



High temperature oxidation and corrosion behaviour of Ni/Ni–Co–Al composite coatings

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ABSTRACT

In the present study, Ni/Ni–Co–Al composite coatings were developed by a potentially simple, scalable, non-vacuum technique namely electrodeposition. These coatings were characterized for their micro-hardness, oxidation and hot corrosion behaviour. An increase in Co content in the matrix from 8 wt% to 70 wt% led to an increase in the Al particle incorporation from 12 wt% to 21 wt%. A change in the surface morphology of the coatings with variation in Co content was seen. The oxidation behaviour of the coatings was studied at temperatures in the range of 400 °C to 1000 °C. The influence of vacuum treatment on the high temperature behaviour of the coatings was also investigated. The intermetallic aluminide phase formation was observed in the temperature range of 600–800 °C and a homogenized structure was seen at 1000 °C. The oxidation rate in terms of weight gain was marginally lower for vacuum pretreated Ni–Al coating annealed at 1000 °C. A significant increase in the oxidation rate was exhibited by Ni–70Co–Al coating beyond 800 °C showing its poor oxidation behaviour. The characterization studies revealed the formation of stable alumina in the case of Ni–Al while, metastable alumina was observed in Ni–Co–Al coatings. The hot corrosion studies showed that Co rich Ni–Co–Al exhibited better resistance compared to Ni rich coatings. An optimum cobalt content of 30 wt% was desirable for high temperature oxidation and corrosion resistance.

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1. Introduction

Gas turbine components are susceptible to oxidation, hot corrosion, and reduction in mechanical properties due to the harsh environments experienced by them. Consequently, these components need to be protected by an environmental coating, alone or in combination with an outer ceramic coating which in the latter case is termed as thermal barrier coating. Metallic coatings are being used as environmental coatings for protection and the most widely used are the diffusion coatings such as the aluminides. Nickel aluminides are predominantly being used [1,2] due to their low density, high strength at elevated temperature and excellent resistance to high temperature corrosion [3]. The aluminium content acts as a reservoir for the slow growth of a strong adherent continuous aluminium oxide layer that protects the coating and the underlying metal from oxidation. These coatings are obtained by chemical vapour deposition (CVD), pack cementation [4], physical vapour deposition (PVD), self-propagating high-temperature synthesis (SHS) and thermal spraying [3]. Recent interests are to adopt electrodeposition which is a two step process involving the

co-deposition of metal matrix Ni and metal particle Al composite followed by a diffusion treatment to form the intermetallic coatings. Compared to the other coating techniques this is a simple, low cost and low temperature process, high productivity and composition controllability coating method [5,6]. The advantages of this method have led to its adoption in the present study for the development of Ni and Ni–Co aluminide coatings. Considerable research has been carried out on the electrodeposition of Ni aluminide coating [3–13]. Initially Susan et al. developed Ni–Al composite coating from sulphamate electrolyte and observed no crack, porosity or other defects in the coating [7]. Heat treatment of the coatings at temperatures of 400–450 °C produced a range of Ni–Al intermetallics [8]. The oxidation kinetics study showed that after 1000–2000 h at 1000 °C, a thick Al₂O₃ layer is found below a thin outer spinel layer [9]. Ni aluminide coating was also obtained by sediment co-deposition method and upon thermal treatment at 900 °C resulted in the formation of NiAl, Ni₃Al phases [3,10]. Zhou et al. studied the oxidation behaviour of Ni–28Al nano-composite coating at 1050 °C and reported that the presence of Al nanoparticles resulted in fast formation of Al₂O₃ scale [4]. Yang et al. have reported that a diffusion homogenization treatment prior to oxidation favours the selective oxidation of aluminium [6].

Although, significant research has been carried out on Ni aluminide coatings, scarce information is available on Ni–Co

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Nomenclature

AP	as-deposited coating
VT	homogenized coatings
NA	Ni–Al coating
NC8	Ni–8 wt%Co–Al coating
NC28	Ni–28 wt%Co–Al coating
NC70	Ni–70 wt%Co–Al coating
EDX	energy dispersive X-ray analysis
FESEM	field emission scanning electron microscope
XRD	X-ray diffraction
XPS	X-ray photoelectron spectroscopy
UHV	ultra high vacuum

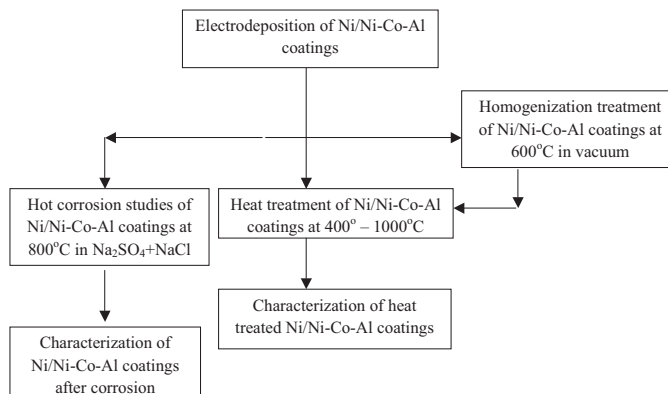
aluminides. The advantage of choosing Ni–Co matrix is that the alloying of Ni with Co hardens it in a sulphur free form by forming a solid solution, which does not embrittle on heat treatment [14,15]. Hence, the influence of Al particle incorporation in various Ni–Co matrices on the structure, oxidation and corrosion behaviour has been aimed in the present study.

