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Effects of Cyclic Stress and Temperature on Oxidation Damage of a Nickel-Based Superalloy

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Abstract

Oxidation damage, combined with fatigue, is a concern for nickel-based superalloys utilised as disc rotors in high pressure compressor and turbine of aero-engines. A study has been carried out for a nickel-based alloy RR1000, which includes cyclic experiments at selected temperatures (700°C~800°C) and microscopy examination using Focused Ion Beam (FIB). The results suggest that the major mechanism of oxidation damage consists of the formation of surface oxide scales and internal micro-voids and oxide particles beneath the oxide scales, which become more severe with the increase of temperature. Applying a cyclic stress does not change the nature of oxidation damage but tends to enhance the extent of oxidation damage for temperatures at 750°C and 800°C. The influence of cyclic stress on oxidation damage appears to be insignificant at 700°C, indicating a combined effect of cyclic stress and temperature. Further energy dispersive x-ray (EDX) analyses show the enrichment of Cr and Ti, together with lower Ni and Co levels, in the surface oxide scales, suggesting the formation of brittle Cr_2O_3 , TiO_2 , NiO and Co_3O_4 oxides on the specimen surface. Penetration of oxygen into the material and associated internal oxidation, which leads to further

material embrittlement and associated failure, are evidenced from both secondary ion imaging and EDX analyses.

Keywords: Oxidation; Cyclic stress; Oxide scale; Micro-void; Nickel-based superalloy.

1. Introduction

Nickel-based superalloys are favoured for disc rotors in high pressure compressor and turbine of aero-engines due to their exceptional mechanical properties at high temperature. In addition to fatigue and creep, oxidation damage is a concern for disc rotors exposed to engine gas environment for prolonged periods. Evidence of oxidation in nickel alloys has been presented for both smooth and crack specimens with appreciable oxide layers on free surfaces and at internal grain boundaries, as a result of oxygen attack [1-8]. Oxidation results in reduced fatigue life by early cracking of surface oxide scale followed by dominantly intergranular cracking with an accelerated growth rate [4, 5, 9].

A number of mechanisms have been proposed for oxidation induced damage, including formation of both external and internal brittle oxides (Ni, Nb, Al, Cr) [4, 5, 9, 10], vacancy injection [11], gas bubble formation (CO or CO₂) [1] and releasing of embrittling alloy elements [12] at the grain boundaries. Recently, it has been acknowledged [13-15] that oxidation-assisted crack propagation in nickel alloys is a result of oxygen diffusion into a local area with increasing tensile stresses, such as crack tips. The oxygen element attacks the grain boundaries by lowering boundary cohesion and prompts accelerated intergranular cracking [13-15]. The diffusion process is facilitated by high tensile stresses near the crack tip [13-15, 16], especially for superimposed dwell periods at maximum load where the crack tip is fully open and experiences sustained high tensile stresses.

Alloy RR1000 studied here is one of the new generation polycrystalline nickel alloys, developed at Rolls-Royce plc, to meet the demands for higher overall pressure ratios, compressor discharge temperatures and rotational speeds for the latest aero-engines. The material was produced through a powder metallurgy route and has a fine grain microstructure, with an average grain size of $4\sim 5\mu\text{m}$ [17]. Fatigue, creep and constitutive behaviour of RR1000 at high temperatures has been extensively studied for validation of the material [18-23]. Very recently, oxidation damage of alloy RR1000 has been investigated by Encinas-Oropesa et al. [24] at temperatures between 700°C and 800°C . Oxidation kinetics of RR1000 has been characterized by mass change data from thermogravimetric analyses of small disc samples at temperatures from 700°C to 800°C , where the oxide scale rich in Cr and Ti has been found to grow in a parabolic dependence with time. On the other hand, FIB examinations of oxidised samples for RR1000 revealed the appearance of the oxide scale on the surface and the formation of recrystallised grains and voids beneath the oxide scales [24]. The oxidation-linked microstructure damage becomes more significant with the increase of testing temperature. The thickness of oxide scale and the depth of internal damage, exemplified as voids along grain boundaries, are more than tripled at 800°C when compared to those at 700°C . In addition, oxide particles can also be observed on the surface of those internal micro-voids from the FIB images [24].

