ELSEVIER

Surface and Coatings Technology 133–134 (2000) 15–22

SURFACE & COATINGS TECHNOLOGY

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# Effect of composition on the oxidation and hot corrosion resistance of NiAl doped with precious metals

C. Leyens[a,1,*], B.A. Pint[b], I.G. Wright[b]

[a] *DLR-German Aerospace Center, Institute of Materials Research, 51170 Cologne, Germany*
[b] *Oak Ridge National Laboratory, 1 Bethel Valley Road, Oak Ridge, TN 37831-6156, USA*

**Abstract**

Cast NiAl alloyed with Cr, Pt, Pd, Ir and Ru was tested in 1-h cycles at 950°C under hot corrosion conditions and at 1150°C in oxygen. For comparison, Hf-doped NiAl variants and a cast NiPtAl alloy resembling the composition of commercial aluminide coatings were included. Cr was the only element that reduced hot corrosion attack of NiAl significantly. However, at higher temperatures, addition of Cr to Hf-doped NiAl accelerated the alumina scale growth rate and promoted spallation of the oxide scale. The results from initial detailed characterization indicate that rejection of chromium at the metal-oxide interface gives rise to the formation of chromium-rich precipitates in the alloy, which apparently modify its oxidation behavior. This suggests that for NiAl-based substrates, hot corrosion resistance and exceptional scale spallation resistance may be mutually incompatible goals.
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*Keywords:* Nickel aluminides; Hot corrosion; Oxidation; Precious metals

## 1. Introduction

Aluminide coatings, deposited either by pack cementation or by chemical vapor deposition techniques (CVD), have been applied to gas turbine vane and blade airfoils since approximately 1970 [1]. Over 80% of all coated first stage blade airfoils are estimated to be coated by these techniques, indicating the enormous market share captured by these coatings. More recently, aluminide coatings were recognized as suitable bond coats for thermal barrier coatings (TBCs), in particular electron beam physical vapor deposited (EB-PVD) TBCs. The need for improved adherence of the protective oxide scale, in particular if used in TBC applications, has led to contemporary use of platinum-modified aluminide coatings for high performance gas turbine components, although the mechanism by which Pt exerts this beneficial effect on scale adhesion is not well-understood. Early experiments by Jackson and Rairden [2] indicated that platinum remains most concentrated at the coating–gas interface, thereby retarding diffusion of certain refractory elements to the coating–alumina scale interface and thus improving oxidation resistance. Schaeffer et al. [3] pointed out that Pt additions improved oxidation and hot corrosion resistance of coated superalloys, although the superiority of platinum aluminides was not fully attributed to a Pt-related adherence effect. Rather Pt changed the growth mechanism of the coating from outwardly growing without any Pt to inwardly growing when Pt was present. Improved isothermal and cyclic oxidation resistance was believed to be mainly attributed to a purity effect with Pt retarding diffusion of deleterious elements to the coating-alumina interface. Improvement of hot corrosion resistance by Pt additions compared with conventional aluminide coatings was attributed to Pt beneficially affecting scale adherence and cracking resistance [3].

* Corresponding author. Tel.: +49-2203-601-2029; fax: +49-2203-696-480.
*E-mail address:* christoph.leyens@dlr.de (C. Leyens).
[1] Part of this work was performed while the author was on sabbatical at Oak Ridge National Laboratory.

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PII: S0257-8972(00)00878-1

From their application in aeroengines and land-based gas turbines, platinum aluminide coatings are known to have superior resistance against Type-I hot corrosion compared to aluminide coatings (e.g. [3,4]). However, the role of Pt is essentially unknown. Haynes et al. [5] and Zhang et al. [6] compared CVD simple aluminide and Pt-modified aluminide coatings on a single crystal Ni-base superalloy substrate and observed the following influences of Pt: (1) improved scale adhesion in cyclic oxidation testing; (2) mitigation of the detrimental effects of high S levels in the substrate; (3) drastic reduction in the amount of voids at the scale metal interface; and (4) no difference in the concentration of substrate elements in the coating. Pint et al. [7,8] also reported a beneficial effect of Pt on alumina scale adhesion but demonstrated that a greater benefit was achieved by the addition of a reactive element such as Zr or Hf. Incorporation of reactive elements into an aluminide coating has not yet been developed for commercial production.

