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October 1982

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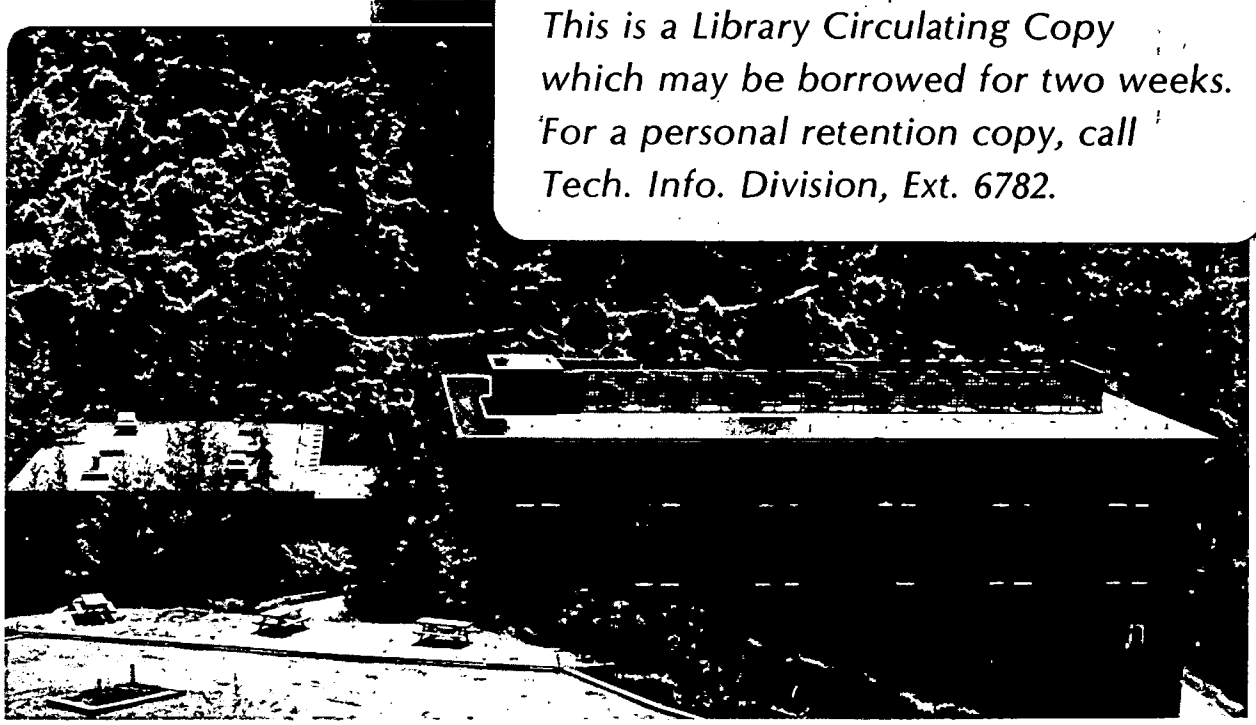

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KINETICS AND MORPHOLOGY OF THE OXIDE GROWTH BY OXIDATION OF NiAl COATINGS ON NICKEL

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KINETICS AND MORPHOLOGY OF THE OXIDE GROWTH BY OXIDATION OF NiAl COATINGS ON NICKEL

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SUMMARY

The oxidation of NiAl coatings on unalloyed nickel has been investigated with respect to the NiAl composition. The two types of NiAl coatings, i.e. Ni-rich and Al-rich NiAl, were prepared by pack cementation. The kinetics of oxidation were determined in the temperature range 750/1000°C. The oxidized surfaces develop in a textured structure along with, in case of the Al-rich NiAl coatings, a thicker alumina scale which can spall off leading to irregular kinetics. In Ni-rich NiAl coatings a martensitic transformation takes place due to aluminium depletion, when oxidized above 980°C.

INTRODUCTION

Although aluminide coatings are commonly used to protect Ni-base superalloys against high temperature oxidation and corrosion in gas turbine construction, there was no exhaustive study published on the kinetics of growth and the morphology of oxides scales formed on NiAl coatings, and only a few on NiAl bulk alloy oxidation. The most extensive studies on oxidization of NiAl alloys have been published during the last four years and concern: oxide growth and microstructure (1), oxide morphology and spallation (2), mechanism of oxide spallation (3), and compositional dependence of oxidation rate (4). However, the first two deal with only one NiAl alloy composition, and the latter refers to only one temperature, viz. 900°C.

The purpose of this study was to undertake a comprehensive investigation of the oxidation behavior of NiAl type coatings with respect to their structures and compositions. The preparation of coatings with an external part of either a Ni-rich or an Al-rich phase has been perfected, which enabled the study of the oxidation of these two types of coatings.

The kinetics of the oxidation, the morphology of the oxidized coatings and the surface morphology of the oxide scale formed on the coatings have been investigated at temperatures ranging from 750 to 1000°C in pure oxygen. As the aim of this study was to investigate the kinetics of the oxidation reaction and the related oxide

morphology, it does not deal with long term oxidation resistance or cyclic oxidation. Consequently, the oxidation durations are, for the most, limited to about 20 to 25 hours.

COATING PREPARATION AND INVESTIGATION TECHNIQUES

NiAl coatings were prepared by a typical halide activated pack cementation process.

Polished and degreased nickel specimens cut in 10x10 mm plates were encased in a mixture of powders, the pack, placed in an alumina retort and heated to the cementation temperature. The pack is constituted of the aluminum to be deposited, nickel to adjust the aluminum activity, alumina as inert filler to prevent sintering and chromium bifluoride as an activator. At the treatment temperature, chemical transport is established by the activator giving rise to a volatile aluminum subhalide, which decomposes at the surface of the specimen with deposition of the aluminum in the form of the coating and releases the activator to promote further gaseous compound formation and material transport. The cementation procedure and the pack preparation used in this study have been formerly described elsewhere (5) and will not be detailed further.

According to the diffusion growth mechanism, two types of coatings formed in the pack cementation process may be distinguished, following the classification of GOWARD and BOONE (6):

1. - coatings formed by predominantly inward diffusion of aluminum from the surface, where Ni_2Al_3 or Al-rich non-stoichiometric NiAl are the main constituting phases. These are formed in pure Al or high aluminum activity packs. The processes using these pack compositions are usually designated as "high activity processes"

2. - coatings formed by predominantly outward diffusion of nickel from the substrate where Ni-rich NiAl is the main coating phase. These are formed in Al-Ni alloys or low aluminum activity packs. The corresponding processes are called "low activity processes"

In a previous investigation (7) we have determined the nature and the composition of the constituting phases of NiAl type coatings with relation to the aluminum activity in packs of varying aluminum compositions.

The NiAl phase extends on both sides of the stoichiometric composition with large departures from stoichiometry. The NiAl composition corresponds to the CsCl-type of structure with the aluminum atoms on the cube corners and the nickel atoms at the cube center. For an excess of Ni in the nickel-rich NiAl, aluminum atoms are replaced substitutionally by smaller nickel atoms. On the other

hand, in the Al-rich NiAl lattice vacancies exist at the nickel sites with aluminum sites remaining fully occupied. A great difference in the metallographic structure results from this difference in the crystallographic structure, and an appropriate etching procedure revealed the separate layers corresponding to the two non-stoichiometric NiAl compounds, which behave like two metallographic distinct phases.

