



EFFECT OF ALUMINIZING-CHROMATE MULTI-DIFFUSION COATING ON MICROSTRUCTURES AND HIGH TEMPERATURE OXIDATION BEHAVIOR OF INCOLOY ALLOY 909 FOR GAS TURBINE ENGINE COMPONENTS

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ABSTRACT

Incoloy alloy 909 is an Fe-Ni-Co based superalloy that is attractive for gas turbine engine applications. The absence of chromium, however, makes the alloy more susceptible to oxidation in high temperature. To improve the oxidation resistance aluminizing-chromate multi-diffusion coating was performed by low temperature pack cementation process. Aluminizing was carried out at 552°C for 20hr. Chromate conversion coating was then produced by spray of the TCS562 solution and baking at 343°C. The composition and structure of the coated layer were analyzed using SEM, TEM, EPMA and XRD. Aluminized layer thickness was about 20 μ m and the layer was divided into two regions that is, a cover layer and a diffusion layer. High temperature oxidation experiments were performed in static air at 482, 538, 593 and 648°C for 7 days with weight measurements in the interval of 10min. Oxidation resistance was improved by aluminizing and aluminizing-chromate treatment. From the potentiodynamic polarization test results corrosion resistance of aluminizing-chromate treated specimen was the best.

Keywords: Incoloy alloy 909, Aluminizing, Chromate

1. INTRODUCTION

Incoloy alloy 909 is Fe-Ni-Co based superalloy and has high strength, constant low coefficient of thermal expansion and constant elastic modulus. It is attractive for aerospace and land based gas turbine engine applications [1]. Its thermal expansion rate is about half the rate of other alloys having comparable strength. The low thermal expansion rate enables closer control of clearances and tolerances for greater power output and fuel efficiency [1,2]. For these reasons, Incoloy alloy 909 is used for gas turbine vanes, casings, shafts and shrouds.

The alloy contains no chromium which increases the thermal expansion rate of alloys. The absence of chromium, however, makes the alloy more susceptible to oxidation in high temperature. Therefore, the alloy may require a protective coating to prevent excessive oxidation and corrosion. For that purpose, several coating techniques are used. Diffusion coating is one of them. It is the method to form aluminide or chromide layers and the advantages of it are low cost of process and mass productivity. Aluminizing process by diffusion coating can be divided into three, that is, slurry, pack cementation and vapor phase deposition methods. Pack cementation is diffusion coating process which is widely used to

deposit protective coating. [3,4]. The advantages are low cost, short production time and the formation of uniform diffusion coating layer, etc[5,6]. Even though, aluminizing treatment can improve high temperature oxidation resistance, it is not enough to further improve corrosion resistance. Therefore, for the additional corrosion resistance, chromate treatment can be applied. Mechanical properties of Incoloy alloy 909 decrease rapidly over 650°C[1]. Therefore, protective coating process must be performed at lower temperature. In this study, aluminizing-chromate multi-diffusion coating was performed to improve the oxidation and corrosion resistance of Incoloy alloy 909. The treatments were carried out below 552°C to prevent from drastic decrease of the mechanical properties. Microstructural modification due to element deposition by aluminizing and chromate process was investigated and high temperature oxidation and corrosion resistance were measured.

2. EXPERIMENTAL PROCEDURES

A substrate was Incoloy alloy 909, and the chemical composition is given in Table 1. The specimens were cut to the size of 25×25×10 mm³. The slabs were polished and immersed in the pack of powder mixture for aluminizing. The pack consisted of 1wt.% NH₄Cl halide salt activator (98.5% pure), 80wt.% Al (99.0% pure, -325mesh) and 19wt.% Al₂O₃ power filler (99.6% pure, -325 mesh). The specimens were cleaned by sand blast and sealed in a container with the powder mixture. They were heated to the temperature of 468, 496, 524 and 552°C and held for 10, 20, 30 and 40hours under Ar atmosphere. The specimen which shows the best coating layer was selected for further chromate treatment. For the chromate treatment after aluminizing, the specimen was thermally cleaned at 343°C for 1 hour. After cleaning, TCS562 solution was sprayed to the surface of the specimen. Then, it was baked at 343°C for 1 hour.

