



Low temperature pack aluminising kinetics of nickel electroplated on creep resistant ferritic steel

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ABSTRACT

A systematic study was made to determine the low temperature (<700 °C) growth kinetics of the Ni₂Al₃ layer formed by the pack aluminising process on the surface of nickel electroplated on creep resistant ferritic steels. The aluminising powder packs were prepared using Al powder as a source for depositing Al, Al₂O₃ powder as inert filler and anhydrous AlCl₃ as activator. The microstructures of the coatings formed were characterized by means of SEM/EDS and XRD. The results obtained were used to quantify the effects of aluminising temperature (*T*), time (*t*) and amount of Al powder (*W*) added into the pack powders on the growth kinetics of the Ni₂Al₃ layer (*h*). It was confirmed that the thickness of the Ni₂Al₃ layer formed can be related to the processing parameters by $h = k_0 W^{1/2} t^{1/2} \exp[-E_a / (RT)]$. The values of the pre-exponential constant *k*₀ and the activation energy *E*_a were determined. Additional experiments were made to demonstrate the effects of purity of the Al₂O₃ powder and amount of halide activator (AlCl₃) added into the powder packs on the growth kinetics of the Ni₂Al₃ layer.

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1. Introduction

With an aim to increase the steam temperature and pressure and hence thermal efficiency of steam turbine power plants, new grades of ferritic steels with higher creep strength at temperatures up to 650 °C have been developed [1]. However, these grades of steels contain only 9 wt.% Cr, which is not high enough to provide the required oxidation resistance, especially in steam environments, at temperatures up to 650 °C [2,3]. Attempts have also been made to develop ferritic steels with increased oxidation resistance by increasing the Cr content in the steels but none of such attempts has been successful so far due to the accelerated decrease in the long term creep strength caused by the microstructure instability in the high Cr containing steels [4–9]. Therefore, a number of detailed studies have been made recently to develop aluminising processes for forming a single layer of Fe-aluminide coating capable of forming the Al₂O₃ scale on the surface of the steels [10–15]. Compared with the Cr₂O₃ and SiO₂ scales, which all suffer accelerated volatility at higher steam temperatures [16–18], the Al₂O₃ scale is chemically more stable and hence can provide long term protection in high temperature steam or water vapour containing environments [19]. Indeed, it has been found that the lifetime of such single layer Fe-aluminide coatings is not determined by the rate of oxidation, but by the rate of interdiffusion at the coating/steel interface, which reduces the Al concentration in the coating at an undesirably fast rate at the intended maximum application temperature of 650 °C for the steels [13–15].

To minimise the shortcoming of the single layer Fe-aluminide coatings described above, a type of hybrid coatings consisting of an outer Ni₂Al₃ layer and inner Ni layer has been proposed recently [20,21]. The oxidation resistance of the coating is provided by the outer Ni₂Al₃ layer and the inner Ni layer functions as a barrier layer that slows down the inward Al diffusion. During service at high temperatures, although the Al concentration in the outer Ni₂Al₃ layer will be reduced and the phase in the outer layer transformed progressively to the Ni-aluminides of lower Al concentrations by the process of the inward Al diffusion into the inner Ni layer, the rate of Al depletion in the outer Ni-aluminide layer has been demonstrated to be much lower than that in the single layer Fe-aluminide coating even at temperatures as high as 900 °C [22]. This effectiveness of the inner Ni layer in resisting the inward Al diffusion may be attributed to the much lower Al diffusion rate in Ni than in Fe or Fe-base alloys.

The hybrid Ni₂Al₃/Ni coating can be produced on the steel surface by firstly electroplating or electroless plating of a high purity Ni layer and then partially aluminising the plated Ni layer using pack cementation process [20–22]; the latter step is similar to the conventional pack process for aluminising Ni base superalloys. The difference is that the aluminising temperature is normally higher than 800 °C for Ni base superalloys whereas it must be kept below 700 °C for creep resistant ferritic steels because the microstructures of these grades of steels are optimised by heat treatment to achieve the highest possible creep strength at their intended service temperatures. Although many studies have made to establish a model capable of predicting the aluminising kinetics for pure Ni and Ni-base superalloys in the temperature range above 800 °C [23–25], the only piece of systematic work on aluminising Ni in the temperature range below 700 °C is that reported by Rashidi

