Growth and surface morphology of hot-dip Al–Si on 9Cr-1Mo steel

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ABSTRACT

The high-temperature oxidation behavior of a commercial cold-rolled 9Cr-1Mo steel sheet that was hot-dipped in Al–7 wt.% Si was studied. The oxidation kinetics showed a two-stage behavior. The oxidation rate was significantly faster during the first hour, followed by a decreased steady-state growth rate at longer times. The hot-dipped steel had a higher oxidation rate than has been reported for FeAl alloys. During oxidation, many large pores and needles or rod-shaped FeAl were formed beneath the oxide scale. The formation of pores was attributable to the consumption of the limited aluminum that is caused by the outward diffusion of aluminum to form γ-Al_2O_3 and the inward migration of aluminum by Fe/Al inter-diffusion. The formation of γ-Al_2O_3 and internal voids filled with oxides in the aluminide layer was responsible for the increase in the rate of oxidation.

1. Introduction

Commercial 9Cr-1Mo steel is an important candidate material for making tubes for use in boilers and heat exchangers that operate at up to 650 °C; it offers attractive oxidation and corrosion resistance with good high-temperature strength. The surface treatment by coating with FeAl intermetallics is well-known and commonly adopted for improving the resistance of steels to oxidation at elevated temperature. Coated steel is generally applied in the electric power industry, the petrochemical industry and energy conversion systems because of its low cost and favorable performance. Much research on this steel has been conducted [1–15]. A slow-growing alumina scale can be formed on the coated steel, acting as a protective barrier to suppress oxidation of the steel substrate at elevated temperature. Although FeAl intermetallics have an excellent high-temperature resistance to oxidation, these alumina formers seem to be particularly susceptible to the generation of pores at the scale/aluminide and the aluminide/alloy interfaces during oxidation at high temperature [16–20]. The formation of interfacial pores beneath the oxide scale is detrimental to the adhesion of the scale, and the Kirkendall effect is responsible for the formation of pores at the aluminide/alloy interface [21]. Meanwhile, many researchers have systematically studied the kinetics of alumina growth on alumina forming alloys at high temperatures [22–25]. However, the effect of both pore formation and alumina growth on the oxidation rate has rarely been investigated. This work explores the degree of parabolic behavior of hot-dipped 9Cr-1Mo steel that was oxidized at elevated temperature. The effect of the growth of alumina scales on the oxidation rate was investigated. Thermal gravimetric analysis (TGA) was adopted to evaluate the oxidation rate constants of coated steel in ambient air at 850 °C. The surface morphologies, microstructure and chemical composition of the aluminide layers after oxidation were examined.

2. Experimental Procedure

A cold-rolled sheet of ASME SA213 T9 steel with chemical composition 0.09% C, 0.36% Si, 0.40% Mn, 0.02% P, 8.25% Cr, 0.93% Mo, 0.04% Ni, 0.01% Cu, 0.225% V and balance Fe (wt.%) was employed in this study. Rectangular specimens were cut

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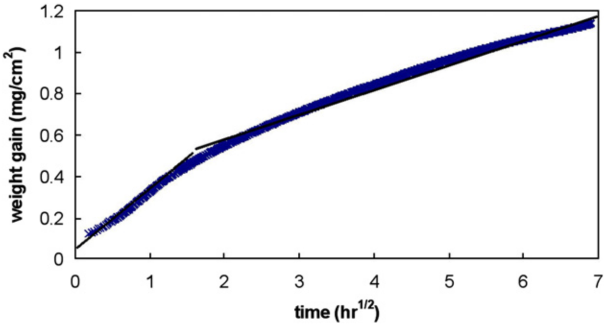

Fig. 1 – Oxidation kinetics of Al–Si coated 9Cr-1Mo steel as function of time at 850 °C.

to dimensions of 15×10×2 mm using a water-cooled cutting machine. The specimens were drilled, hung by stainless steel wires and coated with a uniform weld flux. The weld flux coated on the specimens is helpful in dissolving the oxide films that had formed on the molten aluminum bath during the dipping process. Dried specimens were then immersed in a bath of molten aluminum–silicon (7% Si, 0.3% Mg, 0.2% Fe, 0.2% Cu and balance Al in wt.%) at 700 °C for 16 s and moved up at an appropriate speed to yield a uniform coating on the steel. The hot-dipped specimens were further cleaned using a mixed solution of nitric acid, phosphoric acid, and water in 1:1:1 volume ratio at 60 °C to remove any surface flux that adhered during the fluxing process. The thickness of the aluminized layer was controlled at about 25 μm.