The aluminized, aluminizing-chromate multi-diffusion coating layer and the substrate were analyzed with XRD, EPMA, SEM and TEM. High temperature oxidation test was performed in static air at 482, 538, 593 and 648°C for 7days. The weight change was measured in ten minute interval. Potentiodynamic polarization curves were obtained at a scan rate of 2mA/s and potential range of -0.5V~1.5V in 0.2mol H₂SO₄ solution. The working electrode used was cut in the form of disc from each specimen and has area of 1cm². A Ag/AgCl electrode and a graphite electrode were used as reference and counter electrodes respectively.

3. RESULTS AND DISCUSSION

3.1 Microstructures

The influence of temperature and time on aluminizing was examined by measuring the thickness of the aluminide layer. As shown in Fig. 1, although the thickness of the layer initially increased rapidly, increasing rate was decreased with time. There are three possible reasons for this slow-down: Firstly, the aluminized layer formed on the surface suppresses the diffusion of Al. Secondly, since the aluminized layer has been formed partially by a reductive reaction of active AlCl₃(g), active Cl decreased with time. Thirdly, the amount of active reagent (NH₄Cl) decreased by thermal decomposition[7]. From the results, the best combination of the temperature and time for aluminizing was 552°C and 20 hours, and in that case the thickness of aluminide layer was about 20µm. Fig. 2 shows the cross-sectional microstructure and EPMA line profiles of aluminized specimen for 20hr at 552°C. Aluminide layer was divided into two regions, that is, a cover layer and a diffusion layer. It was confirmed by the micrograph and EPMA line profiles.

Due to TEM observation, it was known that the substrate consisted of the matrix, fine γ' particles, ϵ -type precipitates and Laves precipitates revealed in the grain boundary (Fig. 3). EDX analysis showed that γ' particles contained Ni, Fe, Co, and Ti. Its crystal structure was A₃B

type, that is $[(\text{Ni}, \text{Fe})_3(\text{Ti}, \text{Nb})]$. The size was about 10~30nm and the shape was spheroidal or cuboidal.

The ϵ -type precipitates which were acicular and showed Widmanstätten structure, were distributed homogeneously in the matrix(Fig. 3a). The ϵ -type precipitates which was acicular and shows Widmanstätten structure, were distributed homogeneously in the matrix(Fig. 3a). Its length was 400~500nm and the width was about 50nm. It included the same elements to the γ' phase(Ni, Fe, Co and Ti) and small amount of additional Si. The precipitates appeared near the grain boundary shown in Fig. 4 were large Laves phase which had a polygonal shape. And, its size was about 500nm. From the EDX analysis, main elements were Nb, Ni and Fe. In the Al coating layer, the form and composition were progressively changed from the surface to the interface by diffusion. Near surface area showed fine texture where the size was about 50nm. And, between the textures, many fine precipitates which were 20~25nm in size and where small amount of Ti was distributed in the Nb-rich phase were observed(Fig. 5a). From the EDX analysis, small amount of Fe, Ni, Co and Nb were detected. Also, the fine precipitates which were shown between the textures were Nb-rich phases and distinguished in Fig. 5b (bright field image). The size of the precipitates was too small so that it was not possible to know the exact composition. However, when compared to the EDX analysis from the Fig. 5a, Nb and Ti contents were relatively high. The diameters of the precipitates were about 5~15nm and much smaller than that of the matrix. From the SADP, NbFe, Nb₂Fe and NbNi phases might be mixed together.

On the other hand, near matrix area of the layer had the mixture of fine and coarse textures(Fig. 6). The size of the coarse texture was in the range of 500nm to several microns. The main elements were analysed to be Al and Ni by EDX. From the SADP analysis, the crystal structure was AlNi(B2) and its lattice constant was 2.93~2.99Å. Fig. 7 showed microstructures of the specimen after the aluminizing-chromate multi-diffusion coating and EDX line profiles of them. As shown in the figure, chromate layer was formed in the thickness of 5μ. Main elements of the layer were P, Mg and Cr.