Cementation was carried out by using a series of packs containing Al-Ni alloys ranging from 47 at. pct. Al to 100 percent. The nature of the phases constituting the coatings was revealed by etching. They were identified by microprobe analysis, and the surface composition determined by X-ray diffraction, with the help of the relationship between the lattice parameter and the Ni composition published by TAYLOR and DOYLE (8).

After 20 hours cementation, at 1000°C, the two following NiAl coating structures were observed depending on two domains, with a sharp limit, of pack compositions. In packs containing less than 60 at. pct. Al, a thick layer of Ni-rich NiAl forms over a thin Ni_3Al, underlayer (Fig. 1-a). These coatings exhibit numerous alumina particles from the pack embedded in the surface or throughout the whole coating as a consequence of its growth by nickel outward diffusion. In packs containing from 60 to 100 at. pct. Al, the coatings present a duplex structure with an outer layer of inward Al-rich NiAl over an equally thick inner layer of Ni-rich NiAl (Fig. 1-b). When appropriately etched, these coatings also show a thin Ni_3Al underlayer. The surface composition of the Al-rich phase in the duplex coating has been found invariant from the pack composition (Fig. 3). This has not yet been explained in terms of the actual mechanisms proposed for the pack cementation formation of aluminide coatings on nickel.

According to these conditions of formation of NiAl coatings, three series of coatings were prepared for the oxidation tests:

1° - Ni-rich outward coatings, 60 µm thick, formed by 40 hours cementation at 1000°C, in a "low activity" pack containing 60 at. pct. Al in an Al-Ni power mixture (Fig. 1-a type coatings)

2° - thick Ni-rich NiAl coatings, 40 µm thick, formed in the same pack by 20 hours cementation, partially covered by thin plates of Al-rich NiAl resulting from the incomplete interdiffusion of an initial Al-rich surface layer (Fig. 2)

3° - duplex inward coatings, with an external part of Al-rich NiAl over an inner Ni-rich layer, with a total thickness of about 190µm, formed by 20 hours cementation at 1000°C in an "high activity" pack containing 75 at. pct. Al. (Fig. 1-b type coatings)

The surface composition of the Ni-rich NiAl coating is 41 at. pct. Al, and 54.4 at. pct. Al for the Al-rich outer part of the duplex coating.

OXIDATION OF THE COATINGS; RESULTS AND DISCUSSION

Oxidation was performed in pure static oxygen at atmospheric pressure, in the temperature range 750 to 1000°C. The kinetics were followed by isothermal thermogravimetric recording during oxidation runs of approximately 12 hours.

1-OXIDATION KINETICS

Ni-rich NiAl coatings

Weight gains are very small, presuming a very thin oxide film is formed. The kinetics show an erratic initial behavior, roughly parabolic, with a series of unexpected weight losses before approaching an intermediate plateau, with a practically zero oxidation rate (Fig. 4). The irregularities of the weight change recording may be ascribed to the loss of alumina particles from the pack, remaining stuck to the coating surface due to the outward mechanism of the coating growth, which become loose because of oxidation of the subjacent aluminide. Figure 9-c shows the surface of a Ni-rich NiAl coating specimen before oxidation with numerous particles stuck to it. In Fig. 9-b, on the surface of an identical specimen after oxidation, the mark of a particle that detached after oxidation of the surrounding aluminide has been pointed out.

After the plateau the reaction starts again and the kinetics though still irregular follow an approximately parabolic law without acceleration during at least 100 hours, as in an oxidation run which has been performed with this duration at 740°C.

Al-rich NiAl coatings

Kinetics of the oxidation of thick Al-rich coatings, or of Al-rich plates partially covered Ni-rich NiAl coatings, follow a rather regular parabolic law for a period of 5 to 10 hours depending on the temperature and the specimen (Figs. 5 and 6). The weight gains are higher than in case of oxidation of Ni-rich NiAl coatings, presuming a thicker oxide scale is formed. After this period of parabolic oxidation, the regularity and the reproducibility of the oxidation of Al-rich coatings fail and the kinetics become irregular with fluctuations around a mean regime, with a rate decreasing with time, and diverge from one specimen to another, as shown e.g., in Fig. 7 at 1000°C. The irregular kinetics behavior of the latter stage of the oxidation of this type of coatings will be ascribed to the desquamation and spallation of the oxide scale as observed in Fig. 13.

In the case of the Al-rich NiAl coatings, this irregular kinetics period lasts without appreciable acceleration as long as the oxidation runs.

On the contrary the kinetics of oxidation of Ni-rich NiAl partially Al-rich covered coatings, show drastic acceleration after the first period of parabolic behavior (Fig. 5). As the characteristics of the parabolic periods are very comparable, one may assume that in case of these coatings the parabolic oxidation corresponds to the total oxidation of the plates of the Al-rich phase. The acceleration of the reaction is due to a strong intergranular oxidation of the subjacent Ni-rich NiAl coating (Fig. 10).

A parabolic plot of the oxidation kinetics of Al-rich duplex coatings shows that the kinetics follows a parabolic law after a short preparabolic period (Fig. 8-a). According to the assumptions of HINDAM & SMELTZER (1) this preparabolic period is ascribed to the formation and the recrystallization of an initial sub-microscrystalline oxide film, which recrystallizes during its growth to a textured α-Al_2O_3 film. Activation energy for the parabolic kinetics in the Arrhenius representation in Fig. 8-b, is equal to 38.8 Kcal. $mole^{-1}$, which seems small in comparison with the available data on aluminum or oxygen diffusion in alumina, but is in agreement with the value determined for the growth of alumina on a NiAl bulk alloy by the former authors, viz. 31.8 Kcal. $mole^{-1}$.

2-MORPHOLOGY OF THE OXIDIZED COATINGS

The morphology of the coatings after oxidation was investigated on etched cross-sections of oxidized specimens.

Ni-rich NiAl coatings remain compact, i.e. protective at all the investigated temperatures (Fig. 10-a). Only surface oxidation occurs giving rise to a thin oxide film, according to the small weight gain recorded in the kinetics investigations, and too thin to be observed on these cross-sections. A little oxidation takes place at the grain boundaries (oxide traces in black on the O.M. photo in Fig. 10-a) but does not develop into catastrophic or pest oxidation, as seen in the 100 hours run at 740°C. In specimens oxidized at 980°C some striation appearing near the NiAl/Ni_3Al interface indicates the occurence of a martensite transformation (Fig. 10-a).

