

The Platinum Development Initiative:

# Platinum-Based Alloys for High Temperature and Special Applications: Part IV

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*Under the Platinum Development Initiative, platinum-based alloys were being developed for high-temperature and special applications for good corrosion and oxidation resistance. Parts I–III of the present series of papers dealt with the development of the ternary and quaternary Pt alloys (1–3). In this final paper (Part IV), the corrosion behaviour of selected Pt alloys treated with sodium sulfate salt was compared with that of coated and uncoated CMSX-4<sup>®</sup> nickel-based superalloy (NBSA). Scanning electron microscopy (SEM) results showed that protective alumina scales of different integrities formed on the Pt alloys, whereas the NBSAs failed rapidly even with an additional Pt-aluminide protective coating. Although there were pits on the Pt alloys, they were minor and not visible to the unaided eye. The potential for application of these Pt alloys in the aerospace gas turbine industry was assessed based on their hot corrosion performance.*

## Introduction

There is a need for increased operating temperatures in gas turbines for both the aerospace and land-based gas turbine industries which has led to ongoing improvements in materials for these applications (4). There is a demand to further increase working temperatures to 1350°C (5) to allow improved efficiencies and a reduction in fuel consumption (6, 7), thus reducing the levels of carbon dioxide and hydrocarbon emissions which would result in a greener environment. Unfortunately, these desired working temperatures are excessively high for the current alloys, NBSAs, to withstand (8) as they should not be employed at temperatures exceeding 1100°C (9–12). Further temperature increases would result in the dissolution of the strengthening phases, leading to weakening and possible failure of the components. There is a need for new materials with higher melting temperatures, high strengths and good corrosion resistance which could replace NBSAs (10, 11).

High-temperature strength is usually the primary property of interest when considering materials for

application in high-temperature environments, however high-temperature oxidation is equally as important (10). Increasing application temperatures have resulted in continued corrosion problems for high-temperature materials (13). Hence, it is imperative that during the materials selection process corrosion evaluations be taken into consideration. Apart from catastrophic failures corrosion has a serious economic impact. Significant amounts of money are spent on corrosion-related problems and better corrosion control technologies can contribute towards saving money and extending the operational life of the alloys (14).

An increase in NBSA high-temperature strength, achieved by increasing the aluminium (Al) content and reducing the chromium (Cr) content, resulted in alloys that are more susceptible to high-temperature corrosion. Poor corrosion resistance by the high-temperature materials led to the development and introduction of coatings (15). Today, coatings are applied to gas turbine hot section components (as thermal barrier coatings, overlay coatings and diffusion coatings) because they have the capability to withstand temperatures exceeding the substrate melting temperature, while simultaneously reducing the incoming temperatures and protecting the substrate from environmental degradation (16). However coatings are not completely reliable and alternatives are being sought.

Fischer *et al.* (17–20) and Völkl *et al.* (21) stated that problems encountered in the aerospace industry could be solved by using Pt-based alloys because they perform exceptionally well in various high-temperature applications, including areas such as glass manufacturing and the handling of corrosive substances (18, 22, 23). The present work was directed at studying the corrosion behaviour of Pt alloys in comparison with that of coated or uncoated NBSAs at high temperatures.

### Hot Corrosion Behaviour of NBSAs

There are well-documented literature studies on the high-temperature corrosion behaviour of NBSAs and coatings, including ceramic composites, partially stabilised zirconia and diffusion coatings with high aluminium, chromium and silicon contents. However there is limited literature on the corrosion behaviour of Pt-based superalloys since they are relatively new materials, except in cases where Pt is considered as an alloying element in coatings for gas turbines. Most work to date in this area has been conducted

by the Mintek research organisation in South Africa, or one of their members (1–3, 8, 9, 11–13, 17–20, 23–26).

Hot corrosion is an aggravated and accelerated form of oxidation that occurs in the presence of sodium sulfate ( $\text{Na}_2\text{SO}_4$ ) salt at high temperatures (9). The source of  $\text{Na}_2\text{SO}_4$  can be impurities in the air or trace elements in fuels. It is assumed that oxyanions in the molten salt act as the source of sulfur (27) during corrosion attack. The presence of sodium chloride (NaCl) and vanadium pentoxide ( $\text{V}_2\text{O}_5$ ) salts aggravates corrosion by forming low-melting eutectic compounds that are extremely corrosive to high-temperature materials (28, 29).