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and Amadeh [26]. The powder packs used by these investigators consisted of only Al and NH_4Cl powders. Without the inert fillers such as Al_2O_3 , sintering or partial sintering is likely to occur in the powder packs even at temperatures as low as $500\text{ }^\circ\text{C}$ [27], which would cause considerable inconvenience in practical applications. The present study is thus undertaken to quantify the kinetics of pack aluminisation of Ni electroplated on creep resistant ferritic steels in the powder packs consisting of Al powder as a source for depositing Al, Al_2O_3 powder as inter filler and anhydrous AlCl_3 powder as activator in the temperature range below $700\text{ }^\circ\text{C}$.

2. Theoretical basis of the work

For the powder packs consisting of Al, Al_2O_3 and a halide salt of sufficiently high activating strength such as AlCl_3 or NH_4Cl , the reactions between Al and halide salt in the powder packs at high temperatures will produce a series of Al-halide vapour species such as AlCl , AlCl_2 and AlCl_3 , which will in turn migrate to the Ni surface on which they decompose to deposit Al atoms. The deposited Al atoms will then diffuse into Ni, forming a Ni–Al alloy layer on the Ni surface [23,24]. In the temperature range below $700\text{ }^\circ\text{C}$, the Ni–Al alloy layer formed will be a single intermetallic compound layer of Ni_2Al_3 , which will grow in thickness as aluminising time increases. By following a similar analytical procedure to the one that led to the establishment of the growth kinetics of the Fe_2Al_5 layer on steels [27], the following kinetics can be derived for the growth of the Ni_2Al_3 layer on Ni:

$$h = k_0 W^{1/2} t^{1/2} \exp[-E_a/(RT)] \quad (1)$$

where h is the thickness of the Ni_2Al_3 layer formed, W is the amount of Al powder added into the powder packs, t and T are the aluminising time and temperature in K, respectively, E_a and R are the activation energy and gas constant, respectively, and k_0 is the pre-exponential constant. Eq. (1) relates the growth of the Ni_2Al_3 layer not only to aluminising temperature and time but also to the amount of Al powder added into powder packs. The derivation of it requires the assumptions that there is no Ni loss into the powder pack during the aluminising process and the Al deposited on the surface is reacted fully with Ni to form the Ni_2Al_3 layer [27]. The implications of these assumptions are twofold. Firstly, regardless of the aluminising conditions, the relationship between the Ni_2Al_3 layer thickness (h) and the mass gain of the unit surface area of the specimen (Δm) should obey [27]:

$$h = \frac{M_c}{3M_{\text{Al}}\rho} \Delta m \quad (2)$$

where M_c is the molecular mass of Ni_2Al_3 , M_{Al} the atomic mass of Al, and ρ the density of Ni_2Al_3 . Secondly, the Al concentration in the outmost surface or at the any equivalent depth beneath the outmost surface of the Ni_2Al_3 layer should remain the same in different specimens aluminised under different conditions, i.e., it should not vary with aluminising time or temperature or the amount of Al powder added into powder packs. The purpose of the present study is to demonstrate the validity of these assumptions and consequently to determine the values of k_0 and E_a in Eq. (1) for the growth of the Ni_2Al_3 layer on electroplated Ni at temperatures below $700\text{ }^\circ\text{C}$.

3. Experimental procedures

The substrate used in the present study is a commercial grade of creep resistant ferritic steel P92 with a chemical composition of Fe–8.6Cr–1.8W–0.5Mo–0.53Mn–0.48Si–0.18V–0.07Nb–0.1C–0.04N (wt.%). The dimensions of the specimens used were approximately $20 \times 10 \times 2$ mm. They were ground to a 1000 grade finish and then thoroughly degreased in ethanol. A layer of Ni was then electroplated on the surface of the specimen using the conventional Watt process in a plating solution of 200 g/L

$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, 40 g/L H_3BO_3 , 40 g/L NiCl_2 , and 120 g/L $\text{C}_6\text{H}_5\text{Na}_3\text{O}_7 \cdot 2\text{H}_2\text{O}$. The Ni layer thickness on all the specimens was controlled at approximately 100 μm .