Specimens of Ni-rich NiAl coating partially covered by Al-rich plates, oxidized at 980°C, exhibit strongly martensitic facies beneath the oxide interface or throughout the whole coating (Figs. 10-b; 10-c). On these coatings the alumina scale appears thicker, in accordance with the higher weight gain, and discontinous, corresponding to the oxidation of the partially covering Al-rich plates and to the thick partial oxide scale observed on these surfaces, as shown in Fig. 13-a. Most of these specimens exhibit

heavy intergranular oxidation. The coating grain boundaries are deeply attacked and the oxygen penetrates up to the NiAl/Ni_3Al interface (Fig. 10-b) (oxide appears in bright white under polarized light on the O.M. photo in Fig. 10-b). This intergranular oxidation may be induced by the formation of the thicker alumina scale on the Al-rich plates and involves the acceleration observed in the kinetics after the parabolic period. Martensite transformation should occur in NiAl with an Al composition less than 37 at. pct., when quenched from temperatures higher than 950°C (9-10). The impoverishment of these NiAl coatings in aluminum by both surface oxidation to form alumina and interdiffusion with nickel from the substrate at the NiAl/Ni_3Al interface, leads to an aluminum depleted coating with a lower Al content than the initial composition (41 at. pct. Al) which may allow the martensite to form. Microprobe analysis of the specimen from Fig. 10-c has shown a flat aluminum concentration gradient lying just below 37 atom percent (Fig. 11). These specimens being thinner than the former Ni-rich NiAl coatings, the depletion of aluminum is more important, which may account for the lowering of a uniform Al concentration and the occurrence of the martensitic precipitation throughout the whole coating.

The martensitic transformation involves a volume change which may cause the degradation of Ni-rich NiAl coatings during cooling and reheating of coated turbines blades, and therefore limit their utilization as protective coatings (11).

Al-rich NiAl coatings exhibit, after oxidation, a relatively thick and continous alumina scale (Fig. 12). The attack of the coating is quite uniform, limited to a surface oxide with a small consumption of the outer Al-rich layer and no detectable oxygen penetration.

3-MORPHOLOGY OF THE OXIDE SURFACES

Ni-rich NiAl coatings

The oxidized coatings develop a bare alveole textured surface over the whole specimen and at all temperatures (Figs. 9-a; 9-b). These surfaces are dotted with the alumina particles from the pack powder which are either stuck to, or embedded in the coating as a result of the outward nickel diffusion in the "low activity" cementation process (Fig. 9-c). Alumina starts to grow along the particles embedded in the coating surface which act like structural defects in the textured formed film, and develops in an extended discontinous scale after the reaction has started again after the plateau in the kinetics.

Al-rich NiAl coatings

The oxidation of the Al-rich NiAl phase, i.e. either Al-rich plates partially covering Ni-rich NiAl or thick external Al-rich NiAl

coatings, brings about the occurrence of two coexisting oxide film regions, viz. those of the bare alveole textured surface along with those of a thick oxide scale, white and friable (Fig. 13). The thick oxide appears to spall off and the presence of spall debris suggests that this oxide has covered part of the alveole surface (Fig. 13-a).

The nature of the alveole surface was the subject of a special investigation. As it forms barely by oxidation of the Ni-rich NiAl coating it should be due to the development of a very thin oxide film, according to the small weight gain recorded in the kinetics investigations. This textured surface resembles the oxide lace structure observed by SMIALEK (2) due to reoxidation of the NiAl alloy free surfaces, after spallation of a primarily formed oxide scale. The oxidation of Al-rich NiAl coatings shows (Fig. 13-b) that the alveole ridge spacing varies with the subjacent aluminide grains bringing about an epitaxial relationship of the lace texture with the coating surface which is reminiscent of a thin epitaxial oxide film. This is comparable with the delineation between ridge spacings of the oxide film in the vicinity of an alloy grain boundary reported by HINDAM & SMELTZER (1) for the oxide formed on a NiAl bulk alloy. The fact that the alveoles from this textured surface do not show (Fig. 15-b) any crystallographic facetting, like the voids observed after spallation of the oxide scale in some previous studies (1, 2, 3), confirms that it is an oxide film with a lace structure.

The presence of oxygen in the textured surface has been confirmed by AUGER scanning microscopy. Two regions of a typical oxidized coating surface (Fig. 15-a) have been investigated by scanning and AUGER spectroscopy. The AUGER electron spectra of oxygen indicate the presence of oxygen in both regions (Fig. 16-b). Neverheless the peak intensity, i.e. the oxygen concentration at the surface, is smaller for the alveolate surface. This may be due to a different surface composition, which is in agreement with a small nickel peak also observed in that spectrum. On the other hand the AUGER scanning micrograph of the investigated surface area shows (Fig. 16-a) that the thicker alumina scale is, in some places, not fully opaque to the Auger electrons and reveals that the textured film develops beneath the alumina scale. An AUGER microprobe investigation is intended in order to determine the actual composition of the textured film and to check its occurrence beneath the thicker oxide scale. HUTCHINGS & et al. (3) have, indeed, shown by AUGER microprobe analysis that the concentration profile exhibits two layers in the oxide scale formed by oxidation of a NiAl bulk alloy: an outer layer of Al_2O_3 depleted in nickel and an inner layer, of about 100 Å, with a greatly increased nickel content. But they did not relate this with the existence of two different structures in the oxide layer.

CONCLUSION: MODEL FOR THE OXIDE GROWTH

α-alumina was the only oxide to be identified by X-ray analysis on the oxidized coating surface after 20 hours or more

oxidation. This is in accordance with the thermodynamic considerations of PETTIT (12) and the results of previous investigations on bulk alloy oxidation (1; 2).

Surface examination, using SEM, of the thick oxide regions on Al-rich NiAl oxidized coatings, shows, at temperatures from 900°C and above, the growth over the thick scale of inflorescences of whiskers in which protusions with crystallographic features develop (Fig. 14-a). At higher temperatures the oxide scale is constituted of a "moquette" of whiskers, which have developed completely covering the oxide scale (fig. 14-b). The oxide whiskers and these protusions grow by metal diffusion or by vapor transport of aluminum from Al-rich NiAl, which supply is ensured by aluminum diffusing outwardly through the thick oxide scale. Observation of imprints of the coating grain boundaries on the surface of the alumina scale (Fig. 13-c) reinforces the assumption of oxide growth by outward diffusion of aluminum.

The growth of the alveoles of the lace oxide on the textured surface suggests the preferential grain boundary diffusion of at least one species. It has been established (13) that in polycrystalline alumina the oxygen diffusion is enhanced at the grain boundaries, and that the region of enhanced oxygen diffusion is much larger than the grain boundary itself (14). This may account for the development of the alveole ridge which, referring to the model proposed by HINDAM & SMELTZER, grow by counter diffusion of aluminum and oxygen at the grain boundaries of the lace oxide film. The formation of the whiskers over the alumina scale infers the aluminum transported through this oxide film to react with oxygen at the external scale surface. This leads to the conclusion that the oxide film at the bottom of the alveoles grows by aluminum lattice diffusion. This assumption is consistent with the lattest studies on defect properties of α-Al_2O_3 (15) which propose a point defect model involving aluminum interstitial ions and electrons as the major mobile species.

To conclude, a model may be proposed for the oxide growth on NiAl coatings, assuming the growth of alumina by outward diffusion of aluminum, arising from an Al-rich NiAl coating, through an interfacial thin textured oxide film, which grows by counter diffusion of oxygen and aluminum at its grain boundaries, promoting the development of the texture ridges, and by aluminum lattice diffusion to enable the alumina scale growth, as schematized in Fig. 17.