3.2 High temperature oxidation

High temperature oxidation test was performed with non-treated, aluminized, and aluminizing-chromate specimens for 168 hours(7days) at 482°C, 538°C, 593°C and 648°C. Fig. 8 shows weight change of the non-treated specimen after the test at each temperature. In the case of Incoloy 909 alloy, because Cr is not included, high temperature oxidation resistance is very weak and the use above 650°C is known to be impossible[1]. It was well confirmed from the high temperature oxidation test of non-treated specimens shown in Fig. 8. As temperature increased, weight per unit area increased. However, weight change was small up to 593°C while it was large at 648°C. It means that Fe oxides reduce oxidation rate up to 593°C. However, at 648°C, protection to the oxidation by the Fe oxides was maintained for 15 hours and disappeared quickly after that.

Fig. 9 is EDX analysis result after high temperature oxidation test of the non-treated specimen. As shown in the figure, an oxide layer and an alloy depletion layer were clearly distinguished. In the oxide layer main phase was $\text{Fe}_{x-1}\text{O}_x$ oxide and other oxides such as CoO, and CoFe_2O_4 were co-existed. The oxide layer was formed by reaction of Fe from matrix with O_2 in that alloy depletion layer was generated subsequently. In addition, because of the migration of Fe to the surface Ni in the matrix was diffused to the alloy depleted layer.

Fig. 10 shows weight change of the aluminized specimen after the high temperature oxidation test. There was no big change. This seemed to be because stable Al_2O_3 oxide was formed on the surface and increased oxidation resistance. Through SEM micrograph and EDS

line profiles shown in Fig. 11. Al oxide was formed on the outer surface and alloy depletion layer appeared beneath the oxide layer. Fig. 12 shows weight change of the aluminizing-chromate specimen after high temperature oxidation test. Weight change at 593°C was not large. However, at 648°C, weight was increased up to 130 hours and maintained nearly constant after that. As temperature increased XRD peaks of AlFe, Al₃Nb, and Al₃Co₄ decreased or disappeared while Cr₂O₃, (Cr, Fe)₂O₃, FeCr₂O₄, CrO₂ peaks were increased.

Fig. 13 shows EDX result after high temperature oxidation test of the aluminizing-chromate specimen. Cr oxide layer was formed on the surface and Al oxide layer was shown beneath the layer. Those layers seem to increase the oxidation resistance.

3.3 Potentiodynamic polarization test

The result of potentiodynamic polarization curves of non-treated, aluminized and aluminizing-chromate multi-diffusion coated specimens are shown in Fig. 14. In cathodic polarization behavior, the non-treated specimen showed high current density and aluminizing-chromate treatment specimen showed lowest current density. Corrosion potential was the highest in the aluminized specimen and was similar in the non-treated and aluminizing-chromate treated specimen. Corrosion current density of non-treated specimen and aluminizing-chromate specimen looked similar and the aluminized specimen was the highest. All specimens were transited to passive state at about -0.4V of corrosion potential but aluminized specimen was corroded rapidly at about 0.1V. The non-treated and the aluminizing-chromate specimens showed similar corrosion behavior but the aluminizing-chromate specimen showed lower current density. Finally, through aluminizing-chromate multi-diffusion coating process, corrosion resistance was increased further.

4. CONCLUSIONS

Aluminizing-Chromate multi-diffusion coating was performed to improve the oxidation and corrosion resistance of Incoloy alloy 909 and high temperature oxidation test and potentiodynamic polarization test were carried out. From the results following conclusions can be drawn.

- 1) The substrate consisted of matrix, fine γ' particles, acicular ε -type precipitates and Nb and Ti rich precipitates.
- 2) Aluminide layer consisted of a cover layer and a diffusion layer. The cover layer consisted of textures, fine Nb and Ti rich precipitates and the diffusion layer consisted of textures and AlNi of B2 structure.
- 3) From the results of high temperature oxidation test, oxidation resistance was improved by aluminizing and aluminizing-chromate treatment in every test temperatures
- 4) From the potentiodynamic polarization test results corrosion potential was constant in the non-treated, aluminized and aluminizing-chromate specimens. Corrosion rate of aluminized specimen was faster than that of the others and corrosion resistance of aluminizing-chromate multi-diffusion coating specimens was better than the others.