This work has used cast aluminides with well-controlled compositions to examine the role of precious metals and a reactive element (Hf) in hot corrosion and higher temperature oxidation testing. In order to examine the role of Pt, the performance of Pt-containing NiAl was compared with NiAl with additions of Ir, Pd and Ru. Also, additions of Cr to NiAl were examined as its beneficial role in hot corrosion is well known [9,10]. Results on cast material are intended to provide suggestions on improved coating compositions.

## 2. Experimental procedure

The alloys used in this study included cast NiAl alloyed with Pt, Ir, Pd, Ru and Cr, Hf-doped NiAl variants, a cast version of a (Ni,Pt)Al bond coating alloy containing Cr and Co, and a cast NiCoCrAlY bond coat alloy (Table 1). The alloys were vacuum induction melted and cast in a water-chilled copper mold. The castings were then annealed at 1300°C for 4 h in quartz ampules. Specimens (1.5 cm diam.× 0.1–0.15 cm thickness) were cut from the castings and polished with SiC paper to 600 grit, avoiding preferred orientation of the grinding marks. Prior to testing, all specimens were ultrasonically cleaned in acetone and methanol.

The hot corrosion test procedures used were devised to simulate a scenario associated with the firing of advanced land-based gas turbines with gas derived from the gasification of biomass. Unlike the conditions under which Type-I hot corrosion occurs, in this scenario the fuel gas is expected to be essentially sulfur-free, and the delivery of salt to the turbine hot section occurs by salt particles permeating or bypassing the hot gas filtration/desulfurization system immediately upstream of the combustor. In addition, the high surface temperature of TBC-covered surfaces preclude salt condensation, so that salt is delivered by intermittent impact of particulate matter, and its residence time on the surfaces is short.

Table 1
Chemical composition of cast nickel aluminides (at.%)

| Designation | Ni | Al | Cr | Co | Pt | Ir | Ru | Pd | S[a] | Others |
|---|---|---|---|---|---|---|---|---|---|---|
| NiAl + Hf | 48.84 | 51.05 | < 0.01 | | | | | | | 0.05 Hf[b]<br>0.01 Zr[b] |
| Ni-40Al | 59.68 | 40.27 | < 0.01 | < 0.01 | | | | | < 0.004 | < 0.01 Hf, Zr, Y |
| Ni-50Al | 49.91 | 50.05 | < 0.01 | < 0.01 | | | | | < 0.004 | < 0.01 Hf, Zr, Y |
| NiAl-2Cr + Hf | 48.28 | 49.63 | 2.00 | | | | | | 0.0004 | 0.05 Hf[b] |
| NiAl-5Cr + Hf | 47.76 | 47.20 | 4.95 | | | | | | 0.0065 | 0.05 Hf[b] |
| NiAl-10Cr + Hf | 45.13 | 45.07 | 9.70 | | | | | | 0.0005 | 0.054 Hf[b] |
| NiAl-2Pt | 47.36 | 50.21 | | | 2.35 | | | | 0.0004 | 0.005 Hf[b] |
| NiAl-5Pt | 45.10 | 49.60 | 0.05 | | 5.20 | | | | 0.0009 | |
| NiAl-2Pt + Hf | 47.78 | 49.71 | 0.03 | | 2.35 | | | | 0.0004 | 0.052 Hf[b] |
| NiPtAl + Cr + Co | 46.73 | 38.19 | 3.76 | 4.96 | 6.17 | | | | 0.001 | 0.003 Hf[b]; Mo, W, Re, Si; all < 0.07 |
| NiAl-2Ir | 47.09 | 50.52 | | | | 2.32 | | | | 0.005 Hf[b] |
| NiAl-5Ir | 44.52 | 50.84 | | | | 4.61 | | | | 0.003 Hf[b] |
| NiAl-2Ru | 47.64 | 49.98 | | | | | 2.34 | | | |
| NiAl-5Ru | 44.50 | 50.17 | | | | | 5.28 | | | |
| NiAl-3Pd | 47.55 | 49.84 | | | | | | 2.56 | | |
| NiAl-5Pd | 44.83 | 49.87 | | | | | | 5.29 | | |
| NiCoCrAlY[c] | 40.38 | 23.49 | 17.43 | 18.48 | | | | | 0.0008 | 0.15 Y |

[a] Determined by combustion analysis.
[b] Determined by inductively-coupled plasma analysis.
[c] Ni-22Co-18Cr-12.6Al-0.26Y in wt.%.