The coated specimens were exposed in a furnace to static air at 850 °C for various times and then air-cooled. The oxidation products were analyzed by X-ray diffraction using monochromatic Cu–Kα radiation at 40 kV and 100 mA. The surface morphology was examined using scanning electron microscopy and the composition of the phases that were formed on the coated layer was investigated using energy-dispersive spectrometry. The cross-sectioned specimens were characterized using optical microscopy and electron probe microanalysis with X-ray wavelength-dispersive spectrometry.

3. Results and Discussion

3.1. *Oxidation Kinetics*

Fig. 1 plots the weight gain as function of exposure time of a specimen that was oxidized at 850 °C, revealing the degree of parabolic behavior. In the initial oxidation stage, the oxidation rate was significantly higher during the first hour, followed by a decreased steady-state growth rate at longer times. The rate constants in the initial and steady stage were approximately 2.5×10^{-11} and 3.3×10^{-12} g^2 cm^{-4} s^{-1} respectively, which were one to two orders of magnitude higher than those of published alumina forming alloys [3,17,19,26] and NiAl [27].

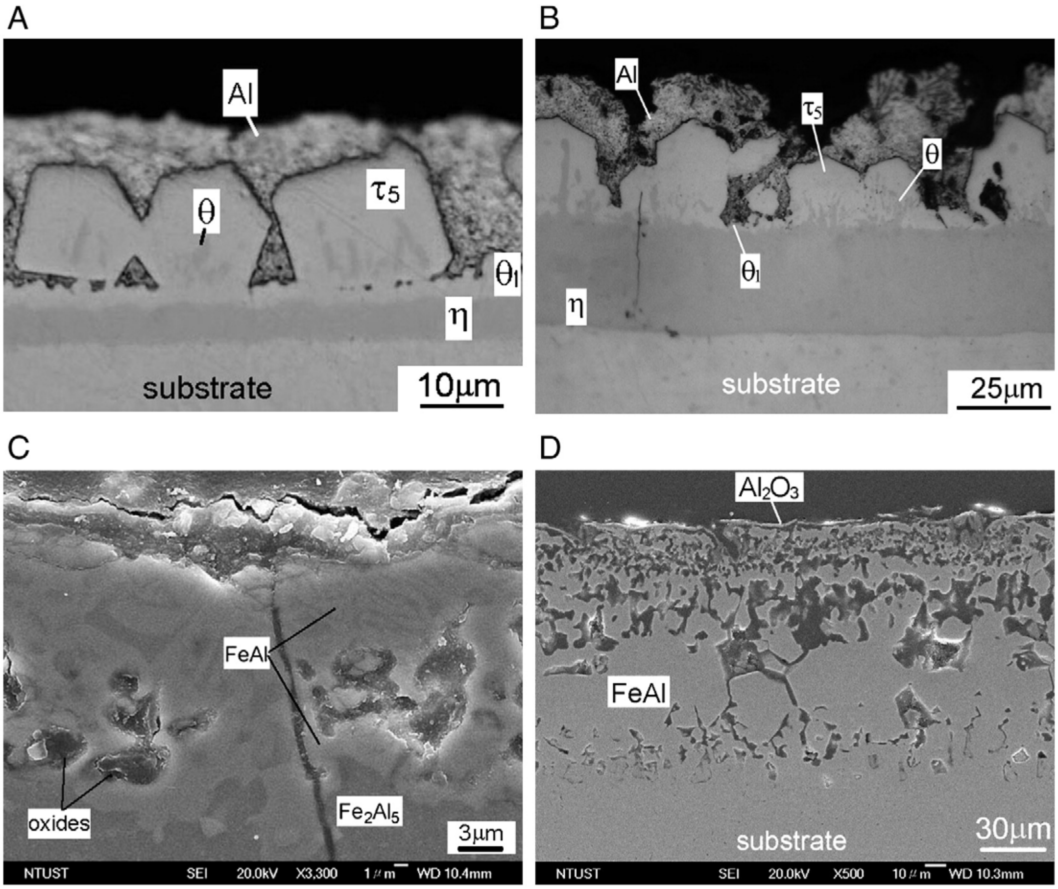

Fig. 2 – Cross-sectional micrographs of as-coated steel oxidized from 10 min to 24 h. (A) three layers — an aluminum topcoat, $FeAl_3(\theta_1)$ and the innermost $Fe_2Al_5(\eta)$, (B) FeAl phase(η) grew rapidly after oxidation for 10 min, (C) some needles or rod-shaped FeAl were formed in the outermost aluminide after oxidation for 1 h, (D) FeAl dominated after 24 h of oxidation.