2. Materials and methodology

Ni/Ni–Co–Al composites were electrodeposited from an additive free conventional sulphamate electrolyte. The size of the Al particles used was in the range of 1–3 μm and a content of 75 g/L. The Co content in the electrolyte was varied so as to obtain Co content of 8 wt%, 28 wt% and 70 wt% in the coating and the coatings are designated as NA, NC8, NC28, NC70 respectively. The deposition was performed at pH 4.0, current density 1.6 A/dm² under ambient conditions using a stainless steel substrate for a time duration so as to obtain a thickness of $50 \pm 5 \mu\text{m}$. The microhardness of the coatings was measured using Knoop's indenter by indenting across the cross-section of the coating at various locations and applying a load of 50 gf. The values reported are the average of various measurements made at different locations. The thermal stability of the coatings was studied by subjecting the composite electroforms to isothermal heat treatment at temperatures of 400 °C, 600 °C, 800 °C and 1000 °C for time duration of 6 h to obtain the aluminide and stable oxides. A comparison was also made with a set of composite coatings that were subjected to homogenization treatment in vacuum ($\times 10^{-6}$ mbar) at 600 °C for 4 h followed by isothermal heat treatment. The heat treatment was carried out under atmospheric conditions in a muffle furnace. The coatings were characterized for the Al and Co contents using EDX and the surface morphology using FESEM in scanning electron (SE) mode. The oxidation products were identified using XRD studies. The oxidation behaviour of the various coatings was determined by measuring the change in weight with respect to temperature. The hot corrosion study was carried out by initially preheating the coatings on a hot plate for 30 min followed by applying a paste of 75% Na₂SO₄ and 25% NaCl using a brush so as to obtain a salt deposit of 4–6 mg/cm². The salt coated deposits were dried and heat treated by placing in a muffle furnace preheated to 800 °C for duration of 6 h. The heat treated coatings were taken out, cooled to ambient conditions, washed with boiling distilled water for 30 min and ultimately dried and weighed. The hot corrosion resistance has been expressed in terms of mass gain.

The XPS were acquired using a SPECS make system using 100 W Al K α radiation. Survey spectra and high-resolution core level spectra of elements were acquired at 70 and 25 eV pass energy, respectively. The samples were cleaned with methanol before loading into the UHV system and sputter cleaned at 1 keV Argon ions for 3 min prior to acquisition of spectra. In estimating the atomic percentages, the entire area under the core level spectra with the

spin orbit doublets was used. The atomic sensitivity factors as given in [16] were used for Ni2p, Co2p, Al2p and O1s core levels. All the binding energies are referenced with respect to C1s core level. The flow chart of the activities is shown below:



3. Results and discussion

3.1. Electrodeposition and surface morphology of Ni/Ni–Co–Al composites

The Ni/Ni–Co–Al composites were electrodeposited and the Al and Co contents were obtained by EDX analysis. In the as-deposited condition the cobalt content in the coatings was 0, 8 wt%, 28 wt%, 70 wt% and the respective Al content was 12 wt%, 17 wt%, 19 wt% and 23 wt%. The increase in Al particle incorporation with increase in Co content can be associated with the good wettability of Co compared to Ni. Similar increase in particle incorporation has been observed by the authors in the case of inert particles like SiC, CeO₂, Si₃N₄ [17–19]. The influence of homogenization treatment on Al incorporation was studied. The Al contents were seen to be 13 wt%, 14 wt%, 17 wt%, 25 wt% for Co contents of 0 wt%, 8 wt%, 28 wt%, and 70 wt% respectively. Minimal change in the microstructure was seen between the as-deposited and homogenized coatings. The surface morphology of the AP coatings is depicted in Fig. 1. The morphology of as-deposited NA coating appears as cauliflower Ni crystallites with spherical black and white particles distributed throughout. Small polyhedral Ni–Co crystallites containing black and white spherical particles distributed throughout are seen on the surface of NC8 composite. Increase in the matrix Co content to 28 wt% resulted in a rough nodular morphology (Fig. 1). Further increase in Co content to 70 wt% resulted in the transformation to acicular morphology with black and white particles distributed. The black and white particles were identified by localized EDX as aluminium. The white particles were rich in Al compared to black or conversely the Ni/Ni–Co content was less in the case of white particles compared to black. Hence, it can be concluded that the black particles are covered with a layer of Ni/Ni–Co film resulting in a low Al content. This can be linked with the fact that those Al particles which are aligned along the preferred orientation of Ni or Ni–Co are covered with its film and the remaining is uncovered. The change in the matrix morphology is due to the change in the Co content. A similar change in the morphology with increase in Co contents has been reported by the authors [20].

The surface morphologies of the VT coatings are shown in Fig. 2. The morphology of Ni–Al coating appears as greyish flakes lying on the surface of the Ni matrix. It appears as though the white particles seen in Fig. 1 have melted during the homogenization at 600 °C to form the flakes. At the edges of the black particles the reaction has initiated. The Al content was seen to be higher on the flake (75 wt%) compared to the reaction product (33 wt%) formed on the surface of the black particles. Thus, the reaction product may

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