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FIGURES

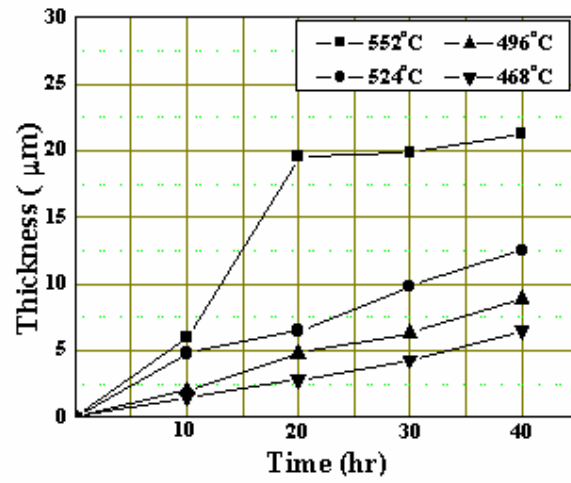


Fig. 1 Thicknesses of aluminide layer plotted as a function of temperatures.

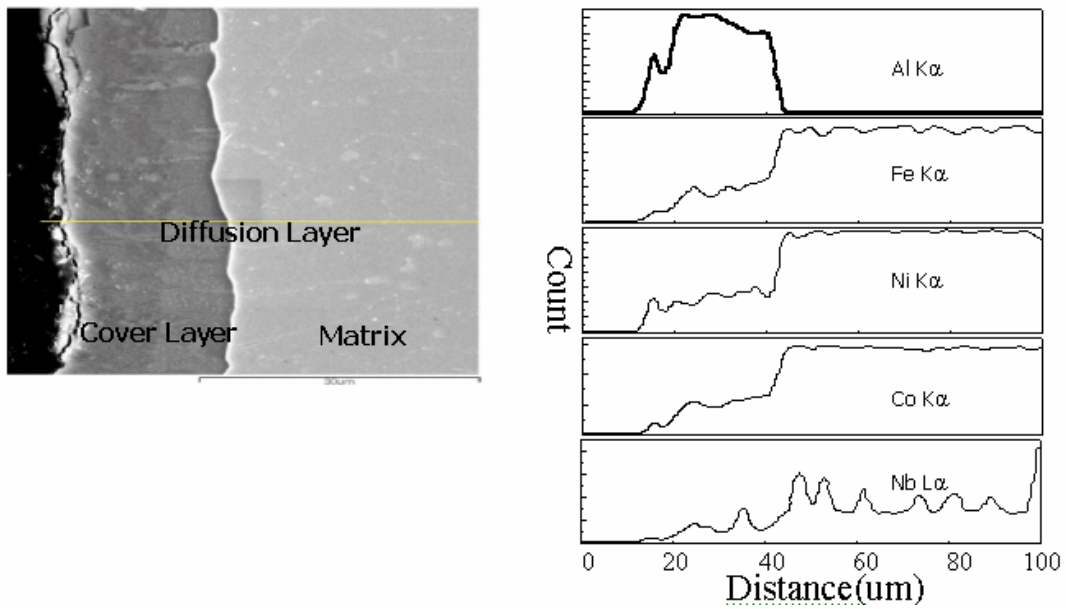


Fig. 2 SEM micrograph and EPMA line profiles of aluminized specimen for 20hr at 552°C.

