JMEPEG (2002) 11:211-214

Microstructural Study on Oxidation of Aluminized Coating on Inconel 625

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(Submitted 15 December 2001; in revised form 8 January 2002)

The high-temperature oxidation behavior of aluminized Inconel 625 has been examined using scanning electron microscopy (SEM) and fine-probe spot and linescan energy dispersive spectroscopy (EDS) microanalysis techniques. The formation of adherent slowly growing metallic coatings is essential for protection against severe environments. The coated (aluminized) and uncoated samples were subsequently oxidized in air at 1000 and 1100 °C to examine the performance of the aluminized coating. The microstructural changes that occurred in both coated and uncoated samples were examined. It was found that coated samples showed superior performance against high-temperature oxidation, as compared to the uncoated samples. The coated samples revealed uniform and adherent oxidized layer, as compared to the uncoated samples. The oxidation behavior and multilayered microstructural morphology observed in the coated samples can be attributed to the interdiffusion and variation in kinetics of oxidation.

Keywords aluminized coating, composition, diffusion, Inconel, linescan microanalysis, microstructure, oxidation, pack cementation, spallation, superalloy

1. Introduction

Surface modification can be applied to most engineering components in order to enhance their performance and service-life under severe conditions. Pack cementation is one of the well-known diffusion coating methods that is employed to improve the surface properties and performance of superalloys that are exposed to high temperature and severe conditions. Aluminized coating can be used for moderate- to high-temperature applications in aircraft and in ground-based and marine engine components. These coatings can produce significant protection and cost advantages, as compared to other techniques.[1-4] The coatings have shown better performance in hot corrosion environments despite the limitations due to formation of NiAl intermetallic compounds in the coating matrix.[5-8] Previous work[9,10] also described the behavior of coatings under rupture stress and creep failure for different kinds of substrate alloys. It is also reported[11] that the coating can exhibit better ductility at relatively higher temperature as compared to lower temperature, and the ductile to brittle transition temperature (DBTT) can be controlled by the level of aluminum in the coatings. However, detailed characteristics of the microstructure of aluminized coatings evolved at different exposures have not been reported for superalloys.

In this part of the work, the microstructural and phase changes in the coated and uncoated samples of Inconel 625 that were oxidized at different temperatures have been examined to optimize the processing parameters for surface modification of a series of superalloys.

2. Experimental

The chemical composition of the Inconel 625 used in this work is presented in Table I. The aluminized coating layer was deposited by pack cementation carried out at 900 °C for 8 h in an argon atmosphere, as per procedure described elsewhere.[5,7] The composition of aluminizing mixture is given in Table 2. The aluminized samples were oxidized in air at 1000 and 1100 °C for 50 h. The samples having an area of 2 cm^2 were prepared and ground to 180 grit SiC paper finish followed by degreasing, drying, and weighing. Standard metallographic techniques were used for the examination of coated and uncoated isothermally oxidized samples. The coated and uncoated samples after oxidation were examined in the transverse direction using a Philips XL 30 scanning electron microscope (SEM) operated at 20–30 kV. Spot and linescan analyses were also carried out using a Link-ISIS energy dispersive spectroscope (EDS) system attached to the SEM.

3. Results and Discussion

3.1 Kinetics of Oxidation

The earlier work[1] presented on oxidation kinetics has revealed significant weight losses in the uncoated samples, as compared with the coated (aluminized) samples. The spallation and internal oxidation was found more severe in the uncoated samples, as compared to the coated samples exposed to the range of high temperature. The coated and uncoated specimens had been tested for their oxidation resistance at 1000, 1100, and 1200 °C, exposing under air for 1, 10, and 50 h. In general,

Table 1 Chemical Composition of Inconel 625 (wt.%)

