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**High-Temperature Oxidation of an Alumina-Coated Ni-Base Alloy**

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HIGH TEMPERATURE OXIDATION  
OF AN ALUMINA-COATED NI-BASE ALLOY

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Abstract

Alumina coatings were applied to Ni-20Cr (wt%) using combustion chemical vapor deposition (combustion CVD). Combustion CVD is an open air deposition technique performed in a flame. The oxidation kinetics of coated and uncoated specimens were measured by isothermal oxidation tests carried out in pure flowing air at temperatures of 800, 900, 1000 and 1100°C. The alumina coatings reduced the oxidation kinetics at all temperatures. The morphologies and compositions of the alumina coatings were characterized by transmission and scanning electron microscopy, energy dispersive x-ray spectroscopy and x-ray photoelectron spectroscopy.

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## Introduction

Alumina coatings are attractive in many applications such as wear resistant coatings, films on electronic devices and coatings for corrosion resistance. This last application is of particular interest since  $\text{Al}_2\text{O}_3$  is considered to be one of the best protective oxides for high temperature applications. Aluminum is, therefore, frequently present as an alloying element in high temperature alloys. An applied layer of protective  $\text{Al}_2\text{O}_3$  can also act as a barrier to oxidation on alloys containing little or no aluminum. For example, alumina coatings have slowed the oxidation of TiN (1), superalloys (2) and stainless steels (3).

Alumina has been deposited by several techniques including atomic layer epitaxy (4), pyrolytic decomposition (5,6) spray pyrolysis (7), ion assisted deposition (8,9), laser physical vapor deposition (10), reactive sputter deposition (2), air plasma spray (11) and chemical vapor deposition (12-21). The alumina coatings in the present work were deposited by a recently developed open-air CVD process called combustion chemical vapor deposition (22). Combustion CVD is a non-line-of-sight deposition technique that uses a flame as both the deposition environment and source of energy for the appropriate reactions to occur. No auxiliary substrate heating or reaction chamber is needed in this process which occurs at atmospheric pressure. Both liquid and gaseous fuel combustion CVD is possible. For liquid fuel combustion CVD, which was used in the present work, the coating precursors are dissolved into the fuel. Previously deposited materials include  $\text{BaTiO}_3$ ,  $\text{Y}_2\text{BaCuO}_x$ ,  $\text{YBa}_2\text{CuO}_x$ ,  $\text{Y}_3\text{Fe}_5\text{O}_{12}$ , Ag, Pt (22),  $\text{SiO}_2$  (23), YSZ (24) and  $\text{CeO}_2$  (25).

## Experimental Procedure

The Ni-20Cr (wt%) substrates were prepared by triple arc melting nickel and chromium (nominally 99.995% Ni and 99.996% Cr). Test coupons were created by punching approximately 0.35 cm x 1.0 cm blanks from the ~0.035 cm thick alloy foil which was cold-rolled from the arc melted alloy button. A hole was drilled near one end of each coupon as an attachment point. After the substrates were sanded to a 600 grit finish, cleaned and placed in an evacuated quartz tube, they were annealed for one hour at 1050°C. This was followed by electropolishing to a mirror finish in a methanol/20% sulfuric acid solution at 0°C.

The combustion CVD apparatus used to deposit the alumina coatings is shown schematically in Figure 1. The fuel/coating precursor solution consisted of ethanol and aluminum-acetylacetonate. Laboratory grade oxygen was used as the oxidant. Specimens were coated individually in the flame produced by the solution, oxygen and a natural gas pilot flame. Each sample was rotated in the flame at 4 rotations per minute during the deposition process for a total of 40 minutes, which allowed coverage of all sides of the coupons. The temperature of deposition, measured with a type K thermocouple held near the surface of the sample exposed to the flame, was approximately  $850^\circ\text{C} \pm 50^\circ\text{C}$ . Ethanol flame-treated control samples were formed by placing a coupon in a pure ethanol flame,

