High Temperature Oxidation of Pt–Al–Cr–Ru Alloy: Scale Morphology and Adherence

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Abstract Heat-treated Pt–11Al–3Cr–2Ru (at.%) alloy samples were oxidized in air for periods between 1 and 500 h in a muffle furnace at 1350 °C. The morphology of the oxidized samples was examined using light microscopy and scanning electron microscopy equipped with energy dispersive spectroscopy, while an x-ray diffractometer and Raman spectroscopy were used for phase identification. The scale layers were found to be composed mainly of α -Al₂O₃; they were adherent, continuous, and protective with neither a zone of discontinuous oxides nor any other internal oxidation. The alumina scale also grew from a submicron thick thin film after 1 h of oxidation to form large irregular-shaped grains after 500 h exposure, indicating increased scale thickness with oxidation time. Good adhesion of the α -Al₂O₃ scale to the substrate of the current alloy was found to be dependent on the mechanical keying of the scale to the substrate due to protrusions of the alloy into the scale, which gave rise to an irregular scale–substrate interface. The results suggest that the current

alloy has a promising potential for high-temperature applications.

Keywords High temperature · Oxidation · Pt–Al–Cr–Ru alloy · SEM · XRD · Raman spectroscopy · Scale adherence

Introduction

High-temperature environments, such as hot sections of turbine engines, contain sufficient reactive gaseous species that nearly all metals and alloys are thermodynamically unstable and form oxides, sulfides, nitrides, or carbides through an oxidation process [1]. Thus, many high-temperature materials, such as nickel-based superalloys, rely on a slow-growing α -Al₂O₃ scale for oxidation protection [2]. This scale serves as a barrier which inhibits further reaction between the substrate metal/alloy and the environment, prevents degradation of mechanical properties and subsequent failure of the components. However, the alumina scale must remain adherent to the substrate, if it is to remain effective and ensure the viability of the substrate alloys/metals for long-term application in turbine engine environments.

Nickel-based superalloys, which are materials currently being used for production of hot section components of turbine engines, have nearly reached their temperature limit. This is because these alloys in some engine applications have already been exposed to temperatures up to 90% of the melting temperature of the base metal [3]. Precipitation-hardened Pt-based alloys with microstructures similar to Ni-based superalloys (NBSAs) are being developed to operate at higher temperatures than NBSAs, up to 1350 °C [4–8]. These alloys are to serve as a possible

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replacement for some of the currently used nickel-based superalloy components in hot-sections of gas turbines, to improve efficiency of the engine, achieves greater thrust, and to reduce fuel consumption and CO₂ emissions [3]. Thus, in this study, the adherence of alumina scale formed during high-temperature oxidation of a Pt–Al–Cr–Ru alloy is examined to determine its suitability for high-temperature applications. This will assist in predicting the long-term reliability of the scale for oxidation protection, and will also help in determining the possible operating performance of the materials, because high-temperature oxidation effects become more significant with increasing operating temperatures.

Felten and Pettit [9] studied the oxidation behavior of binary Pt–Al alloys between 1000 and 1450 °C, and found that continuous and well-adhering α -Al₂O₃ scales developed on the alloys containing between 0.5 and 6 wt.% Al. From their findings, the good adherence of the alumina scale on the Pt–Al alloys was proposed to be due to the mechanical keying resulting from the protrusions of the scale into the substrate. Ternary Pt–Al–X alloys (where X = Cr, Ru, Ir, Re, Ta, and Ti) were observed to display promising oxidation behavior by forming a protective external oxide scale after being isothermally oxidized in air at 1250, 1280, and 1350 °C, for up to 500 h [10]. In another study, Pt–Al–Z alloys displayed better oxidation resistance than the other ternary Pt–X–Z alloys (where X = Ti, Ta, Nb, and Z = Ru, Ni, and Re), due to the formation of an external protective alumina scale layer [11]. Isothermal oxidation tests on Pt–10Al–4Z (at.%) alloys (where Z = Ir, Cr, Ru, and Ti) in air at 1350 °C revealed that the stable protective alumina layer formed after an initial transient period, during which discontinuous internal aluminum oxides were precipitated in the Pt–10Al–4Cr and Pt–10Al–4Ir (at.%) alloys [12]. Süss et al. [13] isothermally oxidized Pt–14Al–3Cr–3Ru (at.%) alloy in air at 1350 °C for up to 100 h. External alumina scales, with no internal oxidation or zone of discontinuous oxides, were developed on the alloy. These scales were observed to be about three times thicker than the scale formed on Pt–10Al–4Cr [12] after 10 h, indicating better oxidation resistance. Wenderoth et al. [14] also observed formation of well-adhering external α -Al₂O₃ scales on Pt–12Al–6Cr–5Ni (at.%) alloys during isothermal oxidation in air between 1100 and 1300 °C for up to 400 h.