Cr	Fe	Mo	Nb	Mn	Si	Ni
22.0	2.50	9.00	4.00	0.20	0.20	62.10

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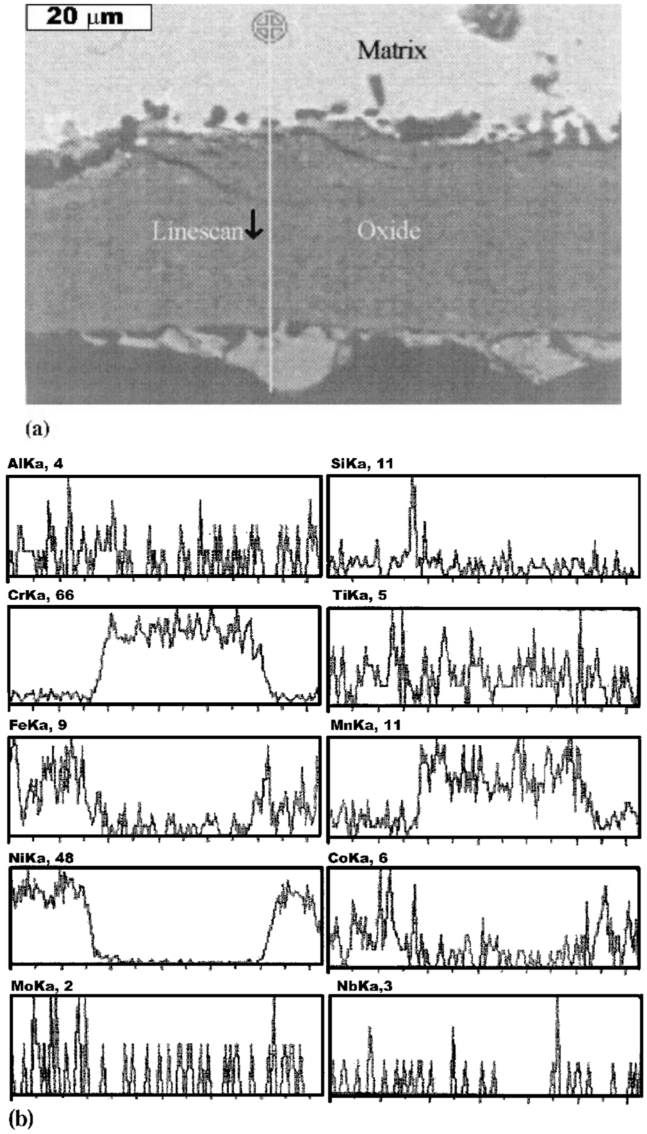


Fig. 1 **(a)** SEM-EDS linescan image and **(b)** concentration profiles of the elements across the oxide layer in an uncoated sample oxidized at 1100 °C

Table 2 Composition of Aluminizing Mixture (wt.%)

Aluminum Powder	Ammonium Chloride	Alumina Powder
15	3	82

cubic rate law was dominated for exposure up to 10 h and parabolic rate law for longer exposures in both sets of specimens. However, the kinetic curves of the uncoated specimens lied in a much higher weight gain regime, as compared to the aluminized specimens.

3.2 Structure of Uncoated Samples

Figure 1 shows an example of the oxide layer on the sample after isothermal oxidation at 1100 °C for 50 h in air. A dense oxide layer with internal microcracks is evident. The SEM-EDS linescan microanalysis revealed that the oxide layer was rich in Cr and Mn (Fig. 1b), indicating the formation of spinel ($MnCr_2O_3$).

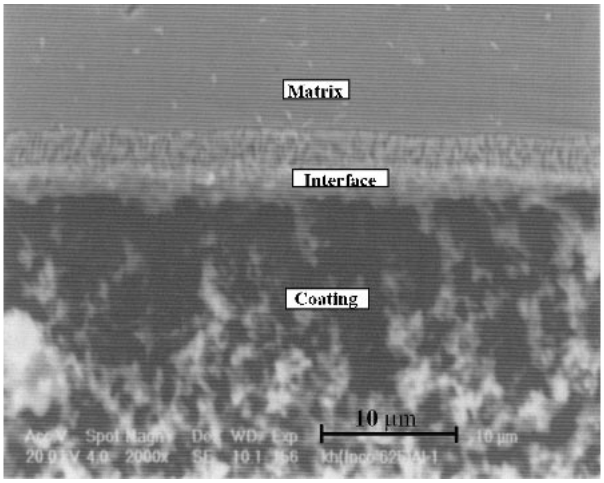


Fig. 2 SEM micrograph showing the layer structure of an aluminized coating in the sample oxidized at 1100 °C