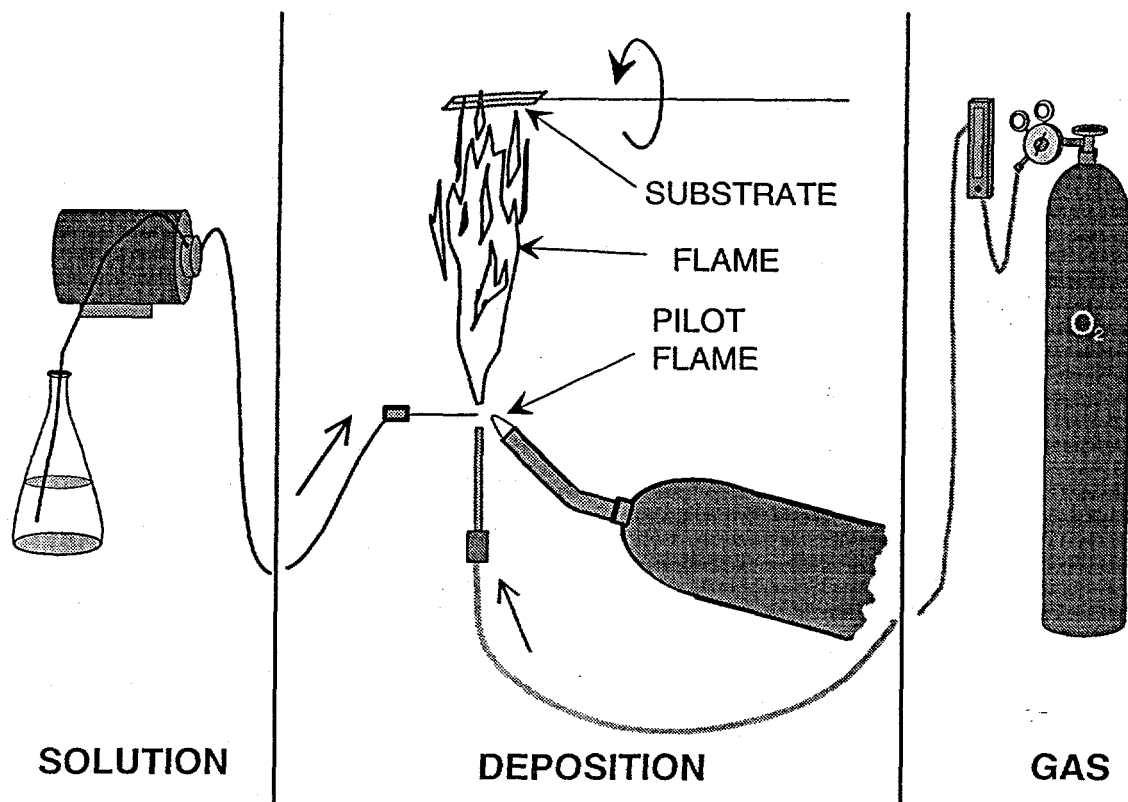


Figure 1 - Schematic of the liquid fuel combustion CVD process.

absent of the Al precursor, for the same time and temperature. Depositions also were performed on fused quartz substrates in order to analyze the resultant coatings without the complication of Ni, Cr or their oxides that may have formed during deposition on Ni-20Cr.

Isothermal gravimetric analysis (TGA) was accomplished using a D-200 Cahn microbalance. Each specimen was lowered into a pre-heated furnace and reached test temperature within 5 to 10 seconds. Coated and uncoated substrates were oxidized at 800, 900, 1000 and 1100°C. A 48 hour duration test was sufficient for the samples to attain steady state oxidation at all temperatures except 800°C, which required 96 hour oxidation tests. The ethanol flame-treated control specimens were oxidized via TGA at 1000°C only.

The investigative techniques used to characterize the alumina coatings and other oxides that formed during oxidation included optical microscopy, scanning electron microscopy (SEM) with an oxygen sensitive energy dispersive x-ray spectrometer (EDS), transmission electron microscopy (TEM) with an ultrathin window EDS detector and x-ray photoelectron spectroscopy (XPS). The microscopy specimens were examined without any extraneous coatings.

## Results and Discussion

### Combustion CVD

Deposition conditions needed to form the alumina coatings were established by preliminary experimentation on quartz substrates. XPS was used to verify the presence of aluminum and oxygen. The films were scraped off the quartz substrates onto TEM grids to analyze the composition of the deposited alumina. The presence of aluminum and oxygen was also confirmed with EDS while diffraction studies revealed the alumina polymorph deposited to be  $\theta$ -alumina (to be presented elsewhere).

The conditions established for deposition onto Ni-20Cr included a solution of 0.00135M Al-acetylacetonate/ethanol, a temperature of 850°C and a time of 40 minutes. Optical microscopy revealed a distinct difference between alumina-coated specimens and ethanol flame-treated specimens. The alumina-coated Ni-20Cr specimens appeared dark and shiny to the eye after combustion CVD with various colors reflecting from the surface of the samples under incandescent light. This was in contrast to the ethanol flame-treated samples which were gray. Such a color difference in the coated versus uncoated specimens after flame exposure indicated differing responses of the Ni-20Cr specimens to a pure ethanol flame versus an aluminum precursor-containing ethanol flame. Scanning electron microscopy revealed the alumina coating to be nodular but continuous over the substrate surface, as shown in Figure 2, and showed distinct differentiation of the substrate grains.

Specimens exposed to a flame containing the alumina precursor experienced mass gains ranging between 0.05 and 0.17 mg/cm<sup>2</sup>. These values included not only the mass of the deposited alumina but also any alloy oxides that formed during deposition. Conversely, the ethanol flame-treated specimens experienced mass gains between 0.35 and 0.46 mg/cm<sup>2</sup>. The lower mass gains of the former specimens illustrate that considerable oxidation protection of the substrate occurred during the combustion CVD process.

### Oxidation

Figure 3 shows the oxidation kinetics of the coated and uncoated samples oxidized at 800, 900, 1000 and 1100°C. The mass gain curves of the alumina-coated samples were consistently lower than those of the uncoated samples. The mass gains of the coated specimens were in part smaller due to the pre-oxidation that occurred during the alumina deposition at 850°C. The significance such initial oxidation might have on the amount of mass gained in the subsequent isothermal oxidation experiments was investigated using the ethanol flame-treated samples, which experienced a pure ethanol flame for 40 minutes (the total time an alumina-coated specimen spent in the flame during deposition). Ensuing isothermal oxidation at 1000°C showed some decrease in the mass gained during TGA compared to the uncoated and untreated TGA specimen, as illustrated in