A Pt-based alloy with composition Pt–11Al–3Cr–2Ru (at.%) was selected for this study based on the previous results from research groups at the University of the Witwatersrand and Mintek [4, 5], which showed that this alloy has promising mechanical properties due to the presence of the desirable two-phase γ/γ' microstructure, higher γ' precipitate volume fraction and hardness values. This quaternary alloy composition was based on the results

by Hill et al. [10, 11] and Süss et al. [12, 13], which showed that Pt–10Al–4Cr and Pt–10Al–4Ru (at.%) alloys have promising high-temperature mechanical strength and oxidation resistance. The presence of Al improves the oxidation properties of the alloy [10], and is necessary to form L1₂ (γ') phase [11]. Addition of Cr as a ternary element was found to stabilize the cuboidal L1₂ (γ') phase, while Ru acted as a solid solution strengthening agent [11, 12]. Recent studies by Odusote et al. [15] have shown that the current alloy exhibited considerably better oxidation behavior than the ternary Pt–Al–Cr and Pt–Al–Ru alloys [12, 13]. This was attributed to the formation of continuous and protective α -Al₂O₃ scales, with no zone of discontinuous oxides or any internal oxidation. The aim of this study is to determine the nature of the scale formed on Pt–11Al–3Cr–2Ru (at.%) alloy during isothermal oxidation at 1350 °C, and the influence of the scale morphology on its adherence.

Experimental Procedures

The Pt–11Al–3Cr–2Ru (at.%) alloys were manufactured by arc button melting the pure (at least 99.9% purity) constituent elements and turning the buttons several times under an argon atmosphere to achieve homogeneity. The as-cast samples were then heat-treated at 1350 °C for 96 h in a muffle furnace followed by air cooling. Approximately 1 mm thick slices were cut from the as-received ingots using electron discharge machining; they were then metallographically prepared, as reported by Bill et al. [16].

Oxidation was performed at 1350 °C in air, using a pre-heated front-loading muffle furnace. Specimens were taken out of the furnace after various time intervals from 1 to 500 h, and the weight gains were measured. Prior to cross-sectional examination, the specimens were mounted in Bakelite, and polished down to 1 μ m diamond suspension finish.

Surface morphologies of the scales were examined using light microscopy and a field-emission scanning electron microscope equipped with a dry silicon drift energy dispersive x-ray spectroscopy (EDS) detector. Scanning electron microscopy was also used for cross-sectional examination of the oxidized specimen morphologies, using the secondary electron (SE) mode. The specimens were carbon coated prior to examination, to reduce the charging and improve the conductivity of the oxide scales. The scale thicknesses were measured during examination of the cross section. Phase compositions of the oxide scale on the surface of the oxidized specimens were identified using an x-ray diffractometer with a Cu K α radiation and Raman spectroscopy.

Results

Surface Morphology of the Oxide Scale

Figure 1 shows light micrographs of the surfaces of Pt–11Al–3Cr–2Ru (at.%) specimens oxidized in air at 1350 °C for up to 500 h. Well-adhering oxide scales, which showed no evidence of spallation during oxidation or after cooling in air, were formed on the specimens at all exposure times. The appearance of the grain boundaries of the substrate on the oxide scales was possibly due to the non-uniformity as well as the thin nature of the scale. However, as the scale grows in thickness with increased exposure time, the grain boundaries disappear.

Surface morphology of the oxidized specimens, as revealed by SEM in Figs. 2 and 3, shows that the oxide scales were tightly adherent to the substrate with no evidence of spallation. The morphology of the scales changed with increased exposure times, with the pores becoming more rounded and less discernible, while the oxide grain size also increased. After 200 h of oxidation, large and more rounded oxide grains of about $2.0 \pm 0.4 \mu\text{m}$ grain sizes had grown (Fig. 4). EDS (Fig. 5) shows that the scale layers are composed mainly of Al and O above 1 h exposure.