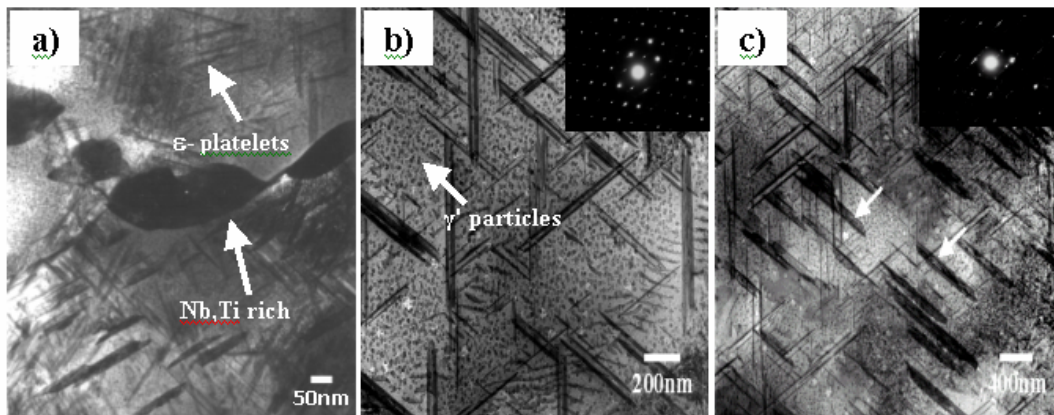


Fig. 3 TEM micrographs of substrate a) matrix, ϵ platelets, Nb and Ti rich precipitates, b) matrix, γ' particles and ϵ precipitates, matrix SADP c) coarsened ϵ precipitates (arrow).

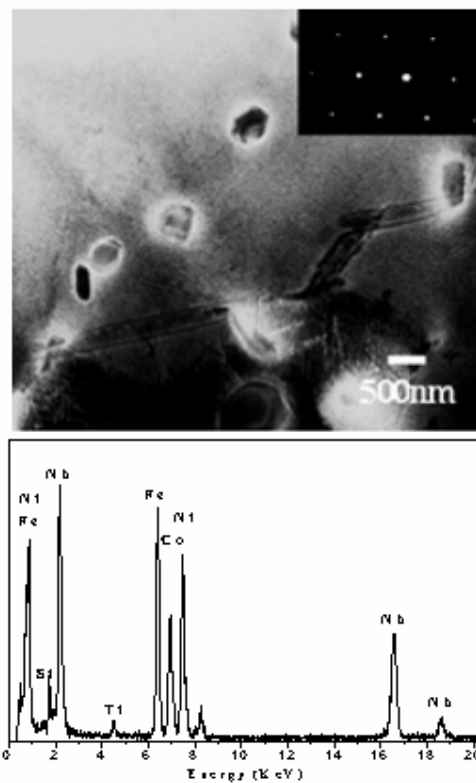


Fig. 4 TEM micrograph, SADP and EDX spectrum of grain boundary precipitates

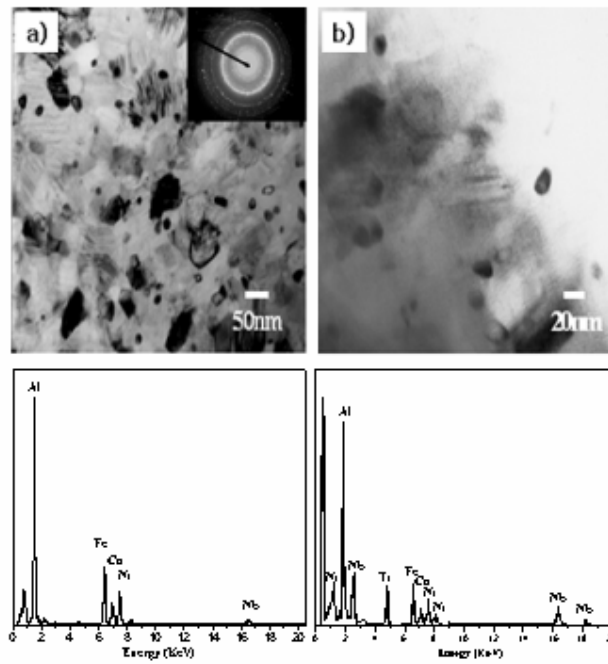


Fig. 5 TEM micrographs, SADP and EDX spectrum of coating layer (a)cover layer, (b) BF image of fine precipitates of the cover layer.