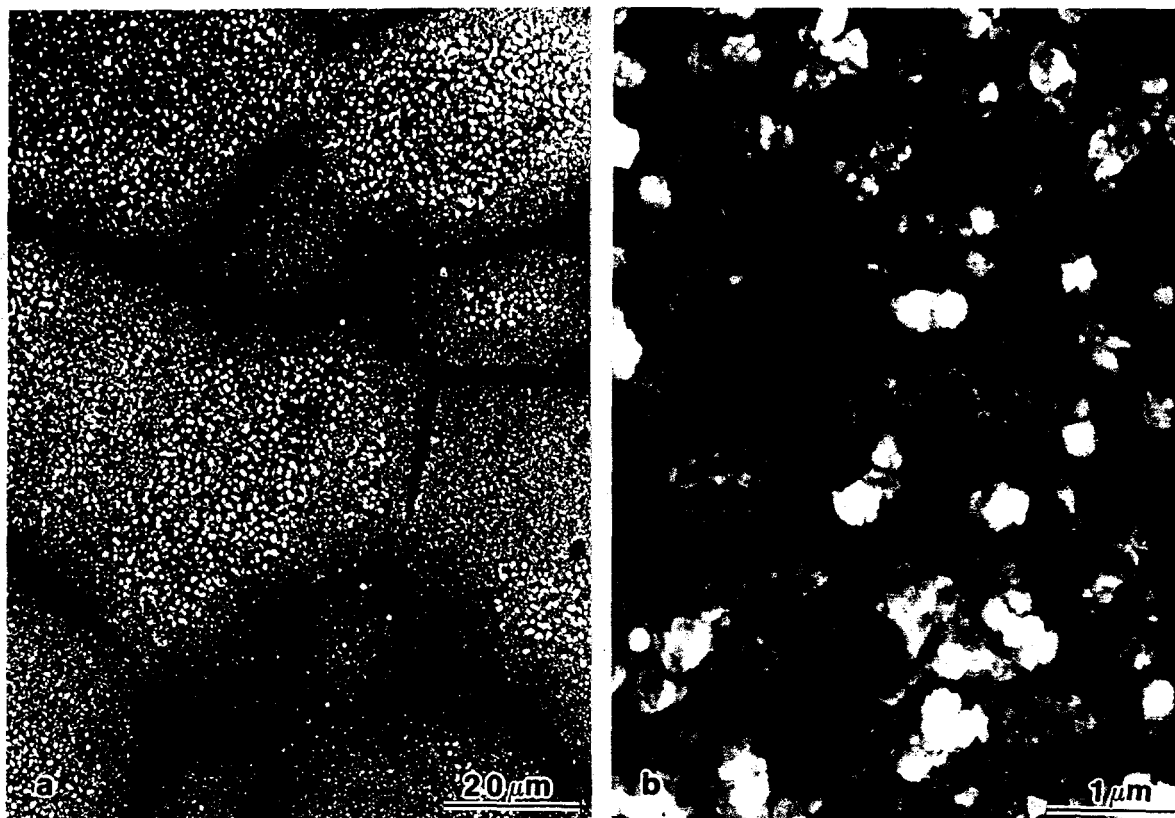


Figure 2 - SEM micrographs at (a) low and (b) high magnification of the deposited alumina on Ni-20Cr.

Figure 3, yet the alumina-coated specimen still gained the least mass at 1000°C. Each substrate mass was tared to zero milligrams prior to oxidation during TGA. Thus, part of the difference in mass gain between the coated and uncoated specimens may have been due to the mass of substrate oxides that had already formed during combustion CVD before the deposited alumina was able to inhibit the substrate oxidation in the flame. However, an additional form of protection beyond that provided by an oxide formed on the flame-oxidized Ni-20Cr can be attributed to the deposited alumina.

The isothermal oxidation of uncoated Ni-20Cr is well understood (26,27). A transient oxidation behavior is exhibited due to the formation of different oxides. The early stage of oxidation consists primarily of NiO external scale formation.  $\text{Cr}_2\text{O}_3$  initially forms an external scale at the alloy grain boundaries. A continuous chromia layer is eventually formed beneath the NiO as a result of chromium diffusing laterally in the alloy from the grain boundaries to intragranular regions. The oxidation rate is then controlled by diffusion through the  $\text{Cr}_2\text{O}_3$  layer. The oxidation kinetics are initially rapid while NiO is forming and then slow to obey the parabolic rate law once a continuous chromia layer is formed.

The uncoated alloy specimens in the present investigations produced the expected Ni-20Cr oxides,  $\text{Cr}_2\text{O}_3$  and NiO with some  $\text{NiCr}_2\text{O}_4$  evident at lower temperatures.