Cross Section of the Oxide Scale

The SEM–SE cross sections (Figs. 6, 7) show that the scales developed on Pt–11Al–3Cr–2Ru (at.%) after oxidation at 1350 °C are continuous, with limited porosity. The scale thicknesses increased with increased oxidation time, and they are non-uniform as indicated by the error bars in Fig. 8. The scale–substrate and scale–gas interfaces were irregular, with no apparent relationship to each other (Figs. 6, 7). No zone of discontinuous oxide or evidence of any internal oxidation was observed. In addition, there was no formation of voids at the scale–alloy substrate interface, indicating that the scales were adhering well and potentially protective. Ridges or intrusions of oxide scale, which gave rise to an irregular interface between the substrate alloy and the scale, were observed to increase with increasing exposure time as shown in Figs. 6 and 7. The oxide scales were also observed to protrude outward at the scale–gas interface.

Phase Identification

X-Ray Diffraction

XRD patterns of all the specimens after oxidation in air at 1350 °C for up to 500 h are presented in Fig. 9. It was

evident that the oxide scales have $\alpha\text{-Al}_2\text{O}_3$ (corundum) as the principal phase. The alumina peak intensities increase with increased exposure times. Diffraction peaks corresponding to (Pt) matrix and $\text{L}_{12}\text{-Pt}_3\text{Al}$ phases were also found, even at longer times. However, the intensities of these peaks decrease with increased exposure time, presumably due to the increase in oxide scale thicknesses. Very low intensities peaks of PtO_2 and CrO_3 were observed at exposure times up to 10 h.

Raman Spectroscopy

The Raman spectra of the oxide scales on the air-cooled specimens isothermally oxidized at 1350 °C for various time intervals up to 500 h in air, revealed the presence of corundum ($\alpha\text{-Al}_2\text{O}_3$) at all exposure times, as shown in Fig. 10. The intensities of the corundum peaks increase with increased exposure times across the oxidation temperatures, indicating an increase in scale thickness. The peak at wavenumber 417.4 cm^{-1} , corresponding to the strongest peak in the reference corundum, was also detected as the strongest peak. Again, no peaks matching PtO_2 or CrO_3 were detected. This may be due to their low concentrations in the scales, or the depth of penetration of the Ar^+ laser probe.

Discussion

EDS shows that the scale layers on oxidized air-cooled Pt–11Al–3Cr–2Ru (at.%) specimens are composed of Al and O (Fig. 5), corresponding to $\alpha\text{-Al}_2\text{O}_3$, as detected by XRD (Fig. 9) and Raman spectroscopy (Fig. 10). This is similar to observation on the water-quenched Pt–11Al–3Cr–2Ru (at.%) specimens after oxidation in air at the same temperature [15]. The scale is slow growing, continuous, well-adhering, and protective with relatively few defects (Figs. 1–4, 6, 7). The oxidation temperature and the high Al contents of the oxidized specimens favoured the formation of protective $\alpha\text{-Al}_2\text{O}_3$, rather than the less protective transition aluminas [17]. Development of $\alpha\text{-Al}_2\text{O}_3$ scale is accelerated on alloys oxidized above 1000 °C, with a sufficiently high Al contents (above 4 wt.%) [17].

The surface morphology of the scale layers also revealed that alumina scale formed at longer exposure time appeared more crystalline and coarser, and thus exhibited a morphology different from those formed at lower temperatures [18, 19]. This observation can be explained by an increase in scale thickness with increasing oxidation time, as shown in Fig. 8.

Cross-sectional examination of the scale layers shows the protrusion of the alloy substrate into $\alpha\text{-Al}_2\text{O}_3$ scale (Figs. 6, 7), giving rise to an irregular interface between the

Fig. 1 Light micrographs showing surface morphology of oxidized Pt-11Al-3Cr-2Ru (at.%) specimens after isothermal oxidation in air at 1350 °C for up to 500 h

Fig. 2 Surface SEM–SE images of Pt–11Al–3Cr–2Ru (at.%) after 100 h oxidation in air at 1350 °C

Fig. 3 Surface SEM–SE images of Pt–11Al–3Cr–2Ru (at.%) after 200 h oxidation in air at 1350 °C

alloy substrate and the scale. The irregular scale–substrate interface increases with increasing exposure time, thereby leading to possible improvement of scale adhesion to the substrate. This is considered to be due to a mechanical keying mechanism, as proposed by Felten and Pettit [9] during oxidation of Pt–Al alloys.

