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NiAl Diffusion Coatings on Inconel 738 using a Pre-Heated $AlCl_3 + H_2$ Gas Mixture

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Abstract

Aluminized diffusion coatings were obtained on an Inconel 738 specimen at 800–1000°C using a pre-heated $AlCl_3 + H_2$ gas mixture with a 0·3 s holding time. It was found that the pre-heating treatment was very effective for a high deposition rate of the aluminide coatings. The thickness of the aluminide coatings was 1·5–4·7 times higher than that without pre-heating. © 1996 Published by Elsevier Science Limited.

1 Introduction

Aero-engine blades are exposed to extremely severe environmental conditions such as high temperatures of 950–1100°C, severe oxidation, carburization and corrosive atmospheres. Under marine and industrial service conditions, the blades encounter even more corrosive and/or erosive atmospheres but the operating temperature range is relatively low, 700–850°C. Accordingly, various surface coatings are applied to these components to withstand such severe conditions and thereby extend component lifetime.

There are two main coating processes: diffusion coatings and overlay coatings. In the production of diffusion coatings, metal layers of Al, Cr, Ni, Si, etc. are deposited on the substrate followed by thermal diffusion to form corrosion-resistant diffusion coatings. Aluminized diffusion layers are frequently used for hot corrosion protection of gas turbine blades. Among the various diffusion coatings, the pack coating process is the most widely used technique for aluminide coatings.[1–4] However, there is some difficulty in uniform feeding of the pack powder into the narrow passages (<0·5 mm diameter) that are used for cooling air and then subsequently removing the powder. The metal–organic chemical vapour deposition (CVD) process has been used for aluminium or aluminide coatings.[5,6] However, the aluminium or aluminide coatings obtained by this process are generally not adherent to a substrate and the growth rate is very low. Furthermore, the coating operation is not easy because the alkyl aluminium source is very unstable to moisture and oxygen, and is flammable in air.

The conventional thermal CVD process using $AlCl_3$ or $AlBr_3$ as the aluminium source has frequently been used for aluminide coatings. Aluminide coatings using the direct hydrogen reduction of $AlCl_3$ or $AlBr_3$ need a temperature above 1000°C, or about 1200°C if thick coating layers are needed. Accordingly, AlCl, which has a high reactivity and is obtained using the reaction of $AlCl_3$ with molten aluminium at 900–1000°C, is generally used.[7–9] However, this process has the disadvantage of some difficulty in the handling of the molten aluminium. Thus, the development of other, more effective processes is needed for obtaining aluminized coatings.

In this work, we have obtained aluminide diffusion coatings on an Inconel 738 specimen at 800–1000°C using an $AlCl_3 + H_2$ gas mixture which was pre-heated at 800–1000°C for about 0·3 s using a graphite pre-heating chamber. The effect of reaction conditions on the formation of the aluminized layers was examined.

2 Experimental Procedure

The $AlCl_3$ gas was prepared *in situ* by the chlorination of aluminium metal by HCl gas at 330°C. The gas mixture of $AlCl_3 + H_2$ was then introduced into the pre-heating chamber prior to introduction into the reaction zone. The reaction chamber (Inconel 601, 350 mm internal diameter × 1·430 m long) was heated from the outside. Bare and Ni and Cr electro-plated Inconel 738 discs (25 mm diameter × 5 mm thick) were used as specimens for the coatings. The pre-heating chamber, which was made of graphite and had a 600 mm path length and 3280 cm^3 of total

