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The role of oxidation damage in fatigue crack initiation of an advanced Ni-based superalloy

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ABSTRACT

The effects of prior oxidation on the room temperature fatigue life of coarse-grained Ni-based superalloy, RR1000, have been investigated. High cycle fatigue tests were conducted, on both machined and pre-oxidised testpieces, at room temperature at an *R* ratio of 0.1. The oxidation damage was produced by pre-exposures at 700 °C for either 100 or 2000 h. Pre-oxidised testpieces tended to fail with shorter fatigue lives than those obtained from the as-machined testpieces although they were also observed to outperform the as-machined test pieces at peak stress levels around 900 MPa. The chromia scale and intergranular alumina intrusions formed during pre-oxidation are prone to crack under fatigue loading leading to early crack nucleation and an associated reduction in fatigue life. This has been confirmed to be the case both below and above a peak stress level of ~900 MPa. The better fatigue performance of the pre-oxidised specimens around this stress level is attributed to plastic yielding of the weaker γ′ denuded zone, which effectively eases the stress concentration introduced by the cracking of the chromia scale and intergranular internal oxides. This γ′ denuded zone is also a product of pre-oxidation and develops as a result of the selective oxidation of Al and Ti. Over a limited stress range, its presence confers a beneficial effect of oxidation on fatigue life.

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1. Introduction

The role of environmental degradation in the mechanical performance of a component is important, particularly for aero-engine rotor discs which operate in high temperature oxidising environments under significant external loads. When these Ni-based alloys oxidise, a protective chromia external scale is formed along with the sub-surface formation of alumina both intergranularly and intragranularly [1]. Beyond these is a weaker region depleted in γ′ particles (nominally $Ni_3(Al,Ti)$) and grain boundary carbides (nominally $M_{23}C_6$). The formation of these sub-surface internal oxides is undesirable and may have a significant detrimental effect on the mechanical properties of the alloy [2,3]. It has been postulated that the internal oxides that form at the grain boundaries have the potential to crack and introduce local stress concentrations which promote early initiation and therefore reduce component lives [2]. Oxides are inherently brittle materials with tensile failure strains of <1% and K_{1c} values for chromia and alumina in the range of 0.4–2.0 MPa $m^{1/2}$ [4]. These are typically much lower than the underlying alloy, so it should be no surprise that it is possible for these oxides to fail under tensile loading.

Several studies on the Ni-based superalloys ME3, and Udimet 720 have found that prior oxidation reduces fatigue life and, in IN100, that the crack initiation life was similar to the number of cycles to cause the oxide to fracture [5–7]. The tests performed on ME3, have shown that prior high temperature exposure in an oxidising environment (>700 °C) for prolonged periods of time (100–2020 h) have a detrimental effect on the high temperature (704 °C) notched fatigue life [5]. The thicker the external scale and the deeper the internal damage the more pronounced the reduction in life [5] which was thought to be due to the dissolution of $M_{23}C_6$ particles. Removal of the internally oxidised region did not lead to a complete recovery in high temperature fatigue life but the removal of the carbide dissolution zone did. Another study [7] using extensive prior exposures of ME3 (704 °C for 439 h) and Udimet 720 (650 °C and 704 °C for 100 or 1029 h) found that the mean lives of pre-oxidised specimens had up to a 70% reduction in high temperature LCF life. A change of crack initiation was also seen from sub-surface (as-received) to surface (pre-oxidised). Performing the prior exposures in vacuum led to no reduction in fatigue life, illustrating that oxidation damage is driving the reduction in life [7].

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The aim of the present work was to extend the previous research and examine the potential for intergranular internal oxides to act as preferential crack initiation sites and therefore affect the overall life of a component. This work concentrates on the fundamental understanding of the initiation between oxidation and fatigue. The use of pre-oxidised samples undergoing fatigue at room temperature was performed to isolate the effect of oxidation on the fatigue life. This allows the exclusion of any high temperature processes by removing the influence of creep deformation or oxidation ahead of the crack tip that would affect the fatigue crack growth. Bend samples have been used to restrict fatigue failures to the top surface region where the oxide resides.

2. Experimental method

2.1. Material

A third generation, powder processed, γ' precipitation strengthened Ni-based superalloy, RR1000, was used in this study. The chemical composition of the alloy is given in Table 1. The material had received a supersolvus solution heat treatment and subsequent ageing treatment which produced a coarse-grained microstructure (30–50 μm) and a bi-modal dispersion of γ' precipitates.