However, the work of Encinas-Oropesa et al. [24] was carried out without considering the influence of mechanical loading such as fatigue, which can have a significant effect on oxidation and corrosion damage for nickel and its alloys. For instance, Moulin et al. [25] studied the influence of external mechanical loading on oxygen diffusion during nickel oxidation. It was shown that a relative O enrichment is observed in the oxide scale under mechanical loading and the ingress of oxygen in the substrate beneath the oxide scale becomes easier with a constant tensile load (creep). In the work of

Berger et al. [26, 27], the application of a constant load induced multiple cracks at the surface of nickel oxide scale as well as an increase of oxygen diffusivity by two orders of magnitude, although the enhancement of oxygen diffusivity decreases with the further increase of load level. The oxidation kinetics curves gained by thermogravimetric analysis (TGA) indicate that the oxidation rate of pure nickel was accelerated by both tensile and compressive external stress [28]. A study of cast nickel superalloy IN 100 showed that matrix oxidation was strongly enhanced by fatigue cycling [29]. Effects of applied tensile stress on the growth kinetics of oxide scales were also studied for Ni-20Cr alloys [2], where the internal oxidation and the oxide layers are shown to be much thicker than that formed without load. This was also the case for internal nitridation of nickel alloy Inconel 718 under mechanical stress [30]. Stress-enhanced oxidation was also found for other metallic materials such as Cr-Mo steel, where both oxide layer and penetration of oxide along preferential paths (e.g., cracks, grain boundaries) increased significantly under strain-controlled low cycle fatigue [31, 32].

The objective of this work is to investigate the effects of cyclic stress and temperature on oxidation damage of alloy RR1000. Waisted specimens were tested under cyclic loads in laboratory air at three temperatures (700°C, 750°C and 800°C). FIB microscopic analyses were performed to examine the effects of cyclic stress level on oxidation damage at three selected surface locations. Measurements were made for the thickness of surface oxide scale, the depth of internal damage caused by the oxidation process as well as the combination of the two. EDX analyses were also carried out to identify the chemical composition of the oxides and the element concentration at varying depth from the specimen surface.

2. Experimental details

2.1. Material and specimen

The alloy under investigation is fine grain RR1000, produced through a powder metallurgy route by Rolls-Royce for disc rotor applications in the latest aero-engines. The chemical composition of RR1000 is 18.5Co-15Cr-5Mo-3.6Ti-3Al-2Ta-0.5Hf-0.03C-0.02B-0.06Zr and balance Ni in weight percentage. The alloy has a two-phase microstructure consisting of γ matrix and strengthening γ' precipitates $\text{Ni}_3(\text{Al, Ti, Ta})$ which are largely responsible for the elevated temperature strength of the alloy. An average grain size of $4\sim 5\mu\text{m}$ was determined from an EBSD (Electron Backscatter Diffraction) analysis [17].

Waisted specimens, as shown in Fig.1, were provided by Rolls-Royce for fatigue testing in air at high temperatures. The specimen has a diameter of 4.4mm and 18mm for the narrowest section and the section at the ends, respectively, and a length of 94mm. All specimens have been carefully cleaned by longitudinal and cross-hatch polishing before the fatigue tests. A surface roughness analysis was also performed on the specimens using a Mitutoyo Surfacetracer SV-C524 and the arithmetic mean value, R_a , was found to be $0.79\mu\text{m}$.

Waisted specimen has a gradual variation of the cross sectional area from the middle position to the ends, i.e., the diameter changes from 4.4mm for the narrowest middle section to 18mm for the section at the ends (Fig.1). Under fatigue loading, this will produce variable stress levels across the specimen length (Fig.2). This geometry allows investigation of the effect of variable cyclic stress on oxidation damage by carrying out the fatigue test on a single specimen for a given temperature.

