation process, and thus to achieve coatings with different Al levels and thicknesses (Table 1). The specimens coated at 650–700 °C were directly embedded in the pack powder in an alumina crucible, similar to the conventional pack process [5]. For the coatings synthesized at 1050 °C, the substrate was hung in a slotted alumina tube to be separated from the surrounding powder, Fig. 2. Approximately 30 vertical slits (~0.18 mm wide) were machined around the 19 mm-OD alumina tube. The reagent gas species were able to interact with the specimen during pack aluminizing, whereas the powders were prevented from reaching the sample surface, leading to a cleaner coating. This non-contact assembly was not used at 650–700 °C, for the coating tended to be less uniform with the increased pack-to-specimen distance at lower aluminizing temperatures [16]. Additional Gr. 91 specimens were heat-treated at 1050 °C in the same arrangement as shown in Fig. 2, except that only inert Al_2O_3 powder (without masteralloy and activator) was placed around the slotted tube in the crucible.

The crucible was then sealed with an alumina lid using an alumina-based cement [17]. After the cement was completely cured, the crucible was loaded into a horizontal resistance-heated tube furnace, and purged with high-purity argon. A vacuum pump was connected to the furnace to aid in the removal of air and moisture, and a vacuum level of 0.13–0.40 Pa was achieved. Pack aluminization was carried out at this vacuum level. The coating time, 6 or 12 h, was defined as the holding time at the aluminizing temperature. The furnace temperature was monitored with a K-type thermocouple positioned in the center of the heating zone, which was also connected to a NI-9211A data logger from National Instruments. In addition, in order to more accurately monitor the temperature of the specimen inside the crucible, small thermocouples were attached to the surface of several specimens. The temperature–time profiles of both the furnace and the specimen were recorded with LabVIEW SignalExpress. After the aluminization was completed, the specimen was allowed to cool to room temperature in the furnace, and then was removed from the crucible and ultrasonically cleaned.

Creep tests were carried out at 650 °C in laboratory air under constant uniaxial loading, with a nominal stress level in the range of 100–120 MPa. For the coated specimens, the stress was calculated on the basis of the sample cross-section before the coating was applied. The uncoated specimens were tested with a 600-grit surface finish, whereas the coated or heat-treated samples were tested in the as-processed condition. The creep testing procedure was based on ASTM standard E139-11 [18]. An average of 2–3 specimens was tested for each condition, and all specimens were crept to rupture.

Specimens were examined by optical microscopy and scanning electron microscopy (SEM) equipped with energy dispersive X-ray analysis (EDXA). For cross-sectional observations, the coated specimens were copper-plated prior to metallographic sample preparation. Vickers microhardness test was conducted on the polished cross sections using a load of 500 gf. Each hardness value was taken as the average of five data points. Villela's reagent (100 ml methanol, 5 ml hydrochloric acid, and 1 g picric acid) was used to reveal the microstructure of the Gr. 91 alloy [19].

3. Results and discussion

3.1. As-fabricated coatings

Fig. 3 shows the cross sections of the as-fabricated aluminide coatings that were aluminized at different temperatures with various Al activities in the pack. When pure Al was used as the masteralloy, Fe_2Al_5 coatings of 80–125 μm thick were formed after 6 h at 650 °C (Fig. 3a), depending on

Table 1
Summary of creep specimens. Note that the pack cementation conditions are given as "temperature/time/masteralloy/amount of masteralloy in wt.%".

Treatment condition	Coating thickness (μm)	Creep rate		Time to rupture (h)	
		Rate (h^{-1})	Increase over "as-received"	Time (h)	Decrease over "as-received"
As-received	0	3.4×10^{-3}	–	344	–
650 °C/6 h/Al/10	105	6.0×10^{-3}	76%	288	22%
650 °C/6 h/Al/20	125	8.1×10^{-3}	138%	245	34%
700 °C/12 h/Cr–15 Al/20	18	5.2×10^{-3}	53%	275	26%
700 °C/12 h/Cr–25 Al/15	35	4.8×10^{-3}	41%	311	16%
1050 °C/6 h/Cr–15 Al/20	290	8.7×10^{-3}	156%	180	51%
1050 °C/6 h, heat-treated	0	N/A	N/A	23	94%