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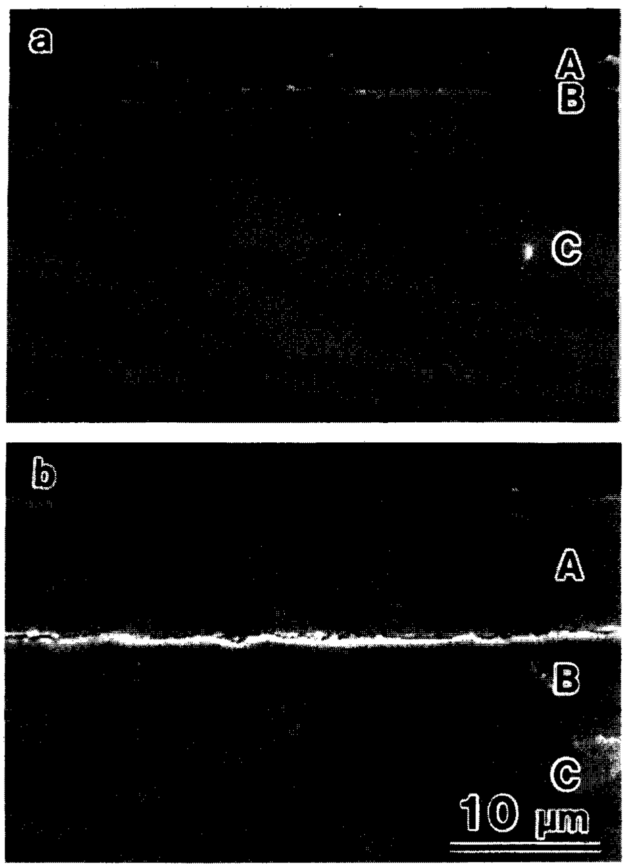

Fig. 1. Polished cross-section of aluminized Inconel 738; reaction temperature = 850°C, reaction pressure = 4 × 10^4 Pa. (a) Without pre-heating, (b) with pre-heating. (A) Outer NiAl layer, (B) inner diffusion layer, (C) specimen layer.

path volume, was set in the lower part of the reaction chamber. The experiment was also carried out without using the pre-heating chamber for reference.

3 Results and Discussion

The aluminized coatings were composed of two layers: an outer and an inner layer as shown in Fig. 1, subsequently analysed as NiAl and a more complex NiAl/Inconel interdiffusion zone, respectively. The total thicknesses of the coatings deposited with and without pre-heating were 14·2 and 3.0 μm, respectively. That is, the total thickness of the aluminized layer with pre-heating is 4·7 times larger than that without pre-heating. The effect of reaction temperature on the total thickness of the aluminized coatings obtained with and without pre-heating after a 4 h reaction time is shown in Fig. 2, in which the pre-heating temperature and time were fixed at 950–1000°C and 0·3 s, respectively. It was observed that the total thickness with pre-heating was 1·5–4·7 times larger than that without pre-heating. That is, the pre-heating treatment of the gas mixture of $AlCl_3$ + H_2 is very effective for obtaining thicker aluminized coatings. This effect was particularly marked at temperatures of 850°C and below. This effect may be caused by the effective formation of reactive AlCl gas species due to the pre-heating, while the holding time of the gas mixture of $AlCl_3$ + H_2 in the pre-heating chamber is very short, 0·3 s. The effects of reaction conditions with pre-heating on the formation of the aluminized coatings were then examined.

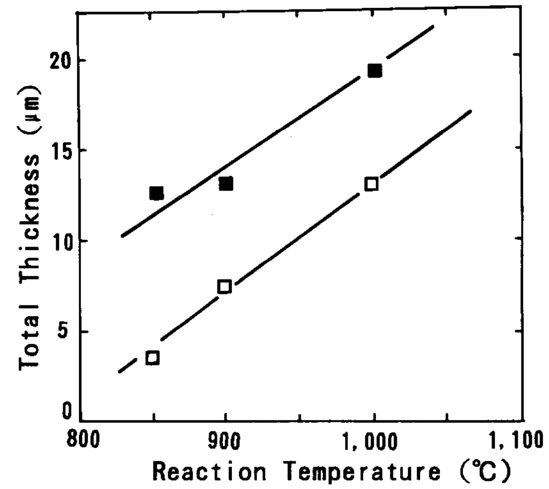

Fig. 2. Effect of reaction temperature on the total thickness of the aluminized layers; pre-heating temperature = 950–1000°C, reaction time = 4 h. (■) With pre-heating, (□) without pre-heating.