2.2. Fatigue tests

The fatigue tests were carried out on a computer-controlled Instron 8500 servo-hydraulic machine with a load cell of $\pm 25\text{kN}$. The machine has a SF868E furnace mounted and the furnace can be

heated up to 1,100°C. The temperature was monitored by three K-type thermocouples placed on the side of the specimen. The temperature has a variation of $\pm 2^\circ\text{C}$ between the thermocouple calibrator and the digital controller, and a variation of less than $\pm 5^\circ\text{C}$ along the specimen. The tests were conducted in laboratory air condition with water vapour content less than 5ppm.

Fatigue tests were carried out at 700°C, 750°C and 800°C under a triangular loading waveform. For 700°C and 750°C, a load of 10kN was applied, with a load ratio of $R = 0.1$ and a loading frequency $f = 0.25\text{Hz}$. For 800°C, the load was reduced to 8kN, considering the reduced mechanical properties at such a high temperature, while the load ratio and loading frequency remain unchanged ($R = 0.1$ and $f = 0.25\text{Hz}$).

2.3. FIB analyses

FIB system was used to cut trenches on the specimen surface to reveal and examine the nature of the oxidised surface after the fatigue tests. The equipment is an FEI Strata 200xP FIB with a fine gallium (Ga^+) beam. The specimen was mounted onto a special grip and then placed inside the FIB chamber where vacuum conditions ($1.6 \times 10^{-5}\text{mbar}$ for platinum strip deposition, $5.9 \times 10^{-6}\text{mbar}$ for coarse milling and $3.3 \times 10^{-6}\text{mbar}$ for fine milling) apply. An area of interest, with a dimension of $25\mu\text{m} \times 2\mu\text{m}$, was selected and a layer of platinum (Pt) deposition was applied on the selected surface to avoid both contamination and stray sputter damage during the FIB milling operations. The cutting operation using the Ga^+ beam was initially in coarse steps (at a current of 6600 pA), followed by finer steps (at a current of 1000 pA) to produce one flat side to the trench [24]. FIB images were produced via detected back scattered electrons that resulted from bombardment of the sample using low power Ga^+ ion beam, which scans the flat side of the trench.

In the present work, two different modes were used for the FIB imaging, i.e., secondary ions and secondary electrons. FIB secondary ion images are particularly sensitive to the presence of oxides and carbides in metallic systems due to the effect of these elements on the secondary ion yield of the metal, which is ideal to reveal oxides and carbides as the oxygen or carbon “enhanced yield” increases the brightness of the area. FIB secondary electron imaging is very effective in revealing the grain orientation and contrast without etching [33].

FIB analyses were carried out for all three specimens to identify the oxide scale and the associated damage inside the material under fatigue-oxidation conditions. Three locations on each specimen, as illustrated by "A", "B", "C" in Fig.1, were chosen to examine the influence of cyclic stress level on oxidation damage. Point A is located at the narrowest section, which has the highest stress level, while points B and C are 15mm and 30mm, respectively, away from the point A and have reduced stress levels due to the increased cross-sectional areas.

2.4. EDX analyses of oxides

EDX analyses of both surface and internal oxides were carried out in a Field Emission Gun Scanning Electron Microscope (FEG-SEM). Locations at selected distances from the oxide scale surface were examined to identify the chemical composition of the oxides at different depths into the specimen. Concentrations of the detected elements were measured in weight percentage and compared with each other to reveal the nature of oxidation ingress into the substrate beneath the surface oxide scale.

3. Results

3.1. Stress analyses

Finite element analyses were first carried out to compute the stress concentration factor for the specimen. A quarter of the specimen was considered due to the geometrical symmetry and meshed into 8-node axisymmetric elements with full integration [34]. The right and bottom edges are constrained in the x and y directions, respectively, to remove the rigid body motion. A distributed load of 39MPa, equivalent to 10kN for concentrated load, was applied to the top surface of the specimen. Contour plot of the normal stress in the y direction is shown in Fig.2a, where the highest stress concentration is located in the narrowest section. The stress concentration factor, defined as the ratio of the normal stress in the y -direction (σ_{yy}) to the applied stress ($\sigma_{app}=39\text{MPa}$), was calculated for the specimen and shown in Fig.2b against the vertical distance from the narrowest section. The middle section has a stress concentration factor as high as 17. The stress concentration is gradually reduced towards the ends of the specimen, which allows the study of the influence of cyclic stress level on the oxidation damage. The stress concentration factors are 17.1, 8.9 and 2.5 for point A, B and C, respectively. Specifically, under the load of 10kN, the stress level is 670MPa, 350MPa and 100MPa, i.e., 65%, 34% and 10% of 0.2% proof stress of the material at 650°C, at point A, B and C, respectively.