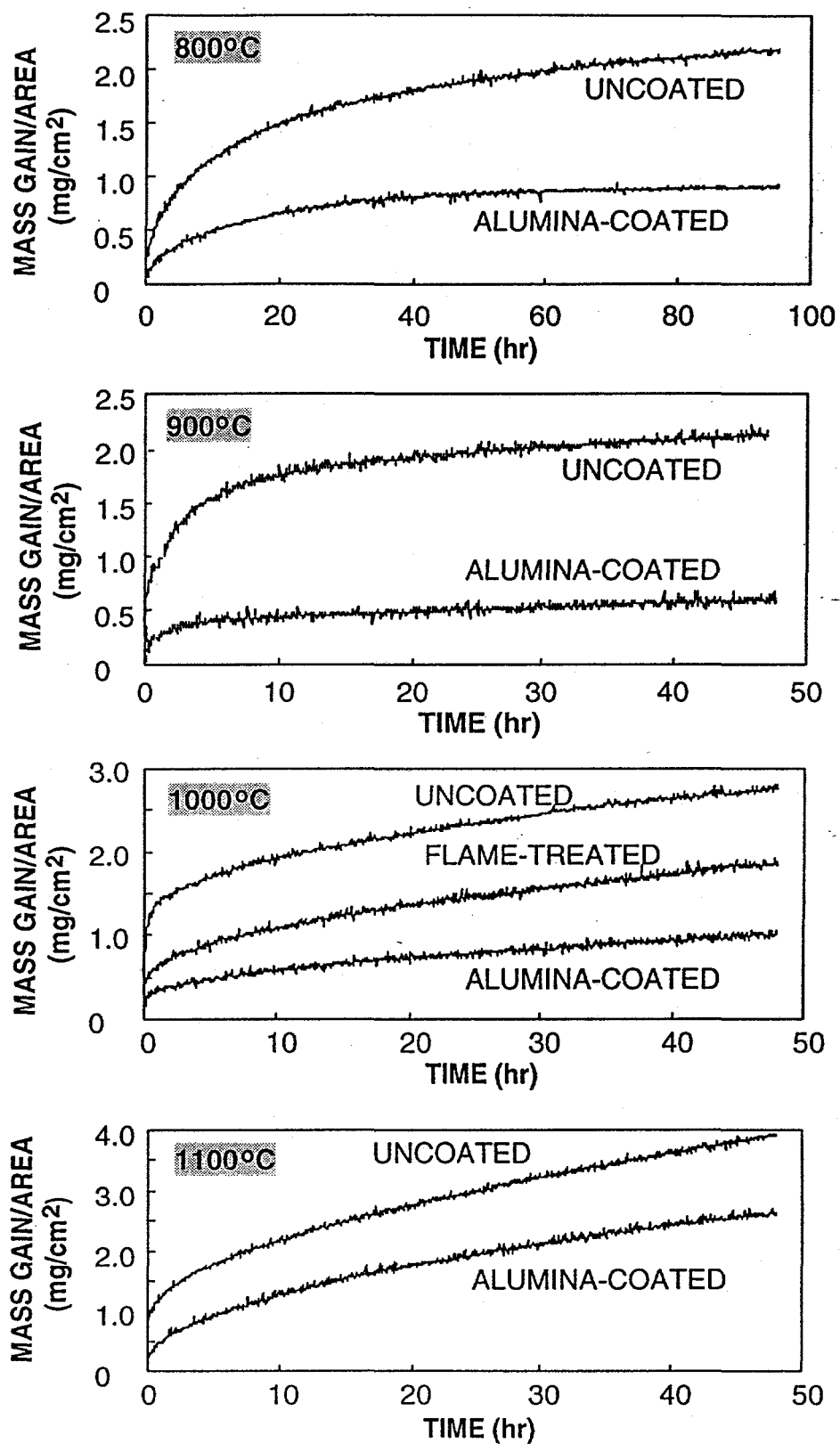


Figure 3 - Mass gain per unit area vs. time plots of alumina coated and uncoated specimens oxidized at 800, 900, 1000 and 1100°C.

Figures 4 and 5 display representative SEM micrographs taken from uncoated samples oxidized at 800°C (96 hours) and 1000°C (48 hours), respectively. EDS analysis of these samples shows strong signals from Ni and O, consistent with the expected formation of NiO at the gas/oxide interface. The faceted oxide scale was consistently present across the entire sample. The 800°C sample contained thinner oxide near the alloy grain boundaries while the 1000°C sample displayed a uniform oxide scale which lacked the easily differentiated alloy grain boundaries observed after the lower temperature oxidation.

The 800 and 1000°C oxidized alumina-coated samples displayed markedly different oxide morphologies, shown in Figures 6 and 7, respectively, as compared to the uncoated samples. At 800°C (96 hours), faceted oxide, which produced strong Ni and O signals, was present on the "interior" of the alloy grains, consistent with a scale of NiO. A strong Cr signal and a substantial Al signal were present on the alloy grain boundaries. The oxide covering the grain boundaries was nodular and had smaller grains as compared to the oxide covering the substrate grains as well as the oxide covering the substrate grain boundaries of the corresponding uncoated specimen. Cross-sectional analysis revealed this oxide to be  $\text{Cr}_2\text{O}_3$ . Thus, the alumina coatings promoted the selective oxidation of chromium at 800°C. Further, EDS on this cross-section reveals an outer NiO scale, an intermediate  $\text{NiCr}_2\text{O}_4$  scale and an inner  $\text{Cr}_2\text{O}_3$  scale on intragranular alloy regions. A layer proposed to be the deposited alumina due to its substantial Al EDS signal was also present between the NiO layer and the spinel.

The alumina-coated specimen oxidized at 1000°C (48 hours) displayed a fairly uniform oxide morphology across the sample surface, as shown in Figure 7. The substrate grain boundaries could no longer be differentiated from the grains. Columns of oxide were interspersed in a continuous oxide layer. EDS signals from this sample showed Ni, Cr and O signals with a stronger Ni signal arising from the oxide columns than from regions between the columns. Analysis of the uncoated and coated cross-sectional samples oxidized at 1000°C (48 hours) reveals the uncoated sample to display an outer oxide of NiO and an inner scale of  $\text{Cr}_2\text{O}_3$ , Figure 8a. The coated samples, shown in Figure 8b, display similar layers but of decreased thicknesses. That is, both the NiO and  $\text{Cr}_2\text{O}_3$  layers are reduced in thickness as a result of the  $\text{Al}_2\text{O}_3$  deposition. This is consistent with the  $\text{Al}_2\text{O}_3$  layer promoting the nucleation of  $\text{Cr}_2\text{O}_3$ , resulting in the lower mass gains shown in Figure 3. Aluminum was not detected on this sample by EDS.