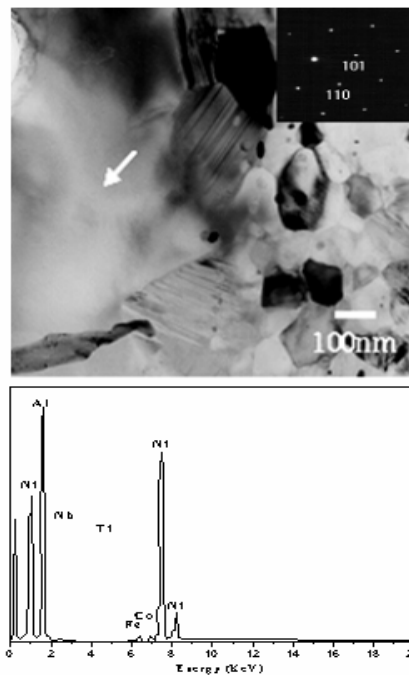


Fig. 6 TEM micrographs, SADP and EDX spectrum of diffusion layer.

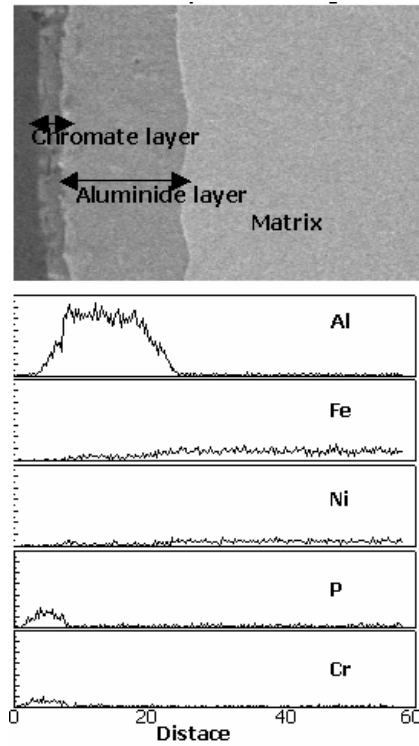


Fig. 7 SEM Micrograph and EDX line profile of aluminizing-chromate multi-diffusion coating specimen.

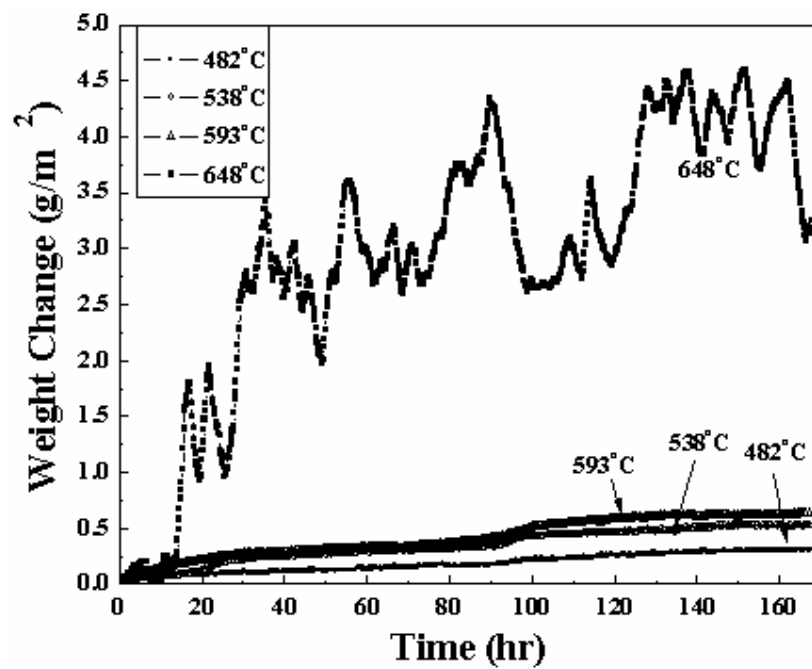


Fig. 8 High temperature oxidation curves for non-treated specimen.