There are two types of hot corrosion of NBSAs: Type I or high-temperature hot corrosion and Type II or low-temperature hot corrosion. Type I hot corrosion is commonly observed at around 850°C to 950°C, while Type II hot corrosion occurs at 650°C to 800°C (29, 30). The major distinction between these two forms is their degradation morphology (30). Typical Type I hot corrosion morphologies are distinguished by the formation of a non-porous protective scale with internal sulfidation and Cr depletion, and three degradation stages are recognised: incubation, initiation and propagation. The underlying morphology of Type II hot corrosion has pitting of the surface with no internal sulfidation. Illustrations and examples of these hot corrosion occurrences and corrosion morphologies can be found in the excellent paper by Eliaz *et al.*, in which the authors describe different case studies (9). In one instance, severe damage to a NBSA turbine blade was found to result from advanced hot corrosion leading to a preliminary crack and eventual loss of material (Figure 1).

Khajavi and Shariat (31) have demonstrated a transition-type corrosion having characteristics of both Type I and Type II. Eliaz *et al.* (9) identified green colouration in failed components as characteristic of Type I hot corrosion, while Khajavi and Shariat (31) equated it with nickel oxide (NiO) and chromium oxide ( $\text{Cr}_2\text{O}_3$ ) formed in both types of attack. Regardless of the type of hot corrosion, it has detrimental effects and should be detected and minimised at early stages.

Hot corrosion increases the corrosion loss by over a hundred times (28) and failure of materials due to hot corrosion could lead to mechanical loss and subsequent catastrophic failure of components (32). In gas turbines hot corrosion is observed in the low-

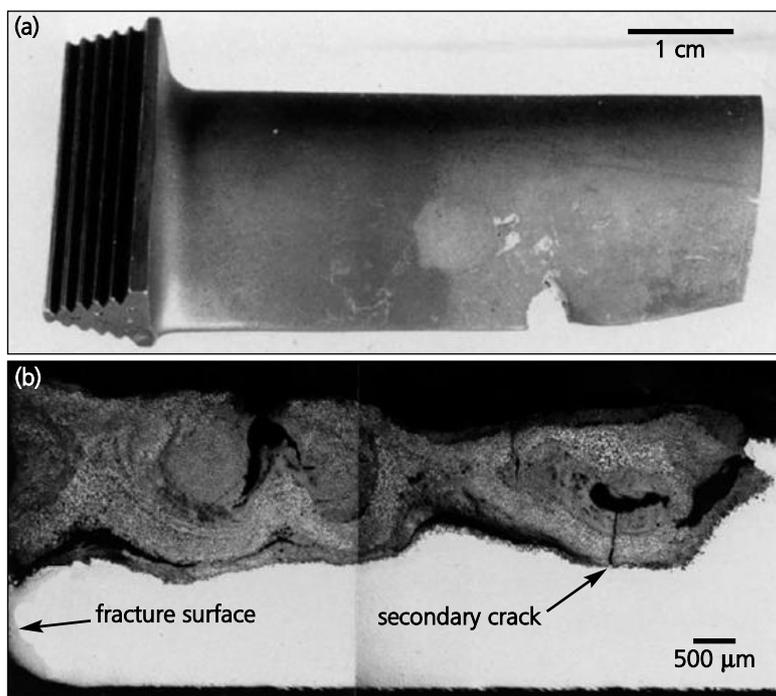


Fig. 1. (a) Macroscopic view of a NBSA turbine blade, showing the loss of a large piece of material along a fracture running perpendicular to the leading edge; (b) Optical micrograph of a cross-section through the origin of the fracture, indicating advanced hot corrosion attack, with a secondary crack propagating from the scale into the base material (9) (Images reprinted with permission from Elsevier)

pressure turbine where contaminants can easily accumulate, rather than in the high-pressure turbine (9, 27). Factors that influence hot corrosion attacks on alloys are alloy composition, thermomechanical properties, contaminant composition, flux rate, operating temperatures, temperature cycles, gas composition, gas velocity and erosion.