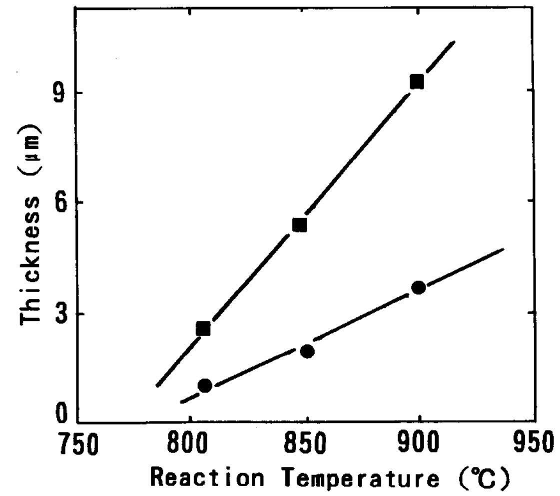

Fig. 3. Effect of reaction temperature on the layer thickness; pre-heating temperature = 950°C, reaction time = 4 h. (■) Outer NiAl layer, (●) inner diffusion layer.

The effect of reaction temperature on the thickness of the A and B layers in Fig. 1(b) is shown in Fig. 3, in which the pre-heating temperature was fixed at 950°C. Both outer and inner layer thicknesses increased linearly with increasing reaction temperature, and the thickness of the outer NiAl layer was about 1·6 times that of the inner diffusion layer.

The effect of $AlCl_3$ gas flow rate on the layer thickness obtained at the reaction temperature of 850°C is shown in Fig. 4. The thickness of the respective layers increased parabolically with increasing $AlCl_3$ gas flow rate. This result suggests that the rate-determining step is the hydrogen reduction of $AlCl_3$ to $AlCl_{3-x}$ and finally to Al

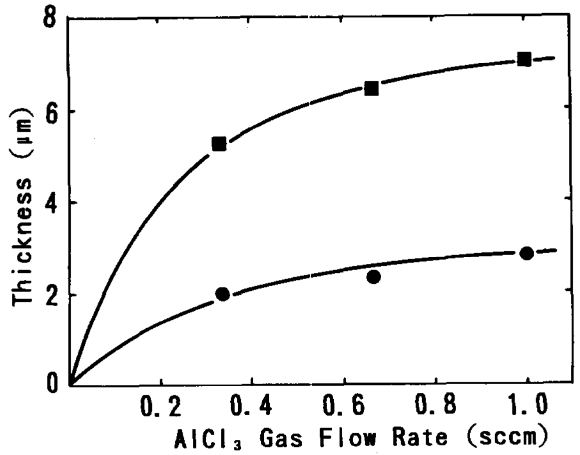

Fig. 4. Effect of $AlCl_3$ gas flow rate on the layer thickness; reaction temperature = 850°C, pre-heating temperature = 950°C. (■) Outer NiAl layer, (●) inner diffusion layer.

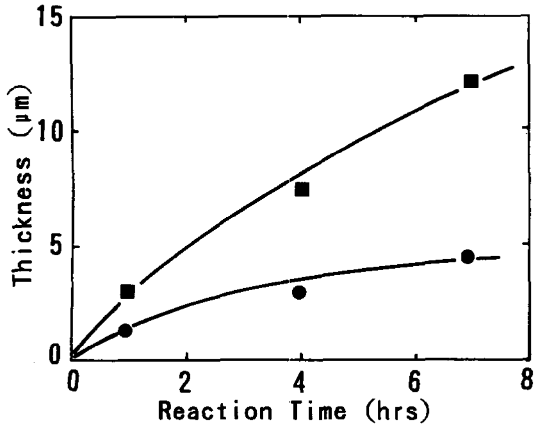

Fig. 5. Effect of reaction time on the layer thickness; reaction temperature = 850°C, pre-heating temperature = 950°C. (■) Outer NiAl layer, (●) inner diffusion layer.