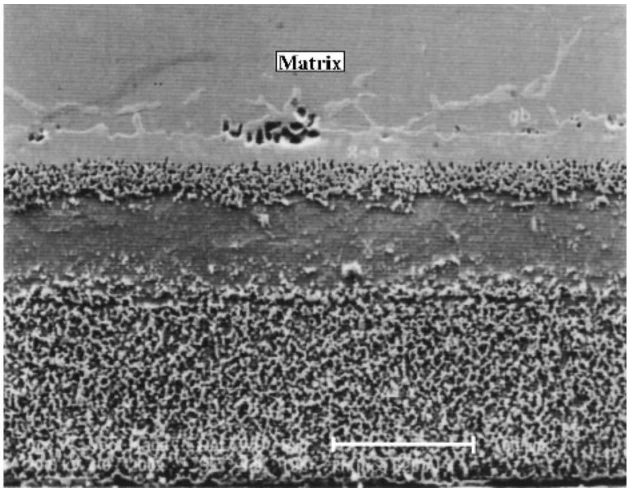
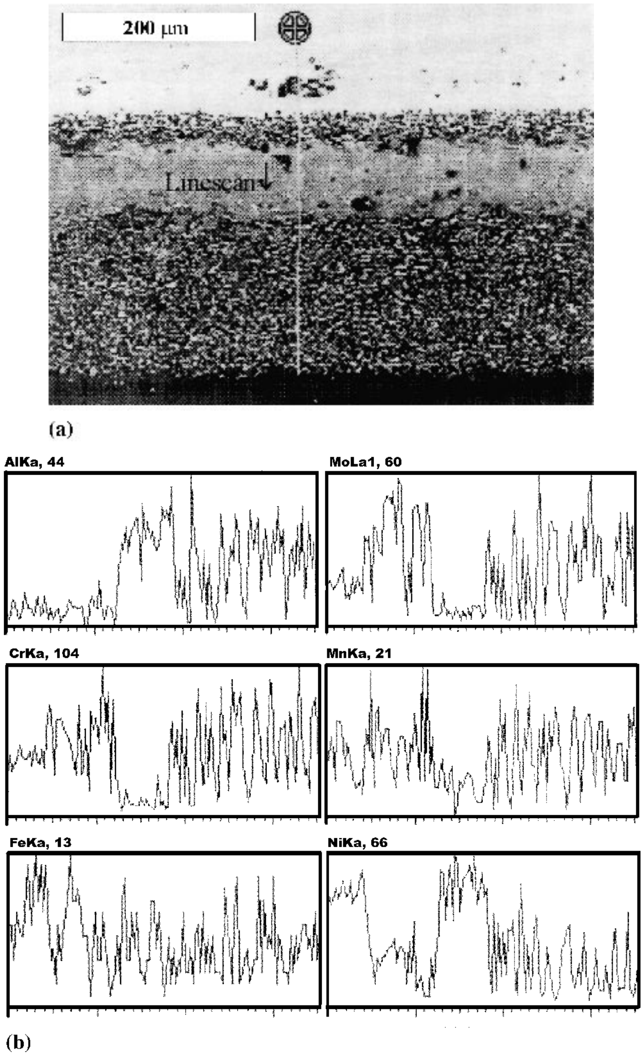


Fig. 3 SEM micrograph showing multilayered oxide structure in the coated sample oxidized at 1100 °C

3.3 Structure of Coated (Aluminized) Samples

Figure 2 shows a uniform and dense aluminized coating prepared by the pack cementation process. The intermediate layer developed between the coating and matrix is also evident. The intermediate phase was identified as NiAl, as discussed in the previous work.[2] The average thickness of the aluminized layer was 200 μm. The aluminized layer was adherent and crack-free and composed of randomly scattered globular nodules formed during the cementation process.

Figure 3 shows a multilayered structure observed in the sample oxidized at 1100 °C in air. The SEM-EDS linescan



(a)

(b)

Fig. 4 **(a)** SEM-EDS linescan image and **(b)** concentration profiles of the elements in an aluminized sample oxidized at 1100 °C

