



Comparison of Chromia Growth Kinetics in a Ni-based Superalloy, with and without Shot-peening



S. Cruchley^a, M.P. Taylor^{a,*}, R. Ding^a, H.E. Evans^a, D.J. Child^b, M.C. Hardy^b

^a School of Metallurgy and Materials, University of Birmingham, Birmingham B15 2TT, UK

^b Rolls-Royce plc, Derby DE24 8BJ, UK

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ABSTRACT

The effect of shot-peening on the oxidation in air of the Ni-based superalloy RR1000 has been investigated over the temperature range 700–800 °C. The surface oxide in both peened and un-peened conditions consisted of isolated grains of rutile on the outermost surface beneath which was a protective Ti-doped chromia scale. Internal oxidation of aluminium occurred within the alloy with the formation of alumina particles within a γ' (nominally Ni₃(Al,Ti)) denuded zone but the morphology of the sub-surface oxides differed between the two surface conditions examined. The kinetics of thickening of the chromia layer were sub-parabolic in most cases but closely approached parabolic behaviour for the un-peened surface condition at 800 °C. An enhancement in the rate of chromia growth was found for both surface conditions compared with a Ti-free chromia layer. This enhancement has been attributed to increased Cr ion diffusion as a result of Ti-doping of the chromia layer but the effect is reduced over time because of Ti-depletion in the alloy. At 800 °C, in the un-peened condition, the reduction in growth rate with exposure time is much less marked and this effect seems to be associated with the formation of a (Ti,Ta)O₂ phase beneath the chromia layer.

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1. Introduction

High temperature oxidation resistance is becoming increasingly important as operating temperatures increase, particularly in aero-engines. This is in response to the need to improve fuel efficiency and reduce harmful emissions. It has been established that shot-peening can extend the service life of critical components through inducing compressive stresses in the surface and thereby improving fatigue crack resistance [1,2]. This process is commonly used on rotor discs in service. Little work has previously been performed to investigate the oxidation characteristics of Ni-based superalloys having undergone shot-peening or similar surface modifications. Most studies have been performed on material with either as-machined or polished surfaces but it is important that the oxidation damage is evaluated on the actual surface condition that is used on the component in service. By contrast, the oxidation behaviour of Ni-based superalloys [3–8], and specifically RR1000 [9–13] in the

polished/machined condition, has been studied extensively and has been the subject of a number of recent publications. Typically during high temperature exposure of specimens in laboratory tests, a continuous surface layer of chromia is formed but with isolated particles of TiO₂ on the outer surface. Internal oxidation of aluminium also occurs and results in the formation of sub-surface precipitates of alumina [3,4,9,12]. Ahead of this zone of internal oxidation lies a region depleted in γ' (nominally Ni₃(Al,Ti)) precipitates and, after oxidation in air at temperatures >800 °C, precipitates of TiN [9,10]. A grain boundary carbide (M₂₃C₆) depleted region underneath the surface oxide has also been reported in a similar Ni-based superalloy, ME3, [4] and similar observations have been made in RR1000 [10]. Sigma phase formation in such alloys is also possible [14].

Shot-peening is expected to enhance chromium diffusion in the near surface region of the alloy, through an increase in dislocation density, and result in the quicker formation of a protective Cr-rich surface oxide layer. This has been demonstrated for boiler steels and for the Fe–Ni based alloy 800H [15,16]. However, shot peening has also been shown to increase oxidation rates in alloys where protective oxide scales are normally found. This is the case in previous work investigating the weight gain kinetics of RR1000 where it was shown that shot-peening had an adverse effect on oxidation at 700 and 750 °C [17].

* Corresponding author.

E-mail addresses: S.Cruchley@pgr.bham.ac.uk (S. Cruchley), M.P.Taylor@bham.ac.uk (M.P. Taylor), R.Ding@bham.ac.uk (R. Ding), H.E.Evans@bham.ac.uk (H.E. Evans), Daniel.Child@Rolls-Royce.com (D.J. Child), Mark.Hardy@Rolls-Royce.com (M.C. Hardy).

The present work provides a more detailed comparison of the oxidation response of the two surface conditions through extensive measurements of oxide layer thickness. The work complements an earlier study [11] on the un-peened RR1000 alloy and detailed comparison with these earlier data will be made in the present work. The earlier work conducted on this alloy by the same authors recorded the mass-gain oxidation kinetics [9,11,17], sub-surface oxidation kinetics [10]. As part of the detailed investigation performed the presence of Ti within the chromia was noted [11]. The analysis of the kinetics of the external chromia growth was performed using extensive metallographic measurements. The chromia growth on this alloy was found to be significantly greater than that observed previously on Ti-free chromia. From this observation a mechanism for sub-parabolic growth of this chromia scale was produced and attributed to increased ionic transport caused by the doping of the chromia scale by Ti [18].

In this paper, particular attention will be paid to the influence of shot peening on the kinetics of thickening of the chromia layer. The role of Ti on this process will also be considered.

2. Experimental procedure

An advanced powder metallurgy Ni-based superalloy, RR1000, was used in this study and was provided by Rolls-Royce plc. The nominal composition is given in Table 1. The alloy consists principally of a two-phase microstructure of a γ (nominally Ni, Co) matrix and approximately a 45% volume fraction of γ' precipitates.