The cross-sectional images also reveal that there is no discernable formation of pores at the scale–substrate interface and no scale spallation, indicating that the scales are potentially protective and adhesive. Tien and Pettit [20] reported that elimination of interfacial cavities improved the scale–substrate adhesion in an yttrium-doped Fe–Cr–Al alloys, such that oxide spallation was not observed. Similarly, no oxide spallation was observed in an Hf-doped Ni–15.1Ta–6.7Al (wt.%) alloy, and oxide protrusions of HfO₂

Fig. 4 Surface high-magnification SEM–SE images of Pt–11Al–3Cr–2Ru (at.%) after 200 h oxidation in air at 1350 °C

were enveloped with a thin layer of Al₂O₃, which provided improved scale adhesion to the alloy [21]. In contrast, a Ni–15.1Ta–6.7 Al (wt.%) alloy doped with 0.04Y showed spallation of oxide scale during cooling. This showed that the thermally induced stresses must have exceeded the cohesive strength of the scale–substrate interface [22].

A number of studies have shown that the presence of Pt in alloys such as Ni–8Cr–6Al [23], Ni–12Cr–9Co (RMJ 2012) and (IN792 +Hf) [24], FeCrAlY, NiCrAlY, and NiCoCrAlY [25] and various single phase and duplex iron-based and Ni-based superalloys [25] improves their oxide scale adherence. It was proposed that the adherence was achieved by mechanical keying of the scale to the substrate due to irregularity of the oxide scale [9, 22], or as a result of a decrease in the tendency to form convoluted scale, i.e., localized scale detachment from the alloy [26]. Development of convoluted scale is caused by the scale growth mechanism, leading to development of compressive stress, and detachment of the scale from the alloy [27–29].

Incorporation of Pt-rich crystals from volatile PtO₂ have been reported to significantly enhance the isothermal stability of α -Al₂O₃ scale, either by influencing the stress relief mechanisms or reducing oxidation growth stress generation [25]. The stress relief mechanism may be achieved through enhanced diffusional creep or grain boundary sliding of the alumina scale, while the reduction in growth stress generation may be by a mechanism similar to those proposed to explain the influence of reactive elements, such as yttrium or zirconium on the growth and adhesion of Al₂O₃ scales on Fe–Cr–Al and Ni–Cr–Al superalloys, as explained by Fountain et al. [25]. Reactive elements improve the scale adherence through the development of inward growing pegs of the oxides of the reactive-elements, which are incorporated in the alumina scale, leading to irregular scale–alloy interface.

Fig. 5 Surface EDS spectrum of air-cooled Pt–11Al–3Cr–2Ru (at.%) oxidized in air for 10 h at 1350 °C, showing the presence of Al and O

Fig. 6 SEM–SE cross-sectional image of oxide scale on Pt–11Al–3Cr–2Ru (at.%) after 50 h oxidation in air at 1350 °C

Fig. 8 Graph showing the variation of thickness with time during oxidation of Pt–11Al–3Cr–2Ru (at.%) in air at 1350 °C. The error bars show that the scale thicknesses are non-uniform

Fig. 7 SEM–SE cross-sectional image of oxide scale on Pt–11Al–3Cr–2Ru (at.%) after 200 h oxidation in air at 1350 °C

Furthermore, Gleeson et al. [30] showed that the addition of Pt to β -NiAl and γ' -Ni₃Al + γ -Ni alloys promoted the formation and adhesion of Al₂O₃ scale, although the adhesion mechanisms were not reported.

Conclusions

The results from the isothermal oxidation study in air at 1350 °C has revealed the formation of external α -Al₂O₃ scale on the air-cooled Pt–11Al–3Cr–2Ru₂ (at.%) specimens. There was no scale spallation during oxidation and after cooling, which is typical for protective oxide scale. In addition, the alumina scale formed was adherent and

Fig. 9 XRD patterns of air-cooled Pt-11Al-3Cr-2Ru (at.%) specimens after isothermal oxidation in air at 1350 °C for 500 h

Fig. 10 Raman spectra of the oxide scales formed on air-cooled Pt-11Al-3Cr-2Ru (at.%) specimens after isothermal oxidation in air at 1350 °C for up to 500 h

continuous, with no evidence of internal oxides or any zone of discontinuous oxides.

Scale thickness increases with increase in oxidation time, and grows from a thin film of oxide to large and more rounded oxide grains. Mechanical keying of the scale, resulting from alloy protrusions into the scale and scale intrusions into the substrate during oxidation, is responsible for good adhesion of the α -Al₂O₃ scale. Thus, the current alloy has a promising potential for high-temperature applications.

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