The oxide morphologies of samples oxidized 48 hours at 900 and 1100°C also were investigated. The 900°C samples oxidized in a similar fashion as the 800°C samples. The alumina-coated sample yielded predominately Ni EDS signals from the substrate grain regions and Cr signals from the substrate grain boundaries. An aluminum signal was also detected at the grain boundaries but was reduced in intensity as compared to that observed from the 800°C coated sample. The 1100°C sample morphologies were consistent with those of the 1000°C specimen. The uncoated specimen exhibited a dense layer of surface oxide with distinct oxide grains visible through scanning electron microscopy and a strong Ni EDS signal.

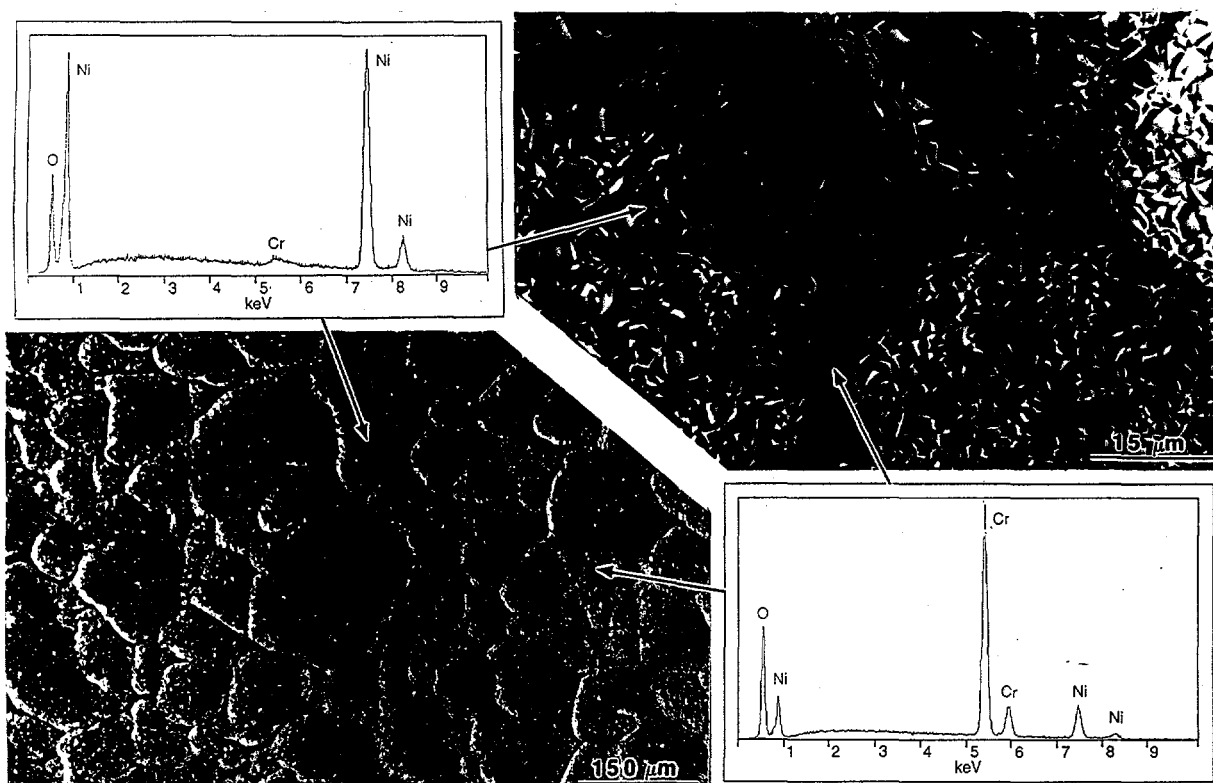


Figure 4 - Scanning electron micrographs and representative EDS spectra from Ni-20Cr oxidized 96 hours at 800°C.