#### Coatings for High-Temperature Materials

In general, coatings extend the life of components as they preserve the mechanical properties of the underlying substrate and simultaneously protect the substrate from degradation mechanisms such as erosion, wear and hot corrosion (6, 15). Coatings rely on the formation of slow-growing, stable, dense and adherent oxide scales (33, 34). For example  $\alpha$ -alumina acts as a diffusion barrier to minimise the diffusion effect of substrate elements (5, 35) owing to its high thermal stability at high temperatures (33) and low solubility in molten salts (36).

Yttria-stabilised zirconia (YSZ) thermal barrier coatings are used in the aerospace industry to protect blades, vanes, gas turbine shrouds and combustion cams (6) in gas turbine engines. Overlay coatings based on MCrAlY (M = Ni and/or Co) are preferred in the most demanding applications because of their excellent diffusional and environmental sta-

bility (16). Their high Cr content makes them useful for protection against Type II hot corrosion. MCrAlY-based bond coatings can be applied as thermal barrier coatings and are resistant to oxidation and hot corrosion (15). Pt-modified coatings such as Pt aluminide are preferred for protection against Type I hot corrosion with improved scale adherence and diffusion protection (37). It is anticipated that the addition of other platinum group metals to Pt-modified coatings would be beneficial based on the use of such Pt alloys for handling molten glass (17). In general better oxidation and hot corrosion resistance are achievable against molten sulfates (14, 38) with coatings than without, and uncoated materials experience excessive weight gains from the formation of non-protective corrosion compounds on their surface (38).

One problem with coatings is that different types of corrosion can occur at different temperatures. 'Smart coatings' based on MCrAlY, a functionally gradient coating system formed by first enriching in Cr and then in Al to provide the chemically graded structure, were developed to overcome this problem. Unlike other high-temperature coatings, smart coatings have the capability to resist both Type I and Type II hot corrosion. Nicholls *et al.* (39) showed that these coatings exhibited good corrosion resist-

ance when compared with a Pt-modified aluminide coating, RT-22<sup>®</sup>, and a multilayered silicon aluminide coating, SermeTel<sup>®</sup> 1515.

There is much work currently being conducted to match coating properties with gas turbine requirements. In the meantime gas turbines will continue to use thermal barrier coating technology (29,33,40–43) to allow for increased inlet temperatures, improved hot corrosion resistance and long-term stability. Coatings are still the best choice to resist hot corrosion in high-temperature materials, adding value up to ten times the cost of the coatings (29).

Thermal cycling and thermal shock can be detrimental for coatings. Variations in thermal expansion coefficients can lead to thermal stresses and subsequent cracking and spalling of coatings. Even the most resistant alloys are susceptible to hot corrosion attacks (44) and no coatings are completely reliable. It is becoming increasingly difficult to design coatings suitable for high-temperature applications due to the lack of compatibility of the thermal expansion coefficients between coatings and high-temperature alloys. There remains a need for further development of high-temperature corrosion resistant materials to overcome these drawbacks.

### Platinum-Based Superalloys for Jet Engine Applications

Platinum was selected and developed for jet engine applications because of its higher melting point compared to nickel (Ni), similar face centred cubic (fcc) structure and better corrosion resistance (1–3, 24). The investigation into potential uses of Pt-based

superalloys has been limited partly by their high cost. Density is also important, especially where rotating components are concerned (45, 46). Lightweight materials are desired because centrifugal forces can be diminished at low densities. This places Pt in a compromising position because of the element's high density compared to Ni (21.45 g cm<sup>-3</sup> for pure Pt compared to 8.91 g cm<sup>-3</sup> for Ni). However, additions of alloying elements such as Ni and cobalt (Co) could effectively reduce the density of the alloy (12, 47) albeit by small amounts. The high cost could be counteracted by the fact that Pt is recyclable and with its good corrosion resistance the alloy would allow for little or no application of coatings (22). Subsequent benefits such as reduction in fuel cost and reduced emissions would also follow.