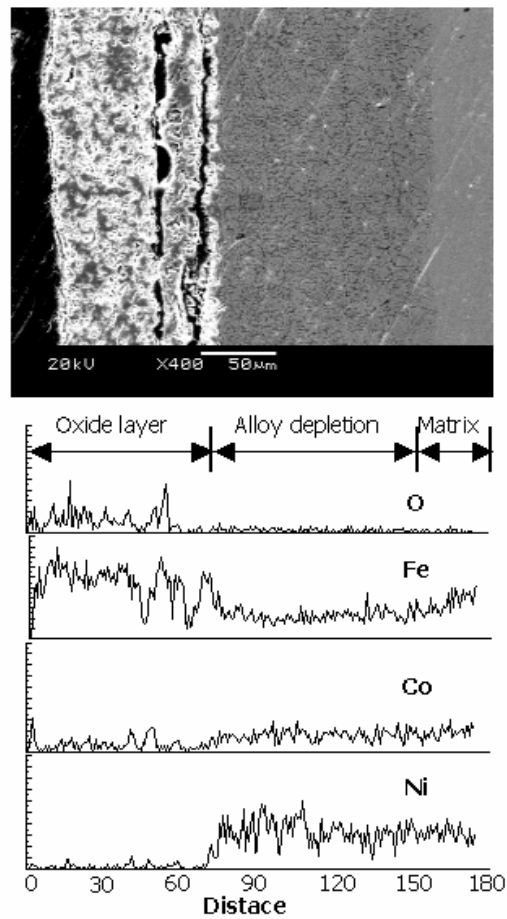


Fig. 9 SEM micrograph and EDX line profiles of oxide layer in the non-treated specimen.

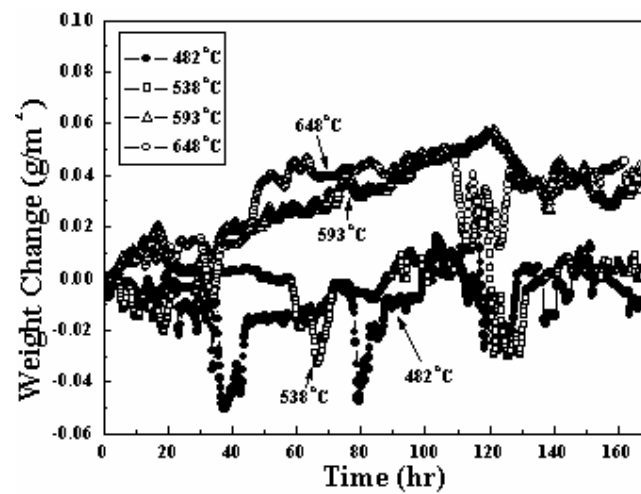


Fig. 10 High temperature oxidation curves for aluminized specimen.

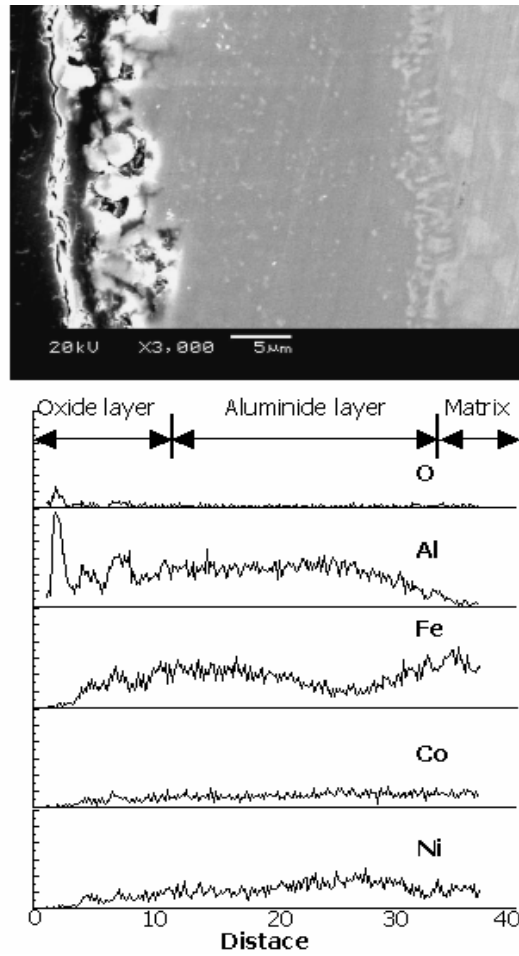


Fig. 11 SEM micrograph and EDX line profiles of oxide layer in the aluminized specimen.