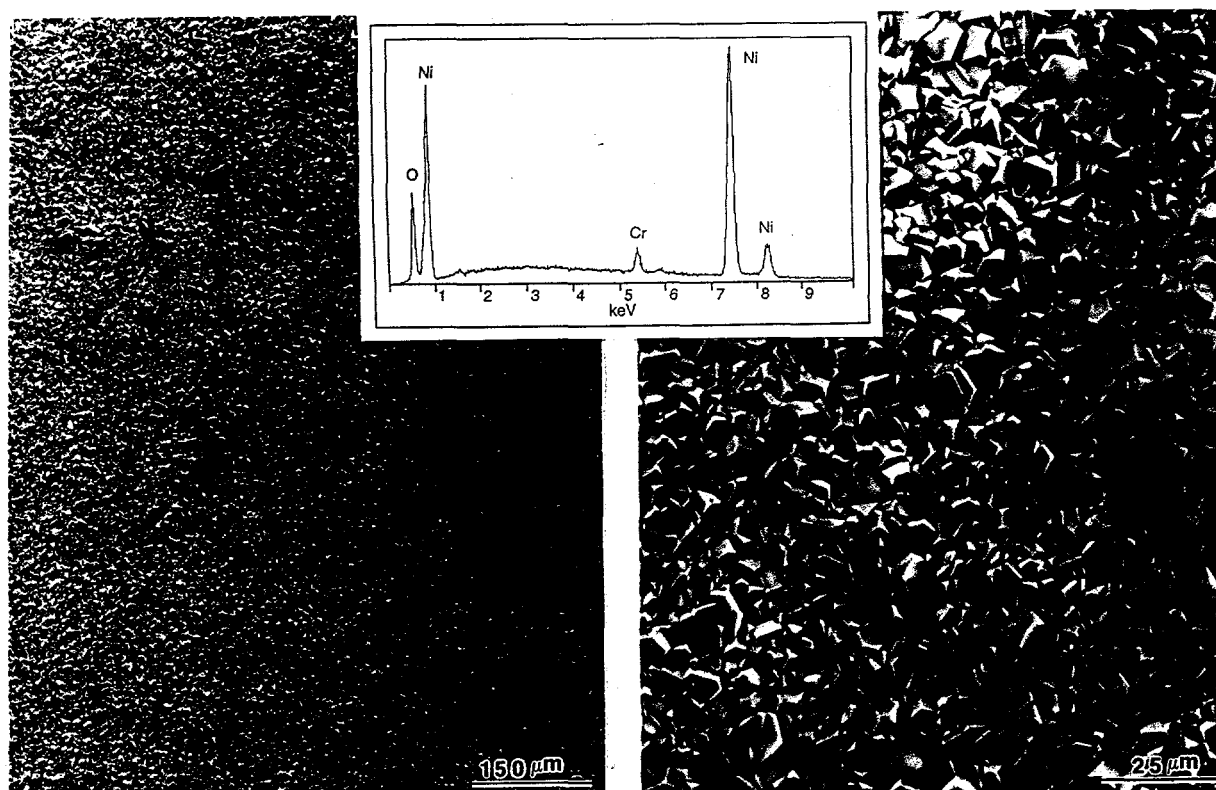


Figure 5 - Scanning electron micrographs and a representative EDS spectrum from Ni-20Cr oxidized 48 hours at 1000°C.

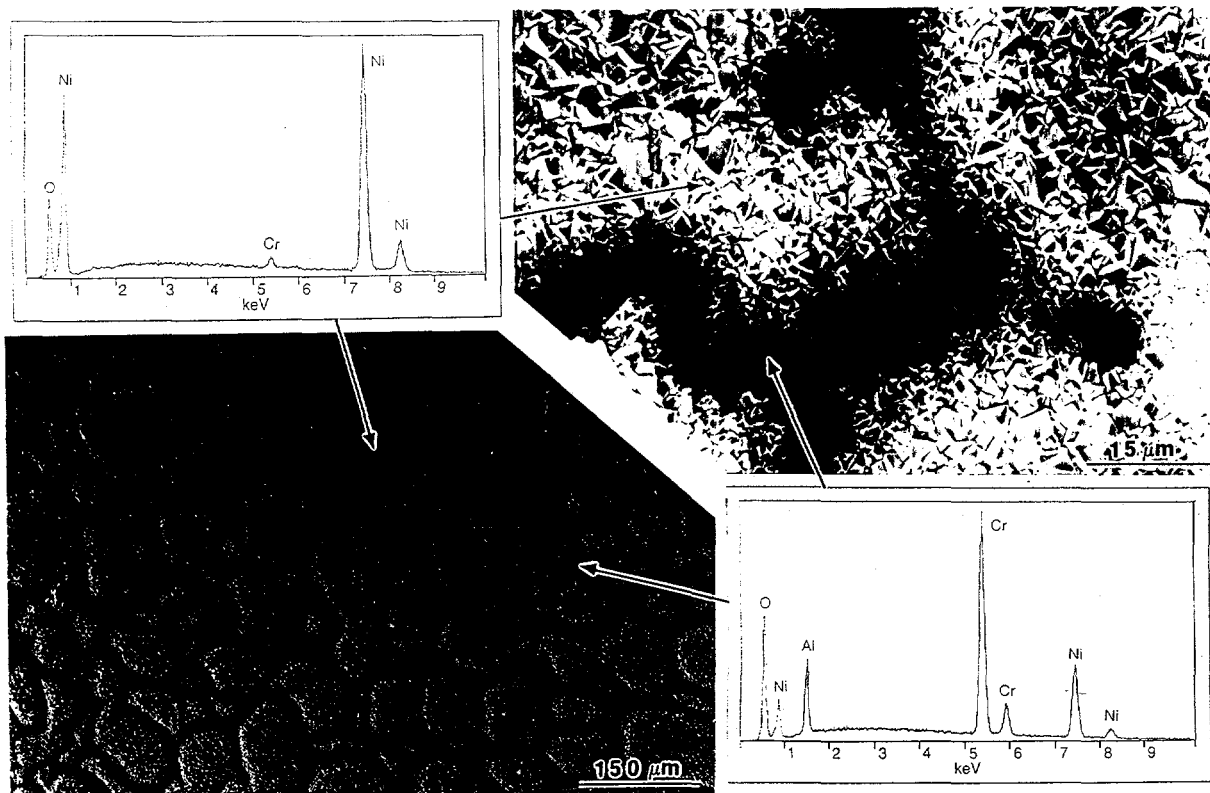


Figure 6 - Scanning electron micrographs and representative EDS spectra from alumina-coated Ni-20Cr oxidized 96 hours at 800°C.