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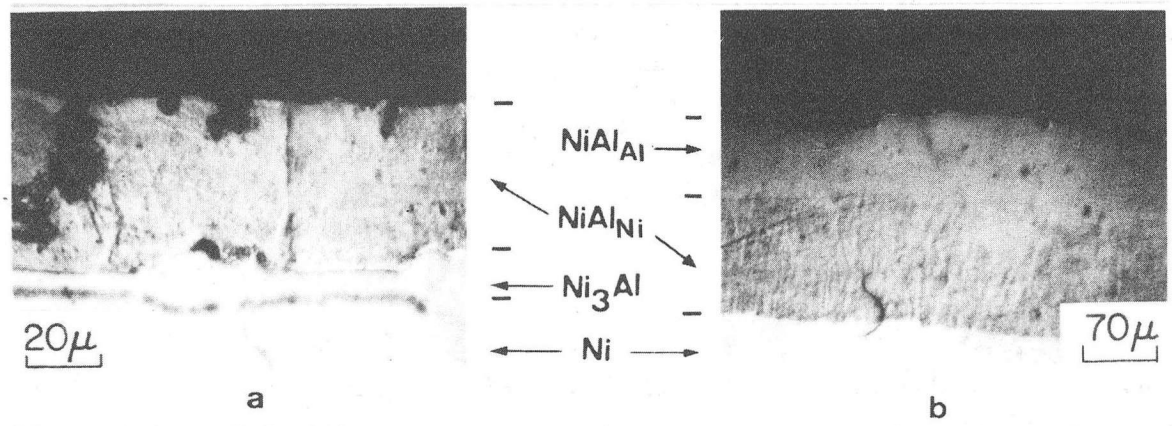


Fig.1.NiAl type coatings;(a)Ni-rich NiAl,(b)duplex Al-rich/Ni-rich NiAl.

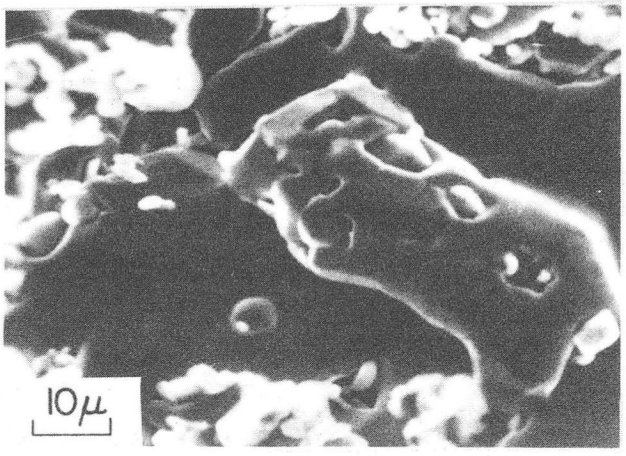


Fig.2.Al-rich plates upon Ni-rich NiAl coating.

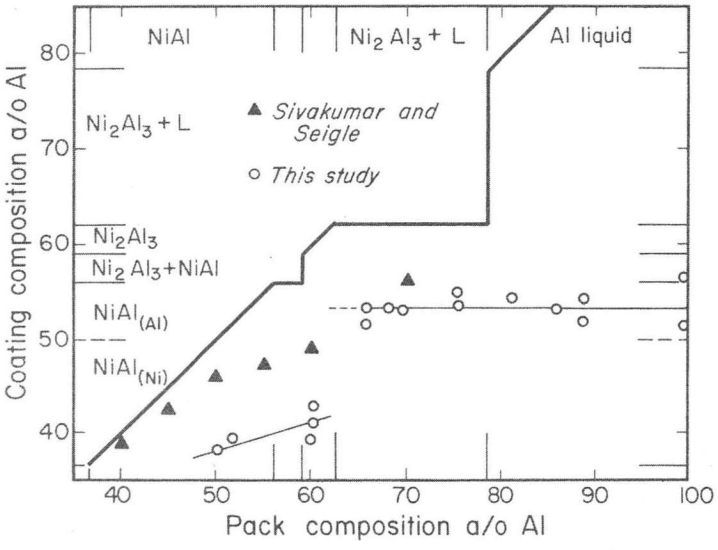


Fig.3.relationship between coating surface composition and pack composition in pack cementation of pure nickel.

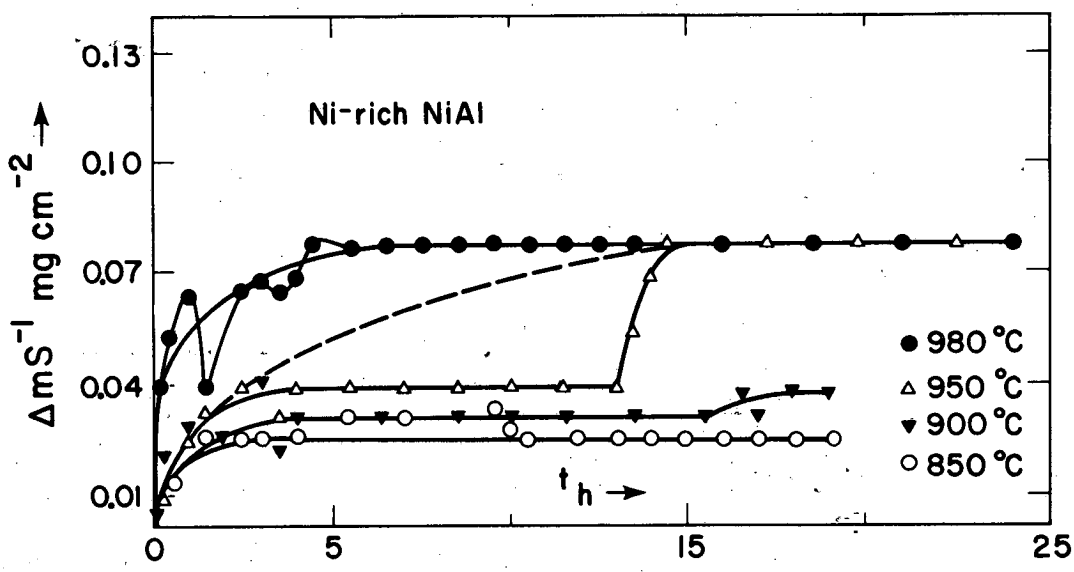


Fig.4.kinetics of oxidation of Ni-rich NiAl coatings.

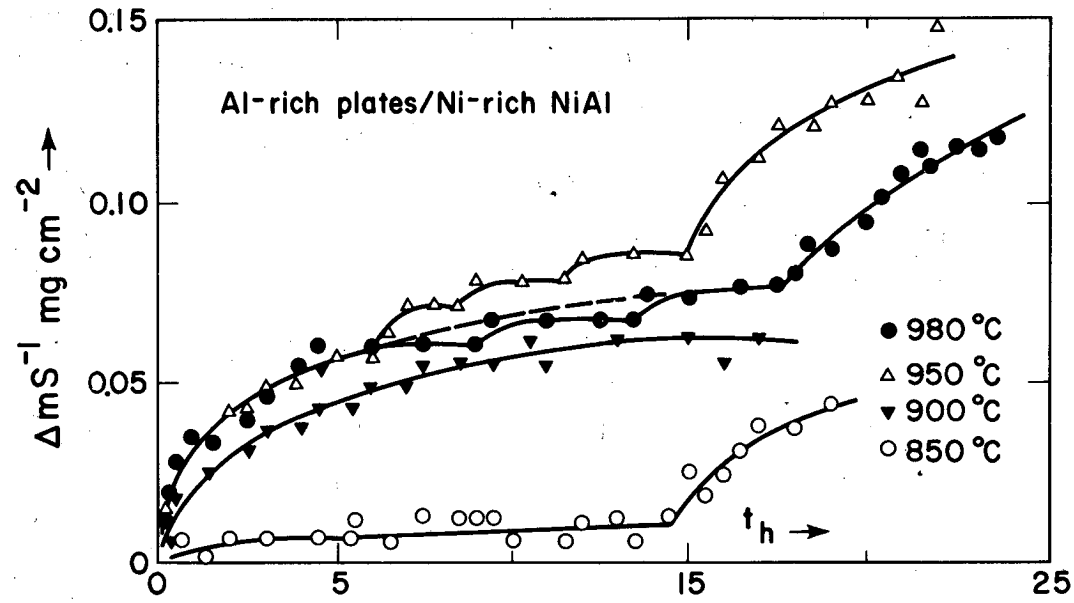


Fig.5.kinetics of oxidation of Ni-rich NiAl coatings partially covered by Al-rich plates.