### Corrosion Testing of Platinum-Based Superalloys

Corrosion testing of Pt and its alloys for the glass industry is well documented (see for example (25)). Pt-based superalloys show potential to be candidates for at least partial replacement of NBSAs based on their mechanical properties (1–3). The high-temperature corrosion behaviour of these Pt-based superalloys was studied by performing a crucible test (26) at 950°C to increase the corrosion kinetics. This temperature was selected because it is within the temperature range at which hot corrosion has the greatest effect (37, 48, 49). The samples consisted of five Pt-based superalloys containing Pt and Al with Cr, Co and/or ruthenium, as listed in Table I, and two samples of a single-crystal CMSX-4<sup>®</sup> NBSA of composi-

Table I

Nominal Chemical Compositions of Selected Platinum-Based Superalloys and Nickel-Based Superalloys Together with Their Corrosion Kinetics after Treatment in Na<sub>2</sub>SO<sub>4</sub> at 950°C for 564 Hours

Alloy name	Nominal composition, at%	Cumulative weight gain during corrosion, mg cm <sup>-2</sup>
RS-1	Pt <sub>86</sub> :Al <sub>10</sub> :Cr <sub>4</sub>	0.00004
RS-2	Pt <sub>86</sub> :Al <sub>10</sub> :Ru <sub>4</sub>	0.00008
RS-3	Pt <sub>84</sub> :Al <sub>11</sub> :Cr <sub>3</sub> :Ru <sub>2</sub>	0.0001
P420	Pt <sub>79</sub> :Al <sub>15</sub> :Co <sub>6</sub>	0.0001
P421	Pt <sub>73</sub> :Al <sub>15</sub> :Co <sub>12</sub>	0.004
CMSX-4 (uncoated)	Ni <sub>66.5</sub> :Cr <sub>6.5</sub> :Co <sub>11</sub> :Mo <sub>0.3</sub> :W <sub>1.7</sub> :Ta <sub>1.8</sub> :Al <sub>11.3</sub> :Ti <sub>0.9</sub>	0.470
CMSX-4 (coated)	Ni <sub>66.5</sub> :Cr <sub>6.5</sub> :Co <sub>11</sub> :Mo <sub>0.3</sub> :W <sub>1.7</sub> :Ta <sub>1.8</sub> :Al <sub>11.3</sub> :Ti <sub>0.9</sub>	0.038

tion (in at%) Ni<sub>66.5</sub>:Cr<sub>6.5</sub>:Co<sub>11</sub>:Mo<sub>0.3</sub>:W<sub>1.7</sub>:Ta<sub>1.8</sub>:Al<sub>11.3</sub>:Ti<sub>0.9</sub>. Starting weights were approximately 2 g. A thin Pt aluminide coating (Pt<sub>2</sub>Al–Pt<sub>67</sub>:Al<sub>33</sub>, in at%) of approximately 1.25 µm-thickness had been deposited on one of the CMSX-4 samples while the other was uncoated.

All samples were covered in analytical anhydrous Na<sub>2</sub>SO<sub>4</sub> salt, which acted as the corrosive electrolyte inside a 20 ml high-alumina crucible that was placed inside a furnace with a static dry air environment. The test was performed for an initial 60 cycles of 1 hour of heating and 20 minutes of cooling to room temperature, followed by up to 11 subsequent long cycles of 72 hours, giving a maximum total of 852 hours of heating. Samples were washed free of salt residues and weighed at the end of each long cycle and fresh salt was provided for every cycle (26). Table I shows the cumulative weight gains during the first 60 cycles of 1 hour, plus a further 7 cycles of 72 hours (564 hours in

total). Weight changes for the first 50 hours are plotted in Figures 2 and 3.

The corrosion morphology and the cross-sectional analysis of the corroded alloys were characterised after 564 hours and again after 852 hours, by scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD) and Raman spectroscopy being used to confirm the phases.