The material was prepared with either a polished or shot-peened surface condition. For the former, samples were cut (10 mm \times 5 mm \times 2 mm), ground and the edges chamfered before being polished to a 6 μm finish ($R_a = 0.3 \mu\text{m}$) using conventional preparation methods. For the shot-peened condition, the same batch of alloy was again cut to provide samples (20 mm \times 10 mm \times 2 mm), whose edges were chamfered and the large surfaces ground to a 1200 grit finish. These specimens were then shot-peened using the following conditions: 110H steel shot, 6–8 Almen and 200% coverage.

Oxidation testing was conducted over the temperature range of 700–800 °C in laboratory air for times up to 8000 h in the polished condition and 2000 h in the shot-peened condition. Prior to testing, the specimens were cleaned in ethanol and dried, before being placed in batches into alumina boats and inserted into horizontal tube furnaces at temperature. The furnace was previously calibrated using an N-type thermocouple to ± 1 °C. At selected time intervals, a specimen was removed from the batch for examination before the high temperature exposure continued for the remainder of the batch. Table 2 shows the time intervals chosen for examination.

After oxidation testing, both surface and cross-sectional examination was performed. The surfaces of the specimens were sputter coated with gold and examined by scanning electron microscopy (SEM) using both secondary (SE) and backscattered electrons (BSE). Cross-sectional analysis was performed by nickel-plating the specimens before they were mounted using vacuum impregnation in a low viscosity resin. The specimens were prepared for cross-sectional analysis by grinding on SiC papers down to 1200 grit using water as a lubricant followed by polishing using progressively finer diamond paste from 6 μm down to 0.25 μm . The cross-sections were examined using a high resolution JEOL 7000F FEGSEM. This equipment is capable of wavelength dispersion spectroscopy (WDS), used here for the identification of oxygen, nitrogen and carbon, and energy dispersion spectroscopy (EDS) used for heavier element compositional identification, mapping and line scans. X-ray diffraction (XRD) analysis of the surface oxides was performed on a Philips XPert system using Cu K α radiation, indexed between a 2θ of 10–100°. Metallographic measurements of chromia

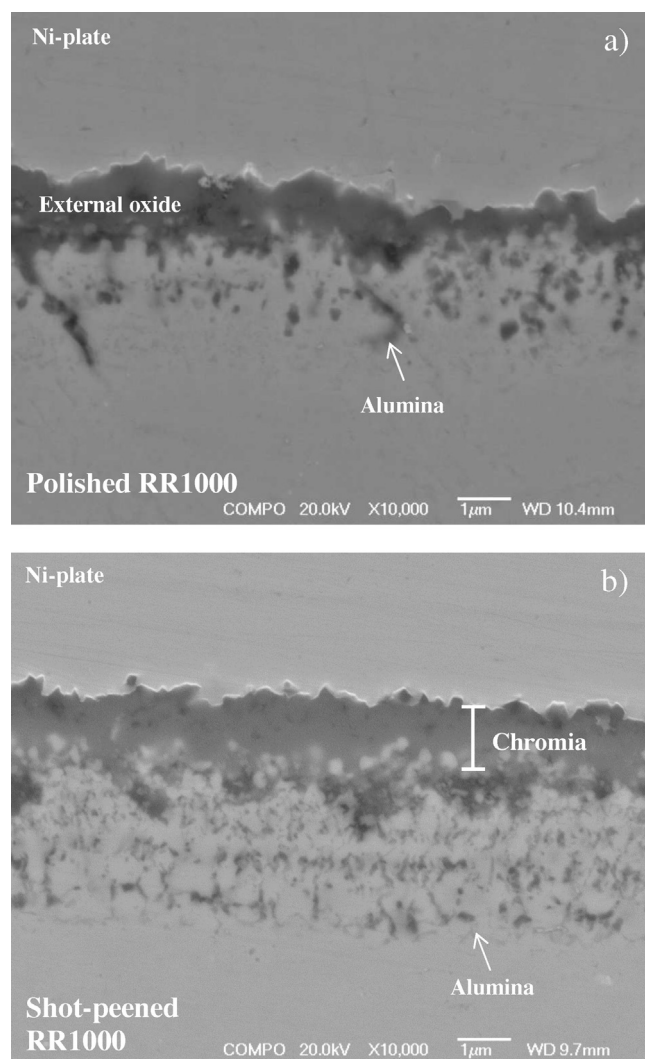


Fig. 1. BSE images of a cross-section through a) a polished RR1000 sample and b) a shot-peened RR1000 sample both oxidized at 700 °C for 2000 h. Both show a continuous external scale of chromia, with sub-surface alumina penetration into the alloy. The figure also illustrates how the chromia measurements were performed.

thicknesses were performed, as described elsewhere [11], using a total of 50 measurements taken from 10 micrographs of representative images of each specimen as shown in Fig. 1. EDS analysis was used to confirm that the measurement conformed to just the chromia part of the external scale.

Thin sections for transmission electron microscopy were produced using focussed ion beam sectioning on a Quanta 3D FEG FIB/SEM dual beam system. EDS compositional analysis and selected area diffraction were then undertaken using a field emission gun Tecnai F20 (S)TEM.

3. Results and discussion

3.1. Oxide characterisation

The typical oxide morphology (both surface and internal) of both polished and shot-peened RR1000 is shown in the cross-sections of Fig. 1. EDS analysis confirmed that the external oxides were chromium and titanium rich and that sub-surface alumina was formed (Fig. 2a). XRD analysis confirmed that the composition of the surface oxide was the same in both shot-peened and polished RR1000, being comprised of chromia and rutile (Table 3). XRD anal-

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