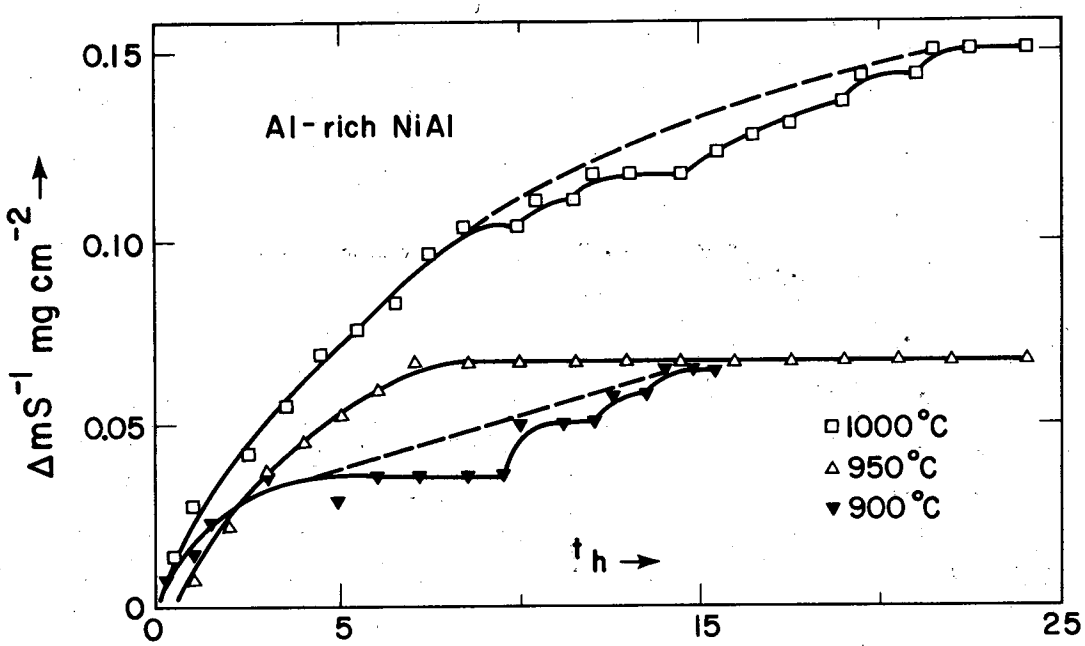


Fig.6.kinetics of oxidation of Al-rich/Ni-rich duplex NiAl coatings.

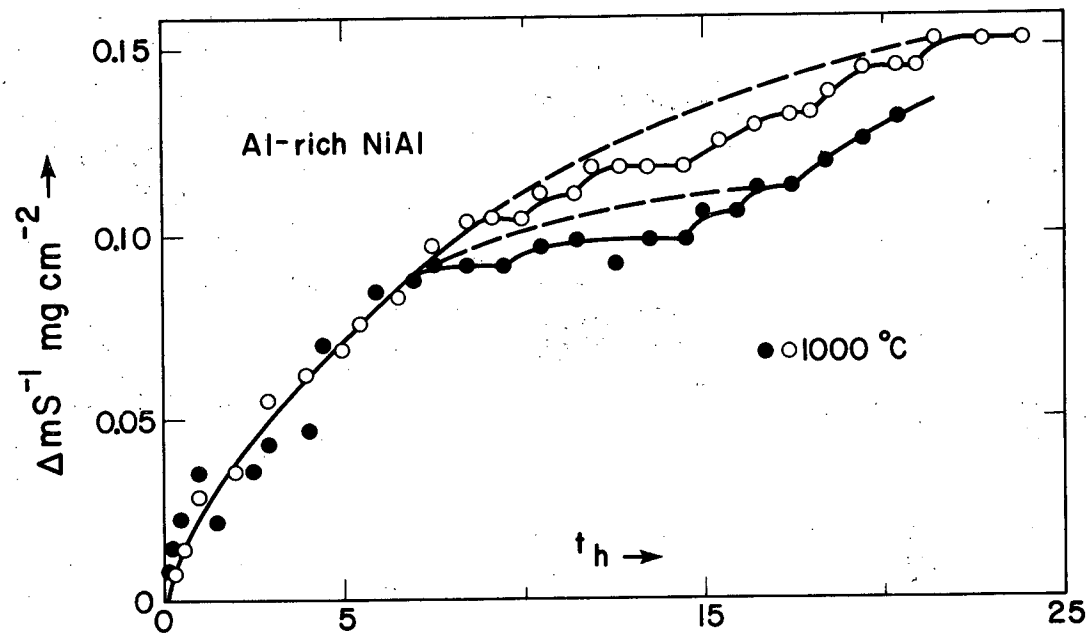


Fig.7.reproducibility of kinetics of oxidation of duplex NiAl coatings.