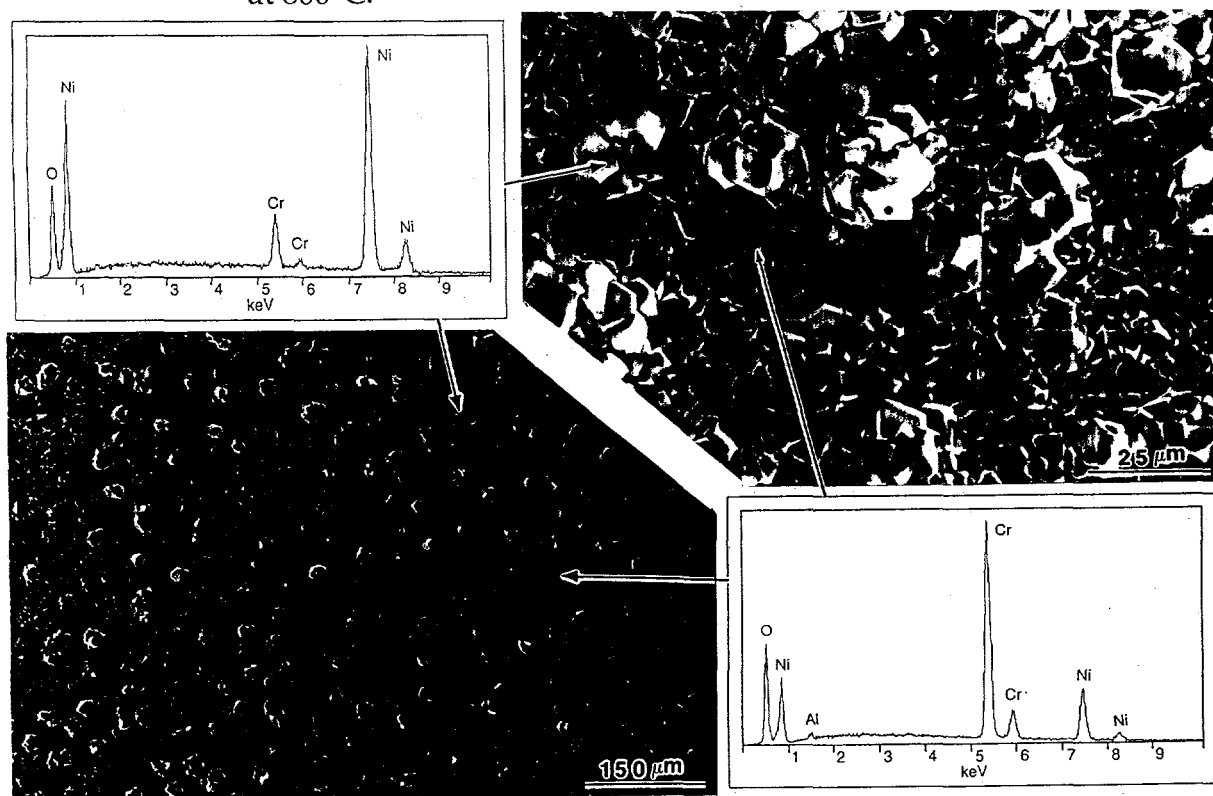


Figure 7 - Scanning electron micrographs and representative EDS spectra from alumina-coated Ni-20Cr oxidized 48 hours at 1000°C.

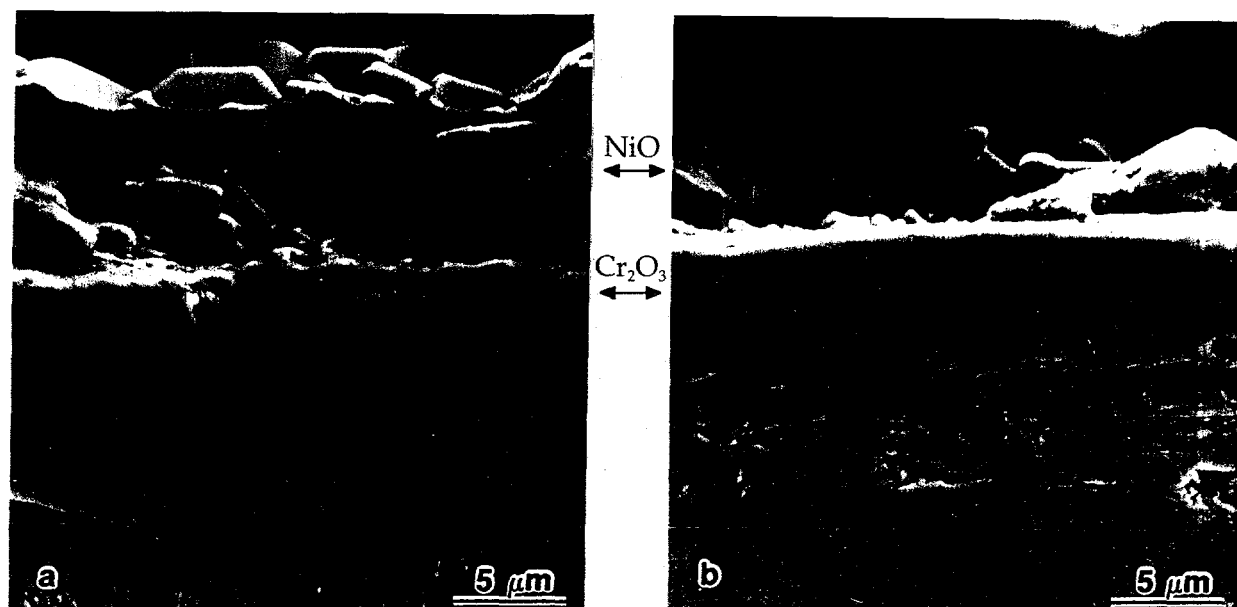


Figure 8 - Scanning electron micrographs from cross-sections of Ni-20Cr after oxidation for 48 hours at 1000°C: (a) uncoated and (b) alumina-coated (absent of oxide columns).