Powders of Al (99.5% purity, Alfa Aesar), Al_2O_3 (99% purity, Acros) and anhydrous AlCl_3 (99% purity, Alfa Aesar) were used to prepare the aluminising packs. The particle sizes of Al and Al_2O_3 powders were proximately 5 and 50 μm , respectively. In all the aluminising experiments, the amount of AlCl_3 activator added was 2 wt.% unless otherwise stated. The temperature range studied was from $525\text{ }^\circ\text{C}$ to $650\text{ }^\circ\text{C}$ and the aluminising time used ranged from 1 h to 16 h. The specimen to be aluminised was buried in the pack powder mixture, which was filled into a cylindrical alumina crucible of 30 mm in diameter and 40 mm in length. In each aluminising experiment, only one specimen was packed into the crucible and care was taken to ensure that the pack powder was filled right to the top of crucible and the location of the specimen in the crucible was approximately the same. The crucible was then covered with an alumina lid and sealed from the outside using high temperature cement. The aluminising process was carried out in a tube furnace using flowing argon as a protective atmosphere. The weight of the specimens was measured before and after the aluminising treatments and the mass gain in mg cm^{-2} was calculated from the weight change and surface area of the specimens.

The cross-sectional microstructure and depth profiles of alloy elements in the aluminised layer were analysed by means of scanning electron microscopy (ESEM) (FEI Quanta200) and energy dispersive X-ray spectroscopy (EDS) (Oxford INCA). The phases present in the surface layer of as-aluminised specimens were identified by X-ray diffraction (XRD) (Siemens D5000 diffractometer with $\text{Cu-K}\alpha$ source radiation). The thickness of the aluminised layer was estimated from the Al depth profiles measured by EDS.

4. Results and discussion

4.1. Morphology and structure of the aluminised layer

The surface of all the specimens aluminised at temperatures below $700\text{ }^\circ\text{C}$ was quite smooth without embedded pack particles, indicating that the aluminised layer was formed by inward diffusion of Al deposited from the vapour phase. The typical cross-sectional structure of the coating consisted of an outer Ni_2Al_3 layer and an inner Ni layer. An example is shown Fig. 1, which is for a specimen aluminised at $650\text{ }^\circ\text{C}$ for 4 h in a pack 6Al–92 Al_2O_3 –2 AlCl_3 (wt.%). Both the depth profiles of Al and Ni in the aluminised layer (Fig. 1b) and the XRD pattern measured from the surface (Fig. 2) suggested that the outer aluminised layer formed on the surface of the electroplated Ni was a compound layer of Ni_2Al_3 in which the Al concentration was slightly higher than the stoichiometric concentration (60 at.% Al) (Fig. 1b).

According to the Ni–Al phase diagram, the outmost layer formed on the surface is expected to be NiAl_3 instead of Ni_2Al_3 . The fact that the NiAl_3 layer was not present in the coating indicates that the partial pressures of the Al-chloride vapour species generated in the pack were not high enough for the formation of such a phase layer. Other phases that may form beneath the outer Ni_2Al_3 layer at $650\text{ }^\circ\text{C}$ include NiAl , Ni_5Al_3 and Ni_3Al . As will be shown in the following section, the parabolic rate constant for the growth of the Ni_2Al_3 layer at $650\text{ }^\circ\text{C}$ is approximately $15.29\text{ }\mu\text{m h}^{-1/2}$, which is far higher than the rate constants for the growths of NiAl ($1.42\text{ }\mu\text{m h}^{-1/2}$), Ni_5Al_3 ($0.16\text{ }\mu\text{m h}^{-1/2}$) and Ni_3Al ($2.50\text{ }\mu\text{m h}^{-1/3.58}$) [21]. Thus, if the latter three phase layers were present in the coating, they would be exceedingly thinner than the outer Ni_2Al_3 layer. The SEM image taken at a much higher magnification showed that two separate thin layers were indeed present at the interface between the outer Ni_2Al_3 layer and electroplated Ni (Fig. 3). The alloy compositions measured using EDS from the two points pointed by arrows 1 and 2 in Fig. 3 were 53.8Al–46.8Ni (at.%) and 52.9Al–47.1Ni (at.%), respectively, suggesting that the layer formed adjacent to the outer Ni_2Al_3 layer was Al-rich NiAl. The alloy compositions

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