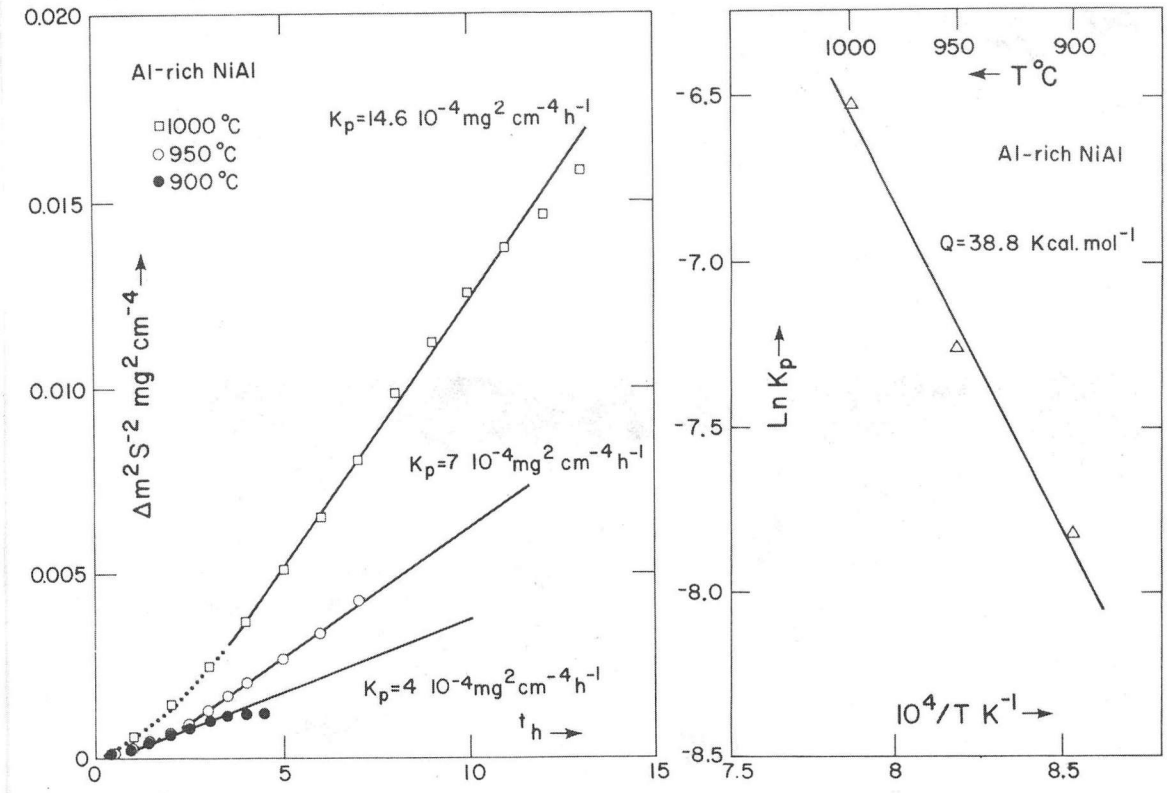


Fig.8. (a) parabolic plot of oxidation kinetics of Al-rich/Ni-rich duplex coatings; (b) Arrhenius plot.

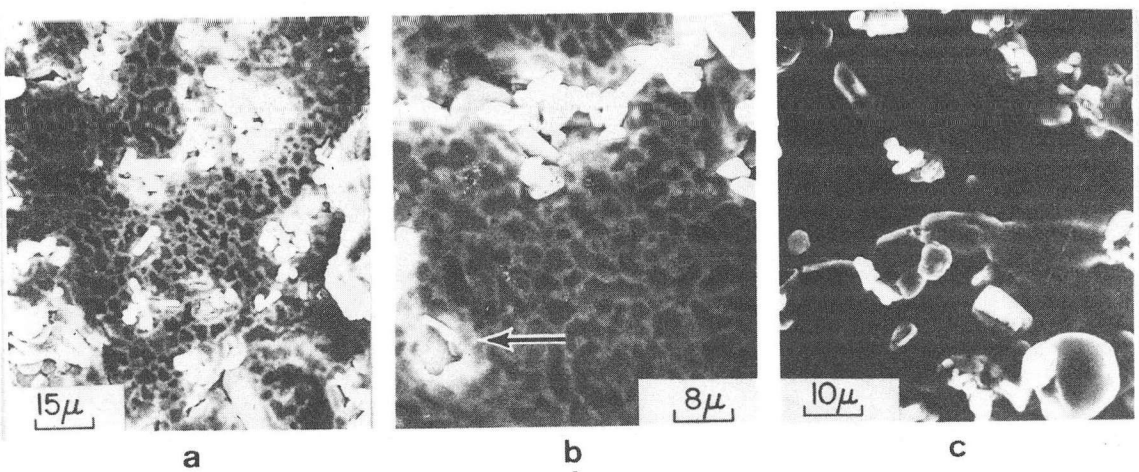


Fig.9.S.E.M. images of surfaces of: (a) Ni-rich NiAl oxidized at 900°C (b) higher magnification of same area; arrow points out mark of a detached particle ;(c)Ni-rich coating as cementated,before oxidation.

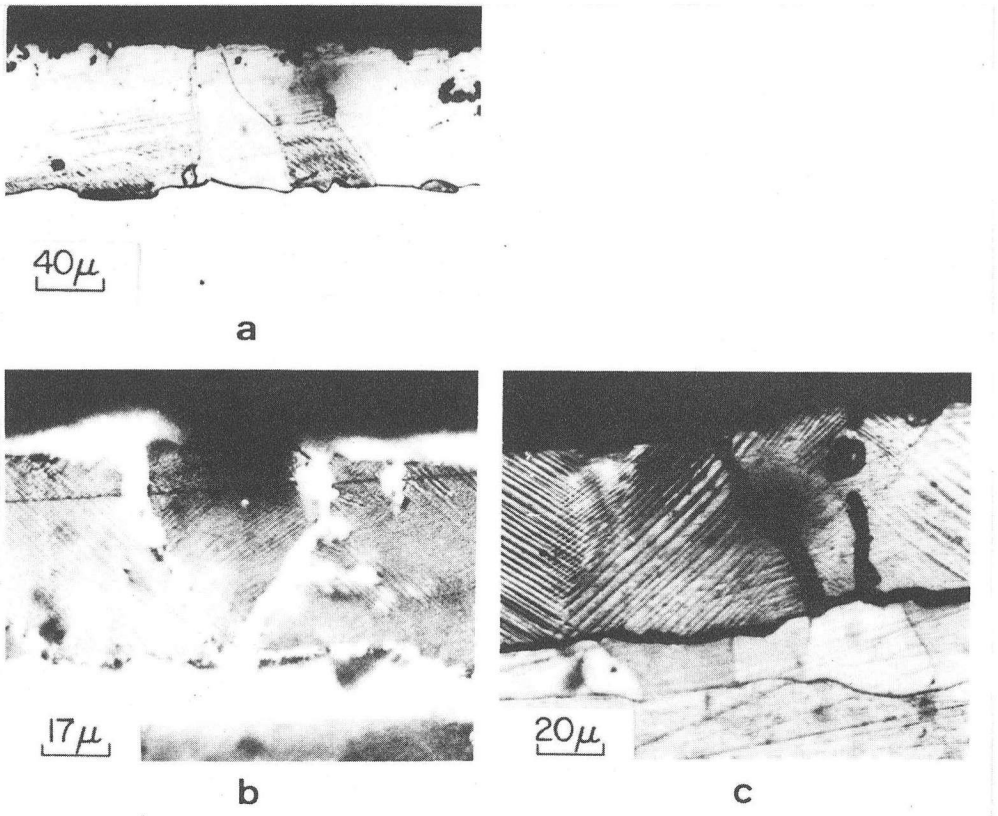


Fig.10.O.M.images of cross-sections of:(a)Ni-rich NiAl coating oxidized at 980°C;(b-c)Ni-rich NiAl coating with surface Al-rich plates oxidized at 980°C.