Consequently, a simplified test procedure was used in which specimens were exposed to 1-h thermal cycles at 950°C in dry oxygen, and were intermittently coated with salt. These tests were performed in automated test rigs at ORNL in which each cycle consisted of 1 h at temperature and subsequent 10 min cooling to room temperature. Dry oxygen flowed continuously into the bottom of the tube furnace during the exposures, and the $SO_x$ additions to the gas which are usually used in hot corrosion tests (on the order of 0.1 vol.%) were omitted. Specimens were attached to alumina rods by Pt-Rh wire hooks in a vertical tube furnace. All deposits were applied by the periodic coating method described in more detail in Leyens et al. [11].

The specimens were coated with a water solution of sodium sulfate ($Na_2SO_4$); this salt was used instead of the sulfur-deficient, potassium-rich salts used in an earlier phase of this program to simplify the test procedure. The salt solution was deposited onto the specimens using a dropper pipette, and the water was evaporated by heating the specimens to approximately 200°C on a hot plate. Specimens were pre-oxidized for one 1-h cycle at 950°C before receiving the first salt coating, and they were weighed before and after salt to assure a salt supply of $1.0 \pm 0.1$ $mg \cdot cm^{-2}$ on each specimen; salt coating was performed after 1 and 100 1-h cycles. A set of Cr-containing NiAl was pre-oxidized for 100 h at 1100°C to form a thick alumina scale.

The oxidation testing was conducted in a similar automatic thermal cycling rig, in slowly-flowing oxygen at 1150°C. In these test cycles, the specimens spent 1 h in the furnace and 10 min cooling. During cooling, specimens were exposed to laboratory air containing natural humidity (not controlled). All specimens were weighed periodically to determine mass change due to oxidation and hot corrosion. Selected specimens were sectioned and examined metallographically at the conclusion of testing.

## 3. Results and discussion

### 3.1. Hot corrosion testing at 950°C

Hot corrosion attack was visible on NiAl-Pt and NiAl-Cr specimens after a few cycles at 950°C. Specimen mass change vs. time curves indicate a significant mass gain after the first inspection interval (20 cycles) for all specimens (Fig. 1). When exposure to hot corrosion conditions was continued, Cr-doped (NiAl-2Cr + Hf and NiAl-5Cr + Hf) specimens formed relatively slow-growing oxide scales, but the Pt-doped (NiAl-2Pt, NiAl-5Pt and NiAl-2Pt + Hf) specimens exhibited poor hot corrosion resistance similar to Hf-doped NiAl. Cast bond coating alloy NiPtAl + Cr + Co demonstrated modest mass gains up until approximately 80 cycles but

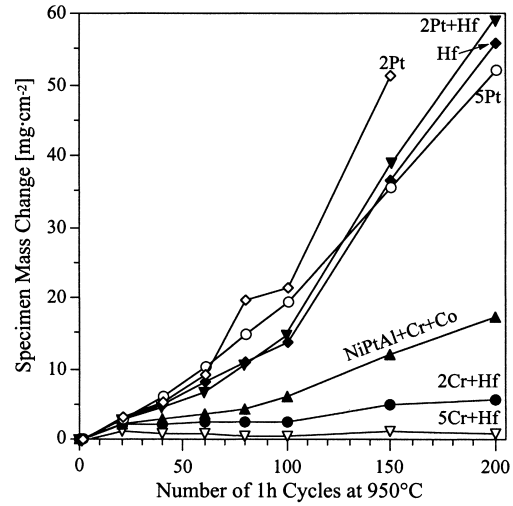


Fig. 1. Specimen mass gain vs. number of 1-h cycles at 950°C for sodium sulfate-coated nickel aluminides. Clearly, Cr rather than Pt provided resistance against hot corrosion attack.