2.2. Preparation of fatigue testpieces and pre-oxidation

High cycle fatigue testing was performed at room temperature under four-point bend loading using a rectangular bar testpiece geometry (100 mm × 9 mm × 10 mm). Testing was performed on either as-machined or pre-oxidised testpieces. In the as-machined condition the corners were chamfered to remove the potential for corner crack initiation and the specimens were subsequently cleaned and degreased in ethanol ultrasonically for a period of 5 min before testing. The surfaces of the specimens were left as-machined with the low stress ground machine marks parallel to the direction of the applied stress. The surfaces of the specimens for pre-oxidation, however, were prepared by grinding and polishing to a R_a = 0.3 μm, with the edges and corners chamfered to the same surface finish to reduce stress concentrations. These were again cleaned and degreased ultrasonically in ethanol for a period of 5 min. An Elite Thermal Systems Ltd. box furnace was pre-heated to 700 °C into which samples were placed for pre-oxidation in laboratory air for time periods of either 100 or 2000 h. Calibration was performed using an N-type thermocouple to ±5 °C. Upon placing the specimens in the furnace a temperature drop of ~50 °C was recorded but the target temperature was regained within 10 min. A pre-oxidised (2000 h) specimen was used to check the effect of the aged microstructure on fatigue life. This was performed by removing all of the oxidation damage (external and internal) by grinding using 240 grit paper. Grinding marks were parallel to the direction of the applied stress.

2.3. High cycle fatigue (HCF) testing

Testing was performed at room temperature under a four-point bending configuration with a maximum applied stress of between 700 and 1000 MPa. An Amsler Vibrophore HCF machine with a load capacity of 20 kN was employed and a frequency of ~75 Hz was achieved on the current testpiece geometry. A span ratio of 20:60 mm and a R ratio of 0.1 were utilised throughout. Testpieces that had not failed after 5×10^7 cycles were deemed to be a runout and the specimen was removed from the test machine for further analysis.

2.4. Fractographic and metallographic analysis

The fracture surfaces of failed specimens were examined in a Phillips XL-30 scanning electron microscope (SEM) in order to understand the influence of pre-oxidation on the fatigue process. The top surface of the specimen, which was subjected to tensile stress during testing, was also examined for any cracking of the oxide scale. Care was taken in the preparation of metallographic sections due to the brittle nature of the oxides. Specimens were first mounted whole under vacuum in a low shrinkage, low viscosity epoxy resin. The region of highest stress was then cut out using a precision cutting machine at a low cutting speed (<0.05 mm/min) before being re-mounted under vacuum in the same epoxy resin. Note that the section plane was parallel to the longitudinal direction and perpendicular to the top surface which had been subjected to tensile stress. Samples were then polished to a final stage of 0.25 μm diamond solution or OP-S colloidal silica solution before being cleaned ultrasonically in ethanol and imaged using a Jeol 7000F field emission gun (FEG) SEM. Chemical etching was performed by swabbing the surface of the sample for 30 s using Kalling's reagent or through a 60 s immersion in a selective γ' etchant (compositions in Table 2).

2.5. Nano-indentation and Microhardness

Nano-indentation was performed on a cross-section of a specimen oxidised for 500 h at 800 °C, using a Micromaterials Ltd. Platform 3 NanoTest machine. The indentations were performed at 1 μm intervals in a diagonal pattern across the specimen, starting within the Ni-plate and progressing through the oxide into the bulk alloy. The indents were performed using a Berkovich indenter under a load controlled setting to a maximum load of 30 mN. A loading rate of 1 mN s^{-1} was used. Once the indenter reached the maximum load this was then held for a short period of time (~30 s) before being removed from the specimen, giving a load vs. displacement graph. The hardness of the material was then calculated from the tangent to the unloading curve.

Vickers microhardness testing was also performed on a cross-section of an as-machined specimen and an aged specimen (oxide removed), using a Struers Durascan 50 automated microhardness machine. It was performed using a 0.3 kg load, with an average value being taken from 12 indents.

3. Results and Discussion

3.1. Surface oxidation damage caused by pre-oxidation

Previous studies on the Ni-based superalloys Udimet 720, ME3 and RR1000 under cyclic fatigue or dwell fatigue conditions found no change in oxide composition compared with the unstressed condition but did record an enhancement of the oxidation damage

Table 1
Nominal composition of RR1000 in both atomic and weight%.

	Ni	Co	Cr	Mo	Ti	Al	Ta	Hf	Zr	C	B
Weight%	Bal	18.5	15.0	5.0	3.6	3.0	2.0	0.5	0.06	0.02	0.03
Atomic%	Bal	17.9	16.5	3.0	4.3	6.35	0.63	0.16	0.04	0.14	0.10

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