3.2. Fatigue experiments

At 700°C, the specimen was loaded for 176,763 cycles, approximately 196 hours, and survived the test duration without failure. While fatigue failure occurred in specimens tested at 750°C and 800°C, with complete fracture at the narrowest section. The fatigue life is 174,392 cycles (194h) at 750°C and 33,473 cycles (38h) at 800°C, indicating a dramatic reduction of fatigue resistance at temperatures up to 800°C.

Microscopic examination of the fracture surface is shown in Fig.3a for the specimen tested at 750°C, where fatigue crack seems to initiate from the surface and propagate through the cross section till the final fracture. The side surface was also examined and shown in Fig.3b for the specimen tested at 800°C (Fig.3b), which shows numerous surface cracks near the fracture surface. Observed surface cracks for 800°C specimen represents the fracture of the surface oxide layer, which was essentially caused by localised severe deformation near the waisted section as a result of the reduced mechanical properties at 800°C. Surface cracks, i.e., cracking of oxide layers, were not observed for specimens tested at 700°C and 750°C due to the less extent of oxidation damage and mechanical deformation near the waisted section.

3.3. FIB examinations

FIB analyses were performed to examine the oxidation damage for the tested specimens and the secondary electron images taken for position "A" are shown in Figs.4a, 5a and 6a for the three temperatures, all of which reveal the surface oxide scales and internal micro-voids beneath the oxide scale. At 700°C, the oxidation is limited to the surface area of the specimen. The oxide nodules on the surface do not appear to be uniform, with patches of oxidised and un-oxidised areas. The micro-voids are almost invisible. The oxide scale above the surface has a thickness of up to 0.8µm and the depth of internal damage (below the surface and into the material) is measured up to 1.0µm. In this paper, the thickness of oxide scale was measured as the distance between the identified specimen surface and the surface of the oxide scale; while the depth of internal damage was measured as the distance between the identified specimen surface and the very tips of internal damage. The measurements were based on the FIB projections at the selected positions, which will vary if a different FIB position is selected.

At 750°C and 800°C, as shown in Fig.5a and Fig.6a, the specimens showed more severe oxidation damage when compared to that at 700°C, with a thicker oxide scale and a deeper damage into the specimen. At 750°C, the thickness of oxide scale is measured up to 1.6µm from Fig.5a, almost twice as much as that for the 700°C specimen. The depth of internal damage measured from the obtained FIB image is up to 2.5µm, which is more than doubled when compared to that for the 700°C specimen. At 800°C, the thickness of oxide scale and the depth of internal damage have values up to 1.7µm and 7.0µm, respectively (Fig.6a). There is an increased propensity for voids at higher temperatures of 750°C and 800°C, as shown in Figs.5 and 6. The voids are considered to be the result of the loss of base alloy elements (e.g., Cr, Ti Ni), which diffuse outwards to the specimen surface to form the oxide scale. Also voids are mainly located along the grain boundaries which are the preferential diffusion paths.

In addition, internal oxide particles can be clearly seen from the secondary ion images in Figs.4b, 5b and 6b, where the bright areas below the specimen surface indicate the oxides within the material. A comparison with the electron images (Figs.4a, 5a and 6a) suggests that internal oxides tend to follow the traces of the micro-voids formed beneath the oxide scale. Therefore, in addition to the outwards diffusion of alloy elements (e.g., Cr, Ti, Ni) to form surface oxides, oxygen also transports into the material through the porous oxide scale and internal voids along the grain boundaries, and chemically reacts with the alloy elements to form internal oxide particles, especially, on the surface of the voids.