then showed higher mass gains. After 200 h, the oxide scale formed on most of the surface of NiPtAl + Cr + Co was relatively thin and uniform except for near the center of the casting where a voluminous oxide was observed. This heavy local attack was attributed to a casting defect and the thin oxide more representative of the behavior of NiPtAl + Cr + Co. Thus NiPtAl + Cr + Co behaved similarly to Cr-containing NiAl + Hf [12]. Pre-oxidation at 1100°C for 100 h, to form a thick protective alumina scale prior to hot corrosion testing led to further improvement in hot corrosion resistance only for NiAl-2Cr + Hf. As shown in Fig. 2, the initial hot corrosion attack on NiAl-2Cr + Hf was significantly decreased compared with that observed for the non-pre-treated reference specimen, whereas no benefits were obtained for pre-oxidizing NiAl-5Cr + Hf or NiAl-10Cr + Hf. No improvement of hot corrosion resistance of NiAl-2Pt + Hf was obtained by pre-oxidation [12]. In general, hot corrosion resistance improved with increasing Cr content (Figs. 1 and 2), however, the improvement from 5 to 10 at.% was not as great as from 2 to 5 at.%. Notably, as little as 2 at.% Cr was needed to decidedly reduced hot corrosion attack relative to Cr-free Hf-doped NiAl.

Additions of Ir, Ru, or Pd to cast NiAl did not result in an improvement in hot corrosion resistance relative to Hf-doped NiAl (Fig. 3). Rather, somewhat higher mass gains were measured for the precious metal-containing aluminides, presumably a result of extensive crack formation in these alloys, leading to a larger surface area compared with uncracked specimens (Fig. 4). Some of these specimens started to disintegrate after 100 cycles as a result of cracking. Macroscopically, precious metal-containing NiAl showed the formation of voluminous oxides; the volume increase asso-

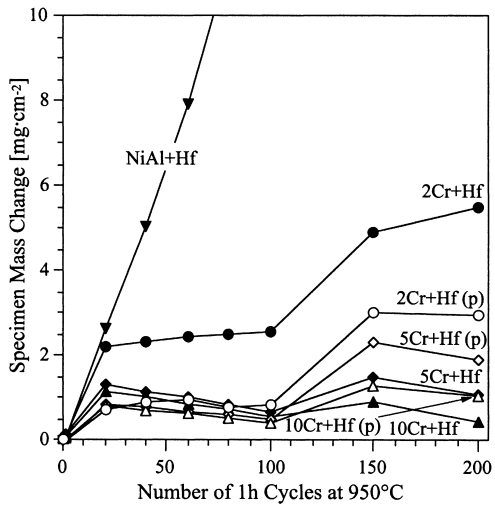


Fig. 2. Specimen mass gain vs. number of 1-h cycles at 950°C for sodium sulfate-coated NiAl-Cr alloys. Pre-oxidation (p) at 1100°C for 100 h improved the hot corrosion resistance of NiAl-2Cr + Hf but had little effect for higher Cr contents.

ciated with internal oxide formation resulted in a visible change in geometrical dimensions in all cases. Again, only Cr provided protection against hot corrosion attack, although the best-performing alloy (2% Cr) was not as hot corrosion resistant as cast NiCoCrAlY (17 at.% Cr), which was included for comparison (Fig. 3) in the absence of data for true Type-I hot corrosion for these alloys.

Metallographic investigations after testing showed significant hot corrosion attack of the specimens in all cases (Fig. 5). Apart from formation of voluminous oxides on some parts of the specimen, NiAl-2Cr + Hf formed a thin protective oxide scale (a). Crack formation and preferential attack along the grain boundaries was found to a lesser extent than for precious metal-containing NiAl (b–d). Pt- and Pd-containing specimens were greatly attacked over the entire cross section with only little metal remaining (b,c). Accelerated attack occurred along grain boundary cracks where corrodants had initially direct access to the metal surface and faced a relatively thin oxide scale during continued exposure. Although not evident from the micrograph displayed in Fig. 4, Ru-containing NiAl formed a network of fine cracks during exposure (Fig. 5d).