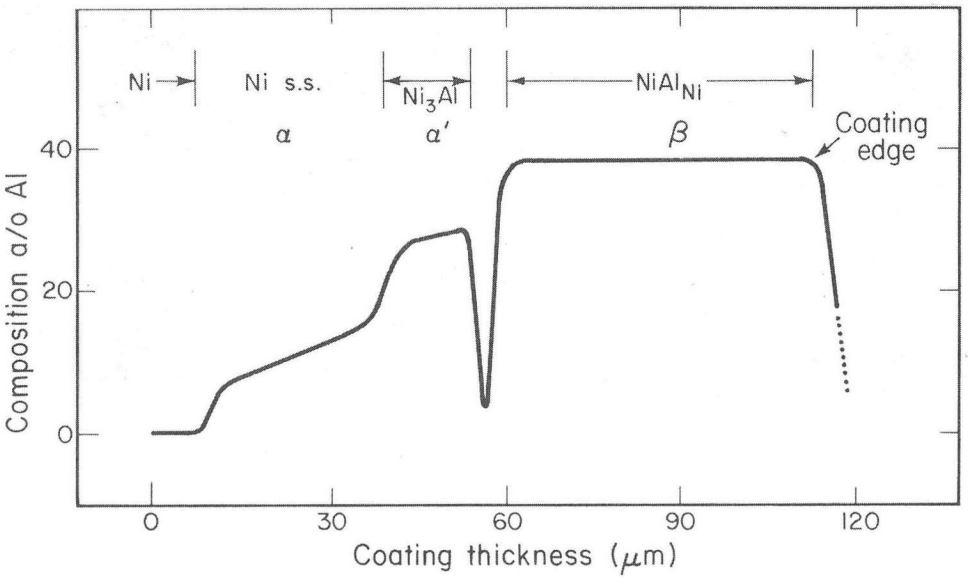


Fig.11.microprobe profil across the coating area of Fig.10-c.

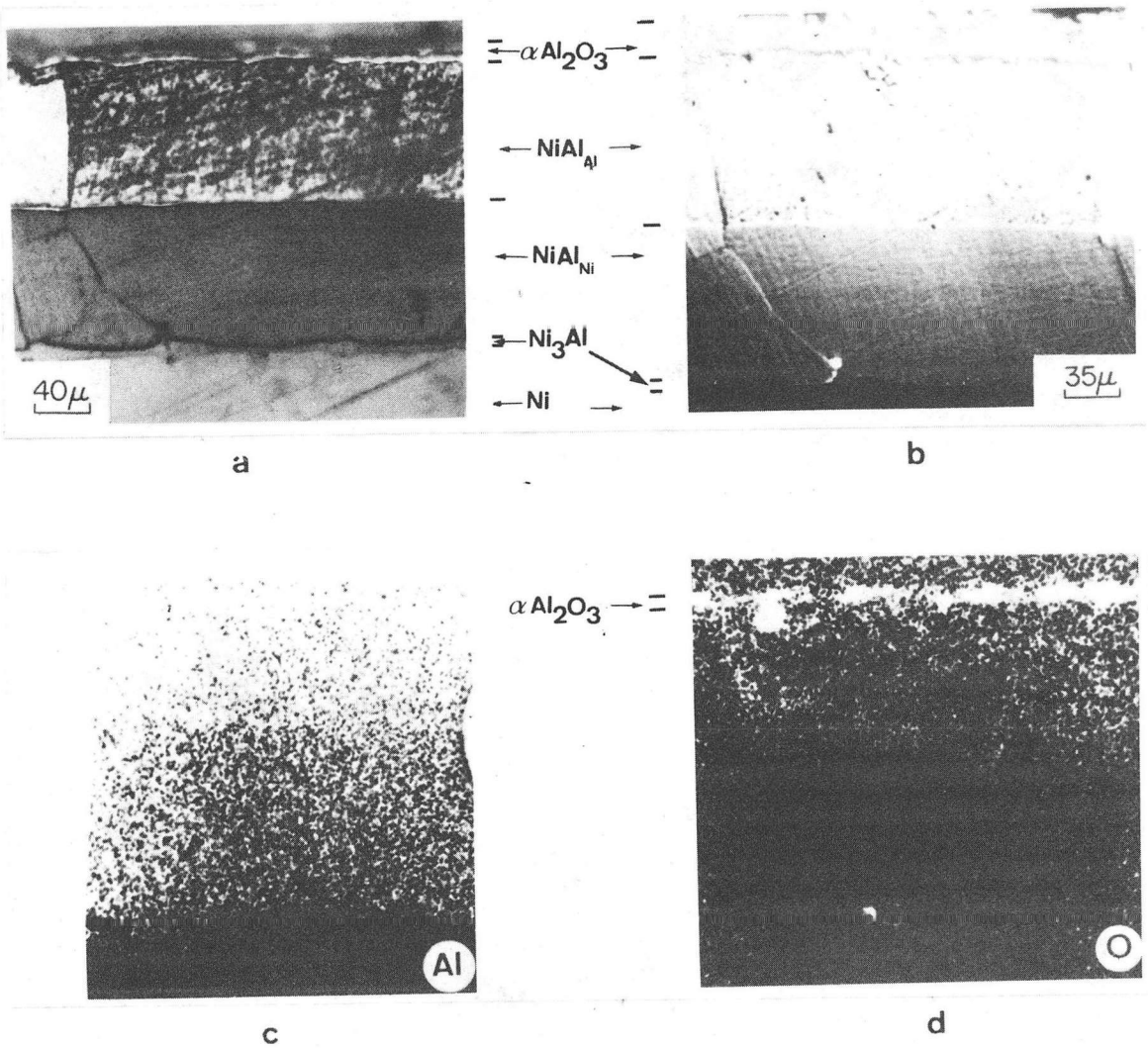


Fig. 12. Microprobe analysis of duplex Al-rich/Ni-rich coating oxidized at 980°C: (a) O.M. image of the cross-section; (b) absorbed current image of same area; (c) aluminum X-ray image; (d) oxygen X-ray image.

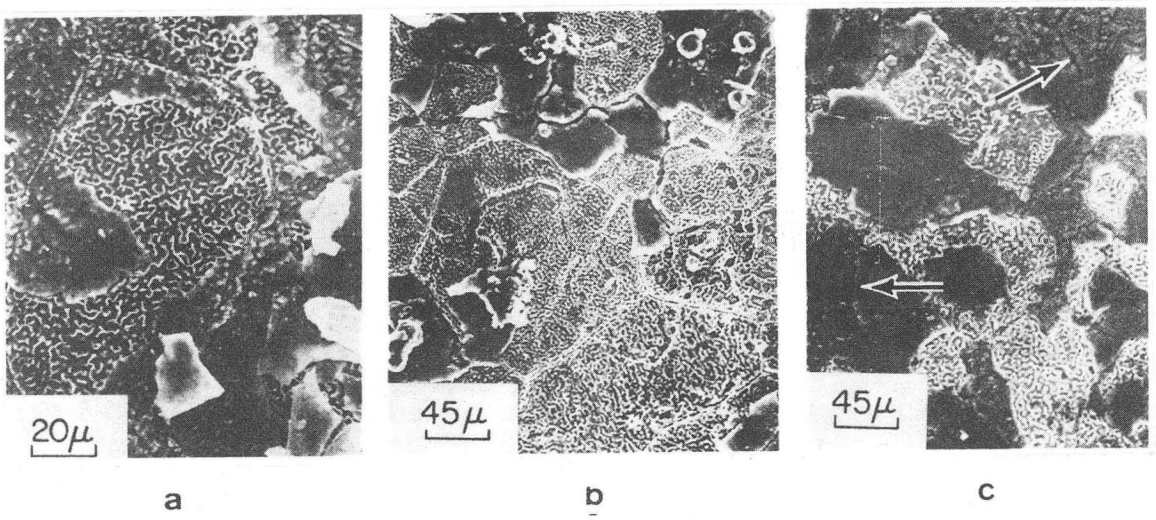


Fig.13. S.E.M. images of surfaces of: (a) coating with surface Al-rich plates oxidized at 950°C;shows spalled-off oxide debris; (b) external Al-rich NiAL of duplex coating oxidized at 900°C; shows ridges spacings varying with subjacent coating grains;(c)external Al-rich NiAl oxidized at 1000°C; arrows point out the imprint of coating grain boundaries.