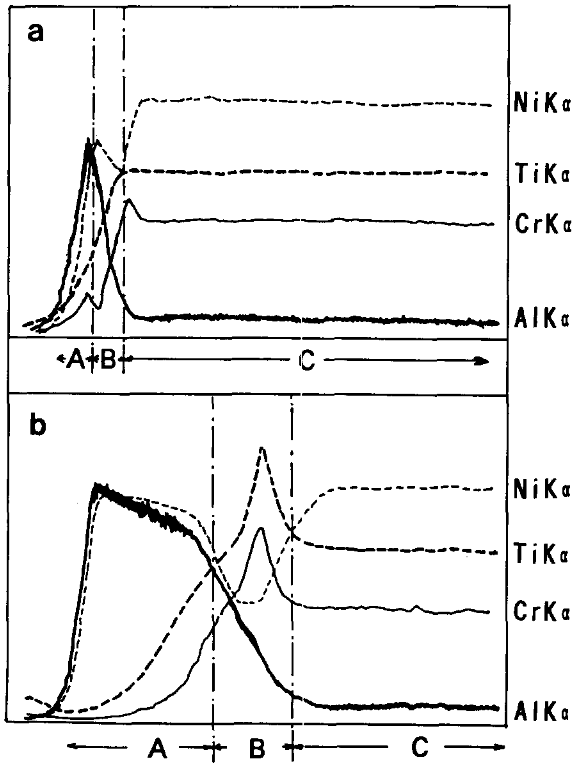

Fig. 6. EPMA images of the polished cross-sections of aluminized specimens. Reaction time: (a) 1 h, (b) 7 h. (A) Outer NiAl layer, (B) inner diffusion layer, (C) specimen layer. Full counts (cps) of the peaks: Ni K_α = 20000, Ti K_α = 2000, Cr K_α = 20000, Al K_α = 50000.

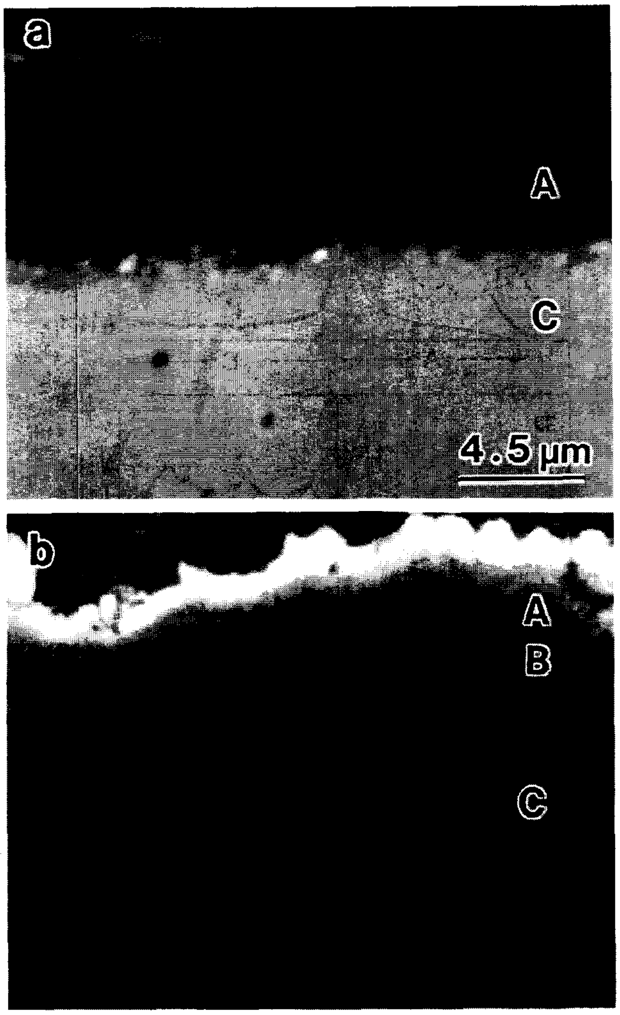

Fig. 7. Polished cross-sections of aluminized Inconel 738 electroplated with Ni and Cr; thickness of electroplated Ni or Cr = about 4 μm, reaction temperature = 350°C, pre-heating temperature = 950°C, reaction time = 4 h. (a) With Ni plating, (b) with Cr plating. (A) NiAl layer, (B) diffusion layer, (C) specimen layer.

metal, and/or the diffusion of Al species through the gas boundary layers present on the specimen surface. The effect of reaction time on the layer thickness is shown in Fig. 5, in which reaction temperature was fixed at 850°C. It was observed that the thickness of the respective layers increased parabolically with increasing reaction time, and a total thickness of about 12 μm was obtained after a 7 h reaction time.

Figure 6 shows the profiles of electron probe microanalysis (EPMA) of the polished cross-section of the aluminized specimen. Enrichment of Cr in the boundaries of the outer/inner aluminized layers and inner/substrate layers was observed during the initial aluminizing stage after 1 h in Fig. 6(a). Figure 6(b) shows that, with increasing reaction time to 7 h, Ti and Cr were significantly enriched in the thickened inner diffusion layer. Figures 6(a) and 6(b) confirm the formation of NiAl in the outer layer.