Despite the complications caused by the formation of cracks in some alloys, the present results on precious metal- and Cr-containing nickel aluminides indicate that, in these model systems, Cr rather than any other alloying addition improved hot corrosion resistance. There was no indication of any beneficial of Pt or other precious metals on hot corrosion resistance. The reasonable resistance of the cast commercial Pt-aluminide coating composition NiPtAl + Cr + Co to these test conditions, up to the point where secondary effects dominated the attack was, therefore, concluded to be due to the Cr (3.8 at.%) present in the alloy, rather than to Pt. [In CVD coatings, Cr is incorporated from the substrate alloy into the coating during the deposition process]. Based on the current understanding of hot corrosion mechanisms [9,10], it appears that dissolution and reformation of the initially protective alumina scale (fluxing) led to accelerated attack of the metal. The role of Cr is to essentially buffer the salt basicity in a range where alumina solubility is at a minimum. Although the authors are not aware of any literature data on the effect of platinum or other precious metals on the solubility of alumina, the results of hot corrosion tests on Pt-containing NiAl-2Pt, NiAl-2Pt + Hf and NiAl-5Pt compared to NiAl + Hf, for example, suggest that Pt had little or even a somewhat detrimental effect—when only 2 at.% Pt was present — on the protective capacity of the oxide scale. Similarly, none of the other alloying additions appeared to have a beneficial effect on hot corrosion resistance, whereas as little as 2 at.% Cr seemed sufficient to provide reasonable hot corrosion resistance. This result is in good agreement with earlier reports by Ellis [13] and Nesbitt [14] who conducted burner rig tests (Type-I hot corrosion) on variously-doped single crystal nickel aluminides and found that only Cr retarded attack. Higher Cr levels further improved hot corrosion resistance (Figs. 1 and 2) and are, therefore, presumably more beneficial than lower Cr levels for long-term durability.

### 3.2. Oxidation testing at 1150°C

During 1000 cycles at 1150°C, no scale spallation was

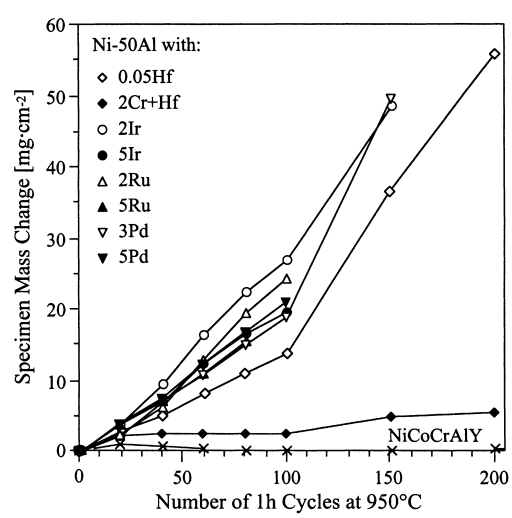


Fig. 3. Specimen mass gain vs. number of 1-h cycles at 950°C for sodium sulfate-coated nickel aluminides. Some specimens were taken out of the test after 100 cycles since they started to disintegrate. For comparison, cast NiCoCrAlY exhibited excellent hot corrosion under these conditions.

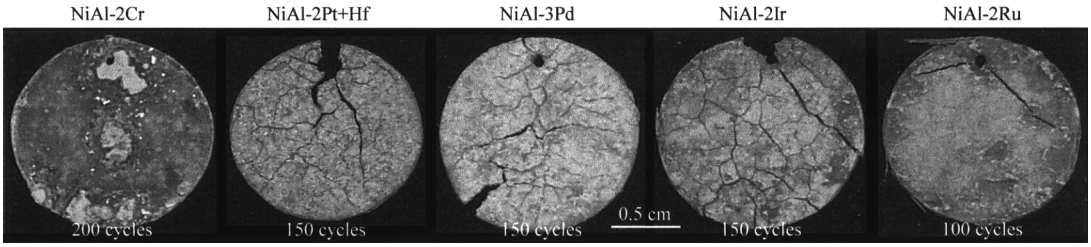


Fig. 4. Micrographs of sodium sulfate-coated NiAl specimens exposed at 950°C between 100 and 200 cycles. Except for NiAl-2Cr + Hf, extensive hot corrosion attack and crack formation was observed.