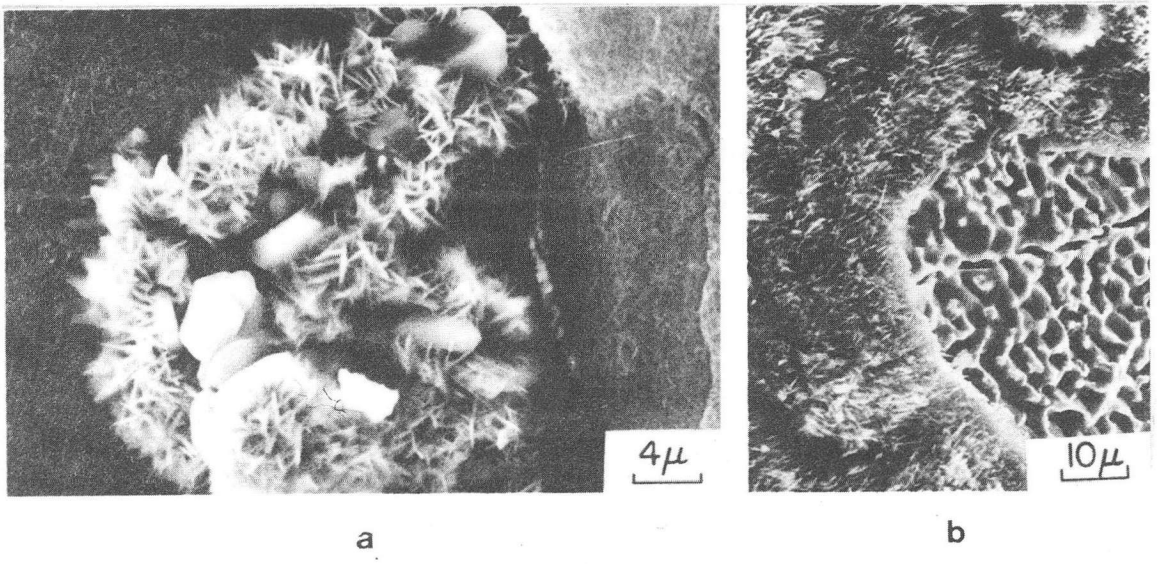
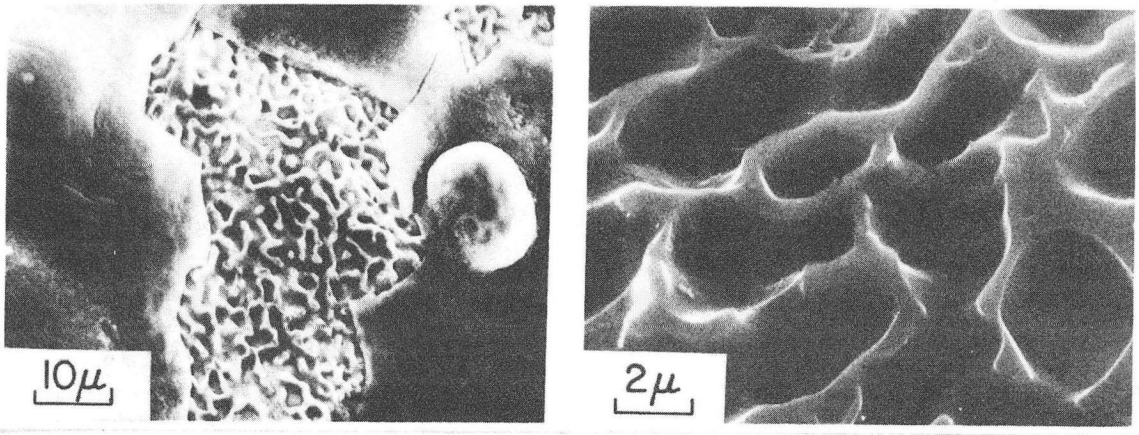
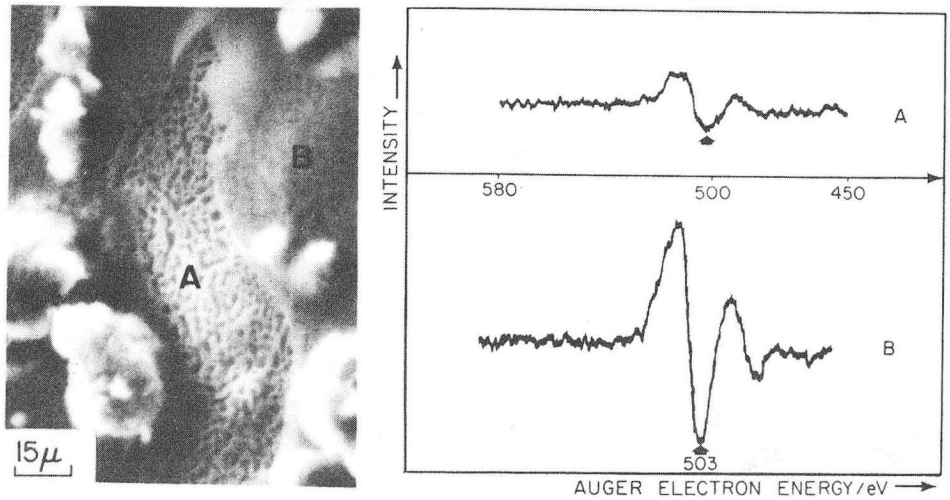


Fig.14. S.E.M. images of surfaces of external Al-rich NiAl oxidized at: (a) 900°C; shows the growth of whiskers inflorescences and protusions over a compact oxide scale;(b) 950°C;shows the development of a moquette of whiskers.



a b

Fig.15.typical features of:(a)the spalled-off oxide scale;(b)the alveole textured surface.



a b

Fig.16.Auger electron spectroscopy analysis of surfaces of NiAl coating oxidized at 850°C;(a)A.S.M.image;(b)oxygen AUGER electron spectra of the corresponding areas.

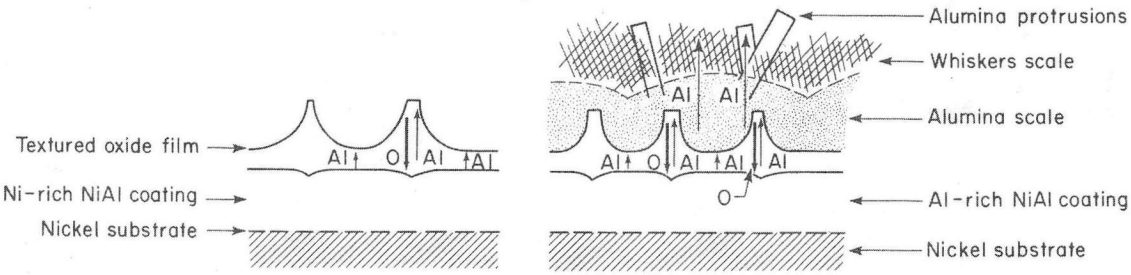


Fig.17.schematic model of oxide growth by oxidation of NiAl coatings.

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