In contrast to the uncoated specimen, the alumina-coated and oxidized specimen displayed a layer of faceted oxide with a uniform grain size covering the substrate surface and larger faceted grains distributed throughout the layer. A strong EDS Cr signal was detected from the coated sample. Cross-sectional analysis of the 900 and 1100°C oxidized coated samples revealed similar oxide layering regimes as observed in the 1000°C specimen. Two distinct layers were observed: an outer layer of NiO and an inner layer of  $\text{Cr}_2\text{O}_3$ . These layers were thinner than on the uncoated sample. No clear alumina layer was detected using EDS. Therefore, at all temperatures investigated, alumina-coated samples had thinner NiO scales, consistent with the alumina acting to promote the nucleation of  $\text{Cr}_2\text{O}_3$ . This is consistent with the findings of Yedong and Stott (28) who found an alumina film on to promote the selective oxidation of chromium Ni-15Cr during the early stages of oxidation at 1000°C.

In addition to promoting the rapid formation of a continuous  $\text{Cr}_2\text{O}_3$  layer, the deposited alumina also serves to slow ionic transport through this layer. This is best seen through comparison of rate constants, Figure 9, obtained from the plots shown in Figure 3 (above 30 hours for the 900 to 1100°C data and above 60 hours for the 800°C data). Although the decrease in mass gain observed previously can be explained by the decrease in alloy oxide thicknesses as evidenced above, the change in rate constants between the uncoated specimens and their coated counterparts implies a modification in oxidation kinetics. We attribute this change to the alumina layer which was applied via combustion CVD. The mechanism by which this occurs is under further investigation. The alumina layer does not appear to simply act as a barrier layer in reducing the rate constant. Recall that the cross-sectional analyses showed a distinct aluminum-containing

oxide layer at 800°C only and not at the other temperatures. Further investigation of these samples is necessary to pinpoint the location of the deposited alumina within the oxide scale.

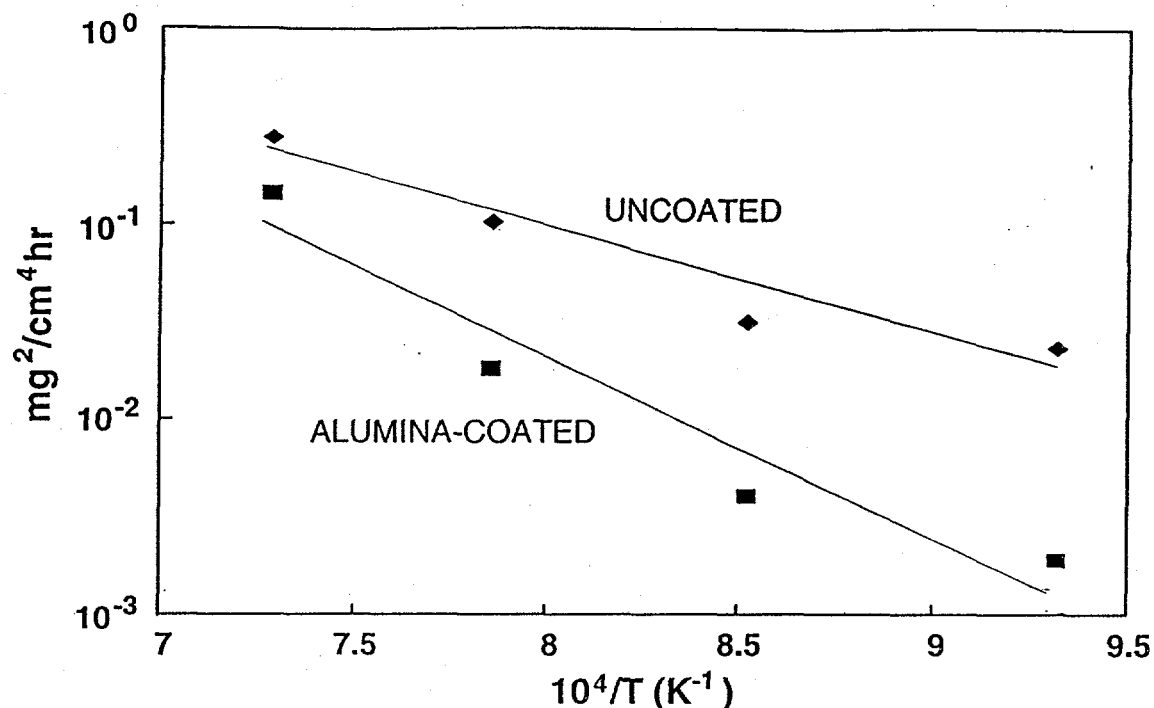


Figure 9 - Oxidation kinetics of coated and uncoated specimens.

### Summary and Conclusions

Combustion CVD was used to deposit  $\theta$ -alumina at 850°C on fused quartz and on rotating Ni-20Cr substrates using a solution of aluminum acetylacetonate in ethanol. The alumina coating protected the alloy substrates from significant oxidation during the deposition process. After subsequent oxidation at 800, 900, 1000 and 1100°C, the alumina-coated specimens had consistently lower mass gains compared to uncoated control specimens. The decreased mass gains were associated with thinner layers of alloy oxides than those that grew on the uncoated specimens. The deposited alumina promoted the selective oxidation of  $\text{Cr}_2\text{O}_3$  at all temperatures. Finally, the oxidation kinetics of the coated specimens were lower than those of the uncoated specimens. The decreased kinetics are attributed to the deposited alumina layer but the exact mechanism of protection is still under investigation.

### Acknowledgments

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