observed for NiAl + Hf and very little for 2 and 5 at.% Cr-doped NiAl + Hf (Fig. 6). However, the addition of Cr appeared to accelerate the scale formation rate relative to the rate observed for NiAl + Hf. Identical accelerated oxidation rates were measured for 2, 5 and 10 at.% Cr for approximately 250 cycles. In contrast to NiAl-2Cr + Hf and NiAl-5Cr + Hf which continued to gain mass after longer exposures, for NiAl-10Cr + Hf the eventual decrease in the slope of the mass change curve indicated increased scale spallation on NiAl-10Cr + Hf. The model NiPtAl + Cr + Co alloy exhibited significant scale spallation after 150 cycles, again after a period of significantly higher initial mass gain relative to NiAl + Hf. Poor spallation resistance of NiPtAl + Cr + Co might be attributed to the low Al level of the alloy along with the casting defects which altered the hot corrosion mass gains. However, relative to undoped Ni-42.5Al, NiPtAl + Cr + Co (38.19 at.% Al) exhibited better spallation resistance (Fig. 6), demonstrating a beneficial effect of alloying additions, in particular Pt.

Detailed examination of the role of Cr on the oxidation behavior of NiAl + Hf showed that, after 2 h at 1200°C, α-Cr precipitates began to form at the metal-scale interface on NiAl-2Cr + Hf. It was assumed that these precipitates formed due to the rejection of Cr from the growing scale and the low Cr solubility in the β-NiAl matrix [15]. Furthermore, it was suggested that these interfacial precipitates would grow with increased exposure time and finally lead to early spallation of the alumina scale, either due to their different coefficient of thermal expansion or by reduced adhesion of the alumina scale. Most likely, for NiAl-10Cr + Hf exposed

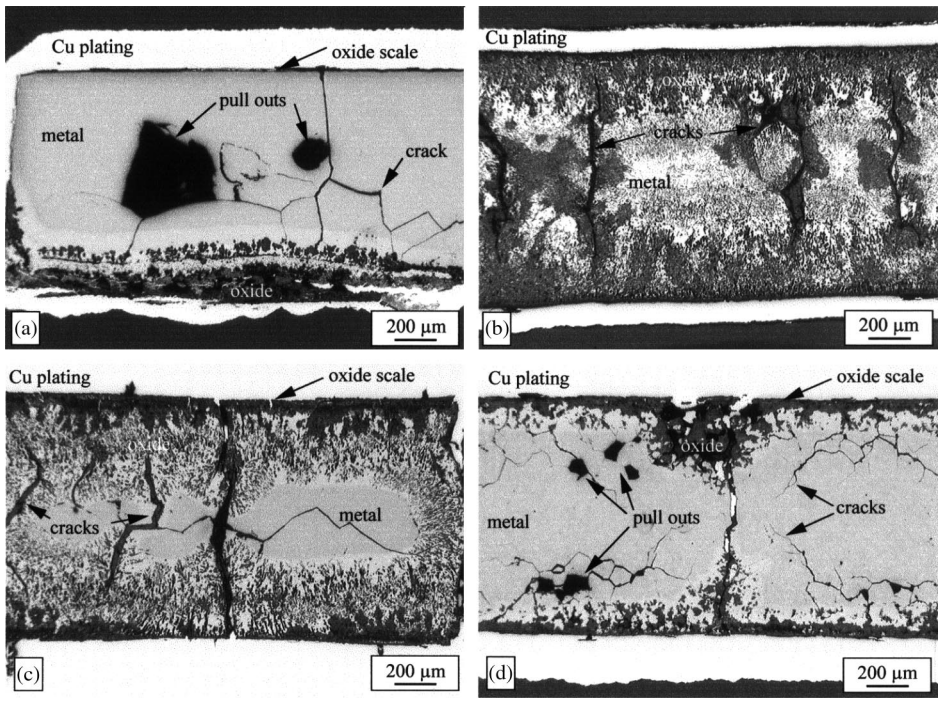


Fig. 5. Metallographic cross section after hot corrosion testing at 950°C of NiAl-2Cr + Hf, 200 cycles (a), NiAl-5Pt, 200 cycles (b), NiAl-5Pd, 100 cycles (c), and NiAl-5Ru, 100 cycles (d). Significant hot corrosion attack of NiAl-2Cr + Hf occurred predominantly locally, whereas precious metal-doped aluminides were attacked more severely. Internal attack and crack formation mainly along metal grain boundaries were present to a large extent. Pull outs were caused by metallographic specimen preparation after the tests.