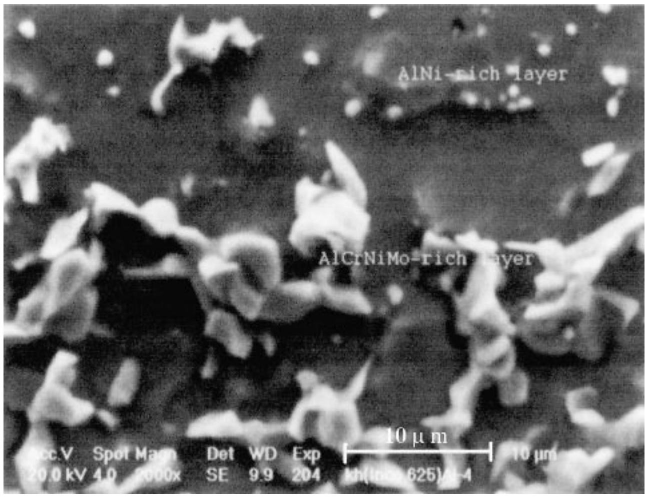
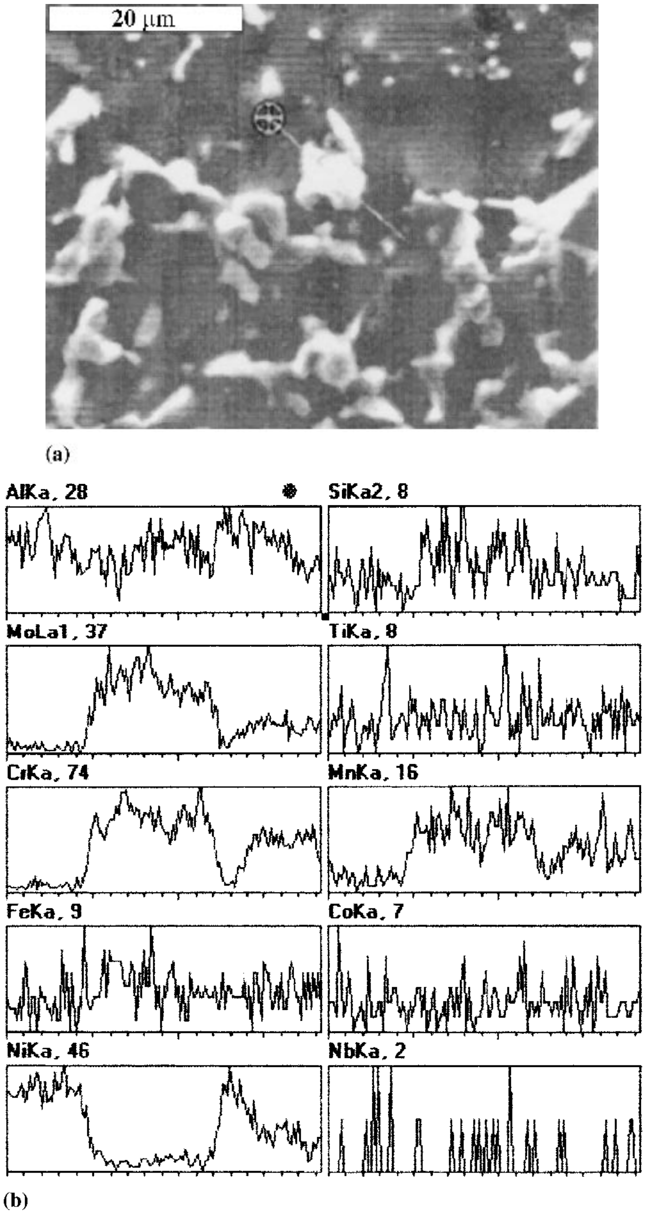


Fig. 5 SEM micrograph showing AlCrNiMo-rich particles in NiAl matrix of an oxidized coated sample



(a)

(b)

Fig. 6 **(a)** SEM-EDS linescan image and **(b)** concentration profiles of the elements across the irregular-shaped Cr-rich particles observed in the sample oxidized at 1100 °C

microanalyses revealed distinct regions of Cr-rich layer at the substrate alloy interface, followed by Ni- and Al-rich outer layers (Fig. 4). The general trends for the interdiffusion of elements were similar to the samples exposed at 1000 °C. This may have resulted from the inward diffusion of Al to combine with Ni in the substrate alloy. Internal oxide layers of Cr- and Mo-rich concentration were also developed along the grain boundaries as a consequence of pipe diffusion, as reported in previous work.[12] Furthermore, growth of irregular-shaped particles has evolved in the interspersed multilayer aluminized structure of the sample (Fig. 5). The particles were analyzed and found rich in CrMnMo contents whereas the matrix of the coating was found richer in Ni and Al, as illustrated in Fig. 6(a) and (b). This result showed formation of different layers and growth of particles as a consequence of the difference in kinetics of oxidation in the aluminized samples. It was observed that the integrity of the aluminized coating had been consistent in the samples exposed at different temperatures. No evidence of cracking and spallation were found in the coated samples, as compared to the uncoated samples.

4. Conclusions

- The oxidation response on the microstructure developed in the aluminized coated and uncoated samples of Inconel 625 was investigated. Significant improvement in the oxidation resistance of the coated samples was observed, as compared to the uncoated samples exposed at high temperatures.
- Evidence of severe oxidation, microcracks, and spallation was found in the uncoated sample whereas a multilayered adherent coating structure was observed in the coated samples.
- The structure of oxidized coating layer also revealed irregular-shaped CrMnMo-rich particles interspersed in the NiAl-rich matrix due to interdiffusion during exposure at high temperatures.

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