The mass gains of samples that had been immersed in Na<sub>2</sub>SO<sub>4</sub> at 950°C for 852 hours indicated that there was very little change for the Pt-based alloys because of the formation of a protective scale on their surface. In the case of the uncoated NBSA there was initial mass gain from the formation of oxides, and further reactions and the formation of non-protective oxides resulted in catastrophic corrosion which led to large mass changes. Testing of the uncoated NBSA was discontinued after 564 h as the sample was degrading

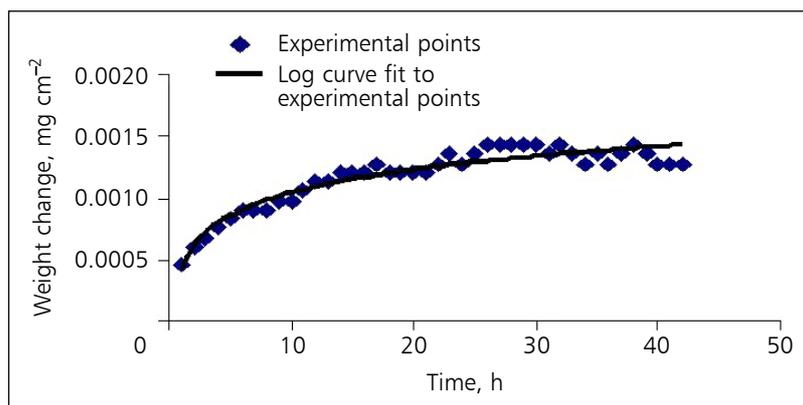


Fig. 2. Corrosion kinetics of the coated NBSA during exposure to Na<sub>2</sub>SO<sub>4</sub> at 950°C for the first 50 hours

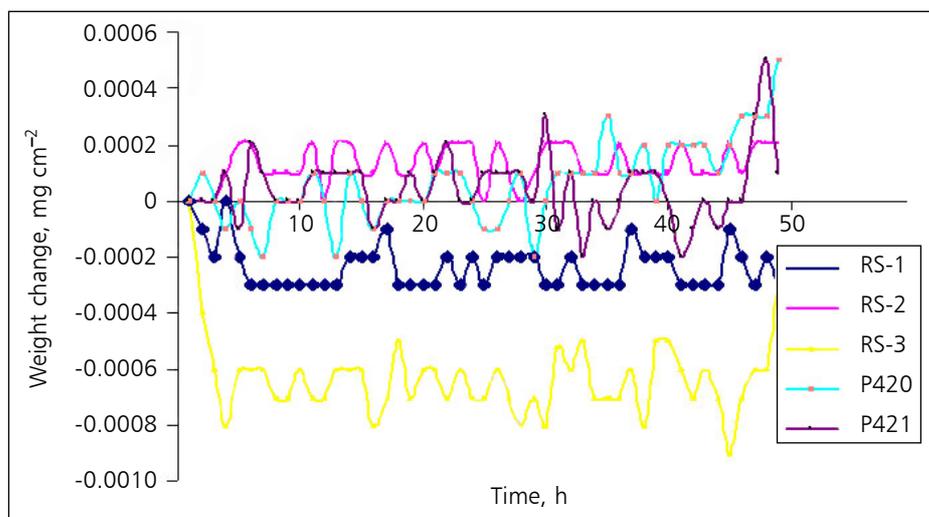


Fig. 3. Corrosion kinetics of five Pt-based alloys of various compositions for the first 50 hours

exponentially at this point. The coated NBSA offered better resistance than the uncoated one, up to the first testing stage after 564 hours, but eventually also degraded before the final testing time of 852 hours was reached and its further testing was therefore also discontinued. This experiment demonstrated that the Pt alloys have good hot corrosion resistance in the presence of molten sodium sulfate salt (Figures 2 and 3), outperforming both a coated and an uncoated NBSA (26).

XRD analysis of the corrosion products on the NBSA samples showed a mixture of compounds based mainly on Na and Ni. Conversely for the Pt-based superalloys XRD analysis showed mainly alumina ( $\text{Al}_2\text{O}_3$ ), which is the oxide coating usually present under atmospheric conditions. This was a good indication that the Pt-based substrate was supporting the protective  $\text{Al}_2\text{O}_3$  layer well. The corrosion morphologies of the Pt-based alloys were much better than those of CMSX-4<sup>®</sup>. Pt<sub>86</sub>:Al<sub>10</sub>:Cr<sub>4</sub> had the best appearance and Pt<sub>84</sub>:Al<sub>11</sub>:Cr<sub>3</sub>:Ru<sub>2</sub> and Pt<sub>86</sub>:Al<sub>10</sub>:Ru<sub>4</sub> were slightly pitted, with the latter showing more pits. Pt<sub>79</sub>:Al<sub>15</sub>:Co<sub>6</sub> appeared good in some places with losses in other places. However it should be noted that in all cases the pits were very small, and can be considered negligible when compared with the damage to the NBSA samples under the same experimental conditions. With the naked eye no change in appearance could be seen in the Pt-based alloy samples (26).