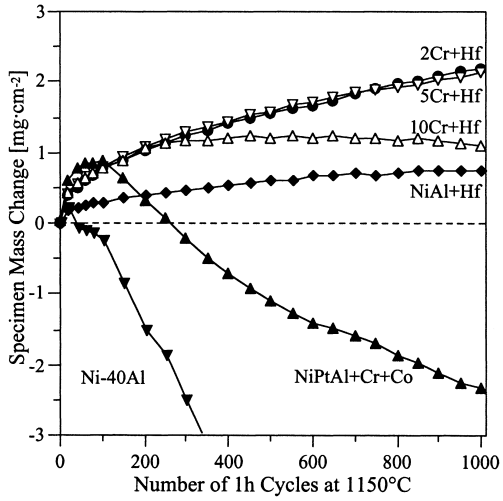


Fig. 6. Specimen mass gain vs. number of 1h cycles at 1150°C tested in flowing oxygen. The addition of Cr to NiAl + Hf decreased its oxidation resistance. 10 at.% were clearly detrimental to oxide scale adhesion. Oxide scale spallation on the NiPtAl + Cr + Co bond coat composition started readily after 100 cycles.

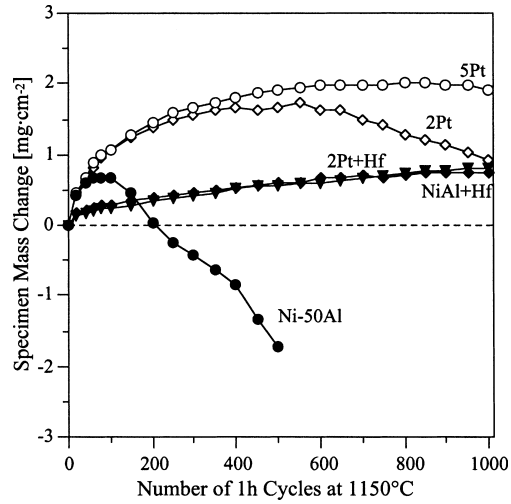


Fig. 7. Specimen mass gain vs. number of 1-h cycles at 1150°C tested in flowing oxygen. The addition of 5 at.% Pt retarded scale spallation for a larger number of cycles than 2 at.% Pt, but only Hf-doping of Pt-containing NiAl provided excellent oxidation resistance.

(at a lower temperature) in the present study, the high level of Cr available at the metal-alumina interface promoted the early onset of scale spallation. However, it is not yet clear why Cr accelerated the oxide scale formation rate (Fig. 6).

The addition of Pt to undoped NiAl improved its oxidation resistance by improving its scale spallation resistance (Fig. 7). As opposed to Cr, increasing the amount of Pt reduced susceptibility to scale spallation. While the oxide scale on NiAl-2Pt began to spall between 300 and 400 cycles, the onset of spallation was retarded and its extent was decreased for NiAl-5Pt. Although the addition of Pt has been demonstrated to improve spallation resistance of NiAl even at 1200°C [16] it was less effective than Hf-doping (Fig. 7). No mechanism has been identified for the beneficial effect of Pt on spallation resistance. Unlike Cr, the addition of Pt to Hf-doped NiAl had no detrimental effect on oxidation kinetics. However, a clear additional benefit of Hf is reducing the scale growth rate. The addition of Pt does not change the scale growth rate compared to undoped NiAl [7], thus by 500 h at 1150°C the scale was more than 10 μm thick which is near a critical alumina thickness where spallation will occur independent of substrate. Doping with Hf reduced the scale thickness after 500 h to 3 μm. No synergistic effect of co-doping with Pt and Hf was apparent.

Addition of 2 and 5 at.% Ir and Pd had a similar beneficial effect as Pt on oxidation resistance relative to undoped NiAl, but again benefits were not as great as for Hf doping (Fig. 8). In contrast, Ru retarded oxide scale spallation only for a limited time before significant mass loss started, and NiAl-5Ru was less oxidation resistant than NiAl-2Ru. While increasing the amount of Ir and Pd in the alloys resulted in greater improvements, the onset of spallation was earlier for NiAl-5Ru than for NiAl-2Ru. The data suggest that Ir and Pd might act in a similar way to Pt in an aluminide coating, whereas Ru would not be as beneficial.