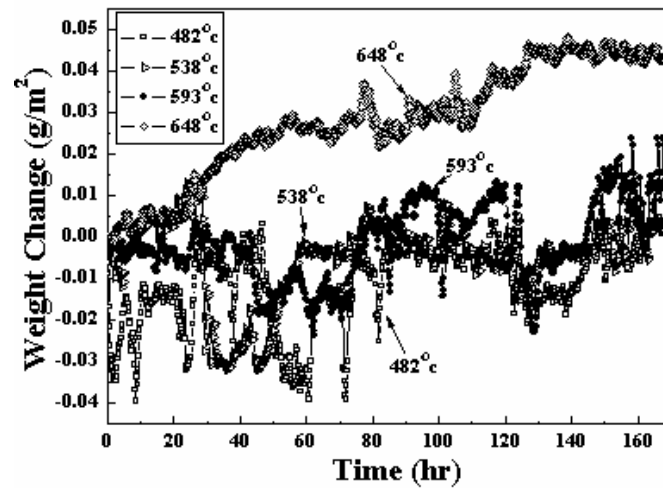


Fig. 12 High temperature oxidation curves for aluminizing-chromate specimen.

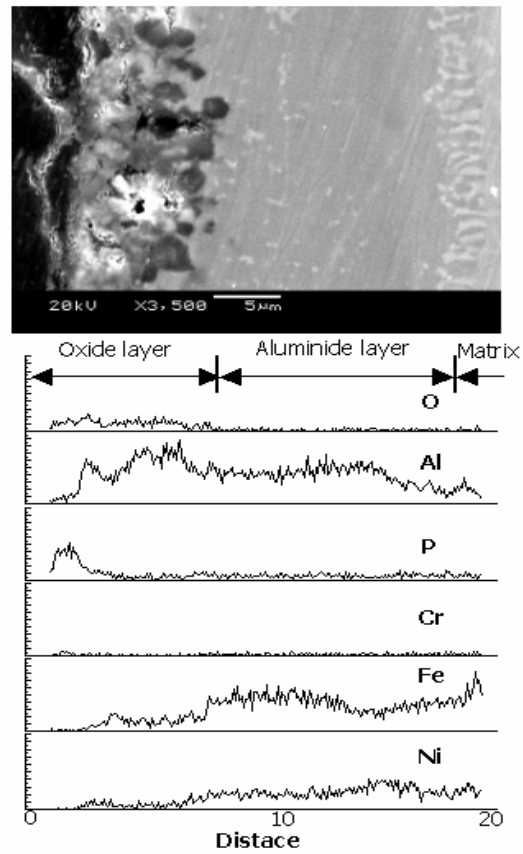


Fig. 13 SEM micrograph and EDS line profiles of oxide layer.

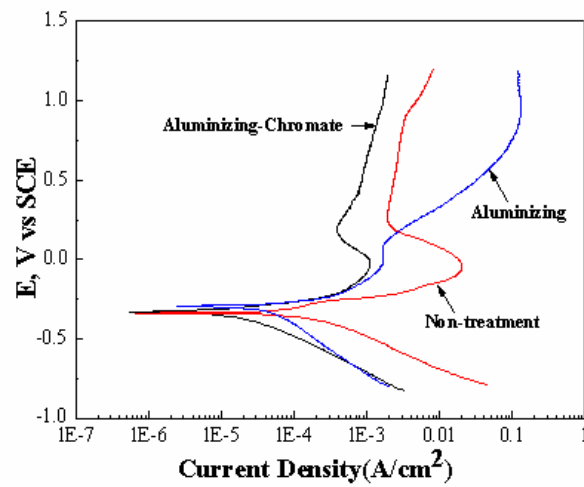


Fig. 14 Polarization curves in 0.2mol H₂SO₄ solution of non- treated, aluminized and aluminizing-chromate treated specimens.