The polished cross-sections of specimens electroplated with ~4 μm thick Ni and Cr films, and

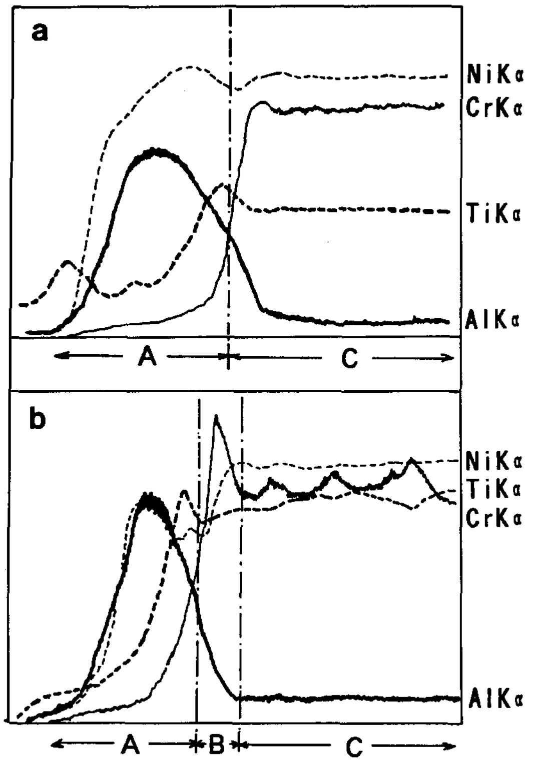

Fig. 8. EPMA images of the cross-sections shown in Fig. 7. (a) With Ni plating, (b) with Cr plating. (A) NiAl layer, (B) diffusion layer, (C) specimen layer. Refer to Fig. 6 for full counts (cps) of peaks.

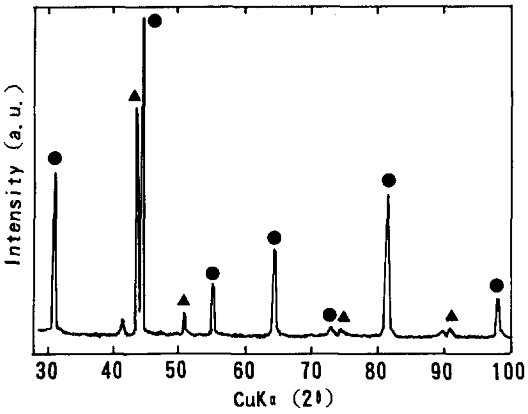

Fig. 9. XRD profiles of the surface of aluminized specimen with pre-heating. (●) NiAl, (▲) Ni_3Al.

then aluminized with pre-heating at 850°C for 4 h, are shown in Fig. 7. With the Ni plating in Fig.7(a), a single 5·6 μm thick aluminized layer only was obtained, because of preferential aluminizing of the electroplated Ni layers. On the other hand, with the Cr plating in Fig. 7(b), double layers with 6·5 μm total thickness were obtained. Figure 8 shows the corresponding EPMA traces of the cross-sections shown in Fig. 7. For the Ni plating in Fig. 8(a), Ti was enriched in the outer NiAl layer adjacent to the boundary, and similar Ti enrichment for the Cr plating is shown in Fig. 8(b). In both cases, Cr was not present in the outer NiAl layer, but was enriched in the inner diffusion layers.

Figure 9 shows the X-ray diffraction (XRD) profile of the surface of the aluminized layer for the Ni-plated specimen. It was observed that sharp peaks of Ni_3Al as well as NiAl were observed. The formation of the Ni_3Al phase is considered to be caused by the Ni-plating layers. On the other hand, a single NiAl phase only was observed for the Cr-plated specimen. Greater Ni enrichment in the outer layer for the Ni-plated specimen than for the non- or Cr-plated specimen may be caused by the formation of the Ni_3Al phase.

In conclusion, we have successfully obtained aluminide coatings on an Inconel 738 disc specimen at the relatively low temperature of 800–900°C using $AlCl_3 + H_2$ gas mixtures which were pre-heated at 950–1000°C for about 0·3 s in a graphite pre-heating chamber. This pre-heating process is very useful from the standpoint of being a simple, flexible and productive process.

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