### 3.3. Practical implications

Resistance to oxide scale spallation of NiCrAlY alloys is decidedly inferior to that of reactive element doped β-NiAl [17]. Therefore, reactive element doped nickel aluminides are considered to offer a higher potential for coating performance improvements, espe-

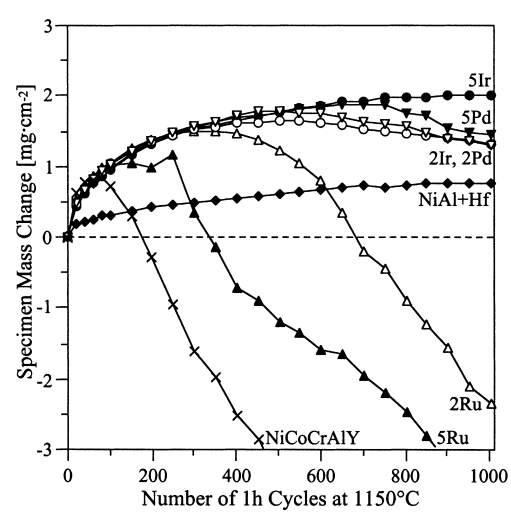


Fig. 8. Specimen mass gain vs. number of 1 h cycles at 1150°C tested in flowing oxygen. Compared to cast NiCoCrAlY, Hf-doped NiAl demonstrated a slow oxide scale growth rate and excellent spallation resistance. None of the precious metal additions improved oxidation resistance as effectively as Hf doping.

cially for future thermal barrier coating applications where ultimate scale adhesion at temperatures significantly exceeding the range of currently used MCrAlY-type bond coats will be required. Even when operating at high temperatures where oxidation rather than hot corrosion is a major problem, degradation of components by contaminants in a gas turbine environment cannot be excluded. This point is important since some components will be exposed to temperatures low enough for hot corrosion attack to occur [18]. Therefore, despite the major design goal to improve high temperature performance of the coated component, reasonable hot corrosion resistance is still required.

From the available data for cast aluminides, it appears that Cr is the only element that improves the hot corrosion resistance in the scenario used in this study, however, at the expense of a higher temperature oxidation resistance. Although Pt-modified aluminide coatings are widely used in practical coating applications, in cast aluminides the only demonstrated benefit of Pt was with regard to oxidation resistance, with no improvement in hot corrosion resistance. Obviously, alumina scale adherence is essential for oxidation resistance of coatings [19]. However, the present data suggest that both oxide scale adhesion and Cr are needed to provide oxidation and hot corrosion resistance. At this time, it appears that combined hot corrosion resistance and exceptional scale spallation resistance is an unattainable goal for nickel aluminide coating performance. As a final note, the specific microstructure of Pt-modified aluminide coatings used in practical applications may play an important role in the observed performance improvements relative to simple aluminides. However, the mechanism by which Pt exerts a beneficial effect in coatings remains unclear. It appears that Pt affects hot corrosion resistance indirectly, by improving scale adhesion compared with a simple aluminide coating.

## 4. Conclusions

From the perspective of coating development for improved high-temperature performance, the test results obtained from cast nickel aluminides highlighted the dilemma that reasonable hot corrosion resistance at lower temperatures (950°C) and exceptional spallation resistance at high temperatures (1150°C) seem to be incompatible goals for advanced coating performance. Pt, Ir, Pd, and, to some extent, Ru improved oxidation resistance of undoped NiAl but were less effective than reactive element doping with Hf. This suggests that for high temperature applications Pt (or other precious metal additions) might be unnecessary if proper Hf-doping can be achieved. Additions of Cr degraded the spallation resistance of all the nickel aluminide variants, and should, therefore, be avoided for optimal high-temperature oxidation resistance; however, Cr appears an essential addition to provide resistance to corrosion by molten salts. Future generations of aluminide bond coats will require careful compositional control to meet significant performance improvement requirements. Further knowledge of the impact of alloying elements to NiAl on both oxidation and hot corrosion resistance is needed, and potential synergistic effects of certain additions have yet to be determined.

## Acknowledgements

The research was sponsored by the German Aerospace Center and the US Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, as part of the Advanced Turbine Systems Program under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation. M. Peters at DLR and J.A. Haynes, D.F. Wilson and J.R. DiStefano at ORNL provided comments on the manuscript which are gratefully acknowledged.

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