These findings were confirmed by SEM and both the coated and uncoated CSMX-4<sup>®</sup> samples suffered

a greater degree of attack than the Pt-based superalloys. Both NBSA samples suffered from acidic fluxing, forming voluminous non-protective oxide scales. The corroded morphologies of Pt<sub>73</sub>:Al<sub>15</sub>:Co<sub>12</sub> and Pt<sub>79</sub>:Al<sub>15</sub>:Co<sub>6</sub> showed a disintegrated scale layer, indicating that the scale was not protective in this environment. The scale morphologies of Pt<sub>86</sub>:Al<sub>10</sub>:Cr<sub>4</sub>, Pt<sub>86</sub>:Al<sub>10</sub>:Ru<sub>4</sub> and Pt<sub>84</sub>:Al<sub>11</sub>:Cr<sub>3</sub>:Ru<sub>2</sub> were similar to each other (Figure 4). They were more tenacious and complete although apparently porous and gave more protection against hot corrosion than in the Pt-based superalloys with Co (26).

The resistance to sulfidation of the alloys investigated correlated with their corrosion resistance to hot  $\text{Na}_2\text{SO}_4$  salt at 950°C, with the Pt alloys with Cr and/or Ru being more corrosion resistant than those containing Co. The NBSAs showed the lowest resistance and disintegrated in a short time (50, 51). NBSAs suffered internal sulfidation, forming Cr and Ni sulfide corrosion compounds. Raman spectra analysis suggested that sulfate salts with possible traces of nitrates formed on the surfaces of the Pt-based superalloys.

#### Future Prospects for Platinum-Based Superalloys

The Pt-based superalloys studied here display a high potential for successful application as high-temperature corrosion resistant materials particularly for applications such as gas turbines for the aerospace industry and possibly also for other high-temperature

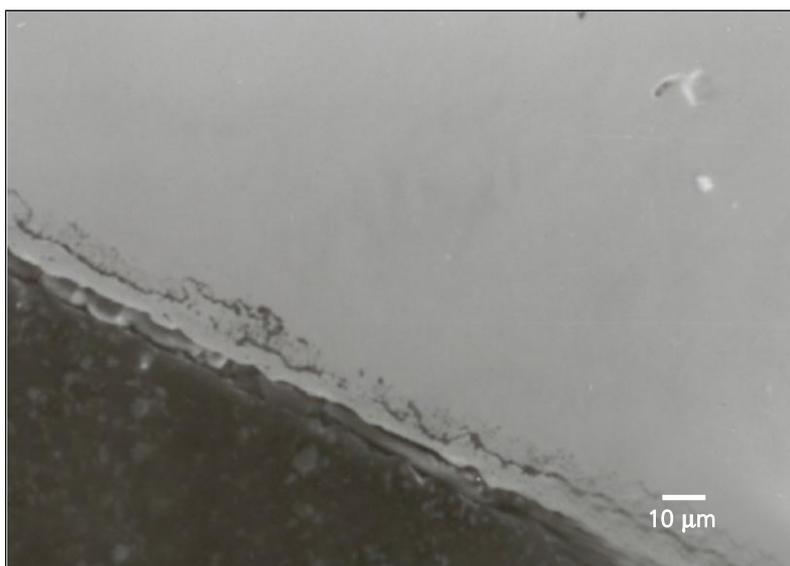


Fig. 4. SEM secondary electron image showing the thin protective scale on the surface of the platinum-based superalloy RS-1, Pt<sub>86</sub>Al<sub>10</sub>Cr<sub>4</sub>

processes (52). Due to the drawback of their high density and subsequent weight the most successful option for future exploitation would probably be as coatings on suitable substrate materials, as Pt used as an alloying element in coatings has been shown to give good protection against hot corrosion in laboratory tests (37). This concept is being explored further with the development of new Pt-based superalloys currently under investigation at Mintek in South Africa.

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