3.4. Effect of stress level on oxidation damage

To examine the effects of cyclic stress level on oxidation damage, FIB images, taken at the three locations A, B and C, were compared. The same types of oxidation damage were observed at all three locations, i.e., the oxide scales built on the surface and the internal micro-voids along the grain

boundaries. This can be seen from the FIB images of oxidation damage presented in Fig.7 for position B and C at 800°C. Furthermore, the thickness of surface oxide scale and the depth of internal damage were measured for the three locations and presented in Figs.8a, b and c for 700°C, 750°C and 800°C, respectively. It can be seen that, at 750°C and 800°C, the cyclic stress has a notable influence on the severity of the oxidation damage. Both the oxide scale and the depth of internal damage increased with the increase of the stress level. At 800°C, the scale of overall damage at the highest stress area (Point A) nearly doubled that at the lowest stress area (Point C), with a measured difference of 3.8µm. At 750°C, the scale of total oxidation damage at Point A is 0.5µm more than that at Point C. However, at 700°C, the effect of cyclic stress on oxidation damage seems to be less clear, as shown in Fig.8a. This might be due to the relatively low level of oxidation kinetics at this temperature [24].

3.5. Chemical composition of oxides

As shown in Figs.4b, 5b and 6b, secondary ion imaging confirms the formation of oxides both on the specimen surface and in the specimen. For further information, EDX analyses of the trench at position "A" were performed for specimens tested at 750°C and 800°C to analyse the chemical composition of those oxides at five different depths from the surface. At 750°C, the five spectrums selected for EDX analyses spread over a depth of 2.2µm from the oxide scale surface, with equal spacing (0.54µm) between each other. Specifically, the first spectrum is located at the oxide scale surface while the fifth spectrum has a depth of 2.2µm into the material. The obtained element profiles at the five spectrums are shown in Figs.9 and 10 for 750°C and 800°C, respectively. As seen from Figs.9a and 10a, the surface oxide scale mainly consists of Ni, Cr, Ti and Co oxides, indicating the formation of Cr₂O₃, TiO₂, NiO and Co₃O₄ oxides on the specimen surface, which is consistent with the work of Encinas-Oropesa et al. [24]. The formation of the Cr₂O₃ layer indicates a stage of

saturation during the oxidation process of the alloy, which may act as a protection from further oxidation. Cr and Ti levels decreased progressively with the increase of depth, accompanied by the progressive increase of Ni and Co levels, a result of approaching the base material. The oxides also contain a small percentage of Mo, Al and Ta, as shown in Figs.9b and 10b. The level of O decreases with the increase of depth at 750°C (Fig.9b), while it seems to remain steady at 800°C (Fig.10b) probably due to the presence of multiple surface cracks which facilitated the penetration of oxygen into the specimen. This suggests the ingress of oxygen into the material and formation of internal oxide particles, as also evidenced from the secondary ion images (Figs.4b, 5b and 6b). At 800°C, a reduced percentage of O element was obtained when compared to that at 750°C (see Figs.9b and 10b), which may be due to the lower density of oxides at 800°C caused by the increased propensity and depth of micro-voids (Figs.6 and 7), as well as the multiple surface cracks (Fig.3b) generated under fatigue loading. The patchy presence of oxide scales for the specimen tested at 700°C made the EDX analyses rather difficult and inaccurate. Also, the internal damage for the 700°C specimen is too limited to obtain a meaningful concentration-depth profile and therefore EDX analyses were not carried out for 700°C specimen.

4. Discussions

Oxidation damage of alloy RR1000 has been investigated by Encinas-Oropesa et al. [24] for small disc sample at temperatures between 700°C and 800°C, in the absence of mechanical loading. Oxidation kinetics has been characterized by mass change data from thermogravimetric analyses, where the mass gain has been found to grow in a parabolic function with time. In addition, FIB examinations of oxidised samples revealed the appearance of the oxide scale, rich in Cr and Ti, on the surface and the formation of recrystallised grains and micro-voids beneath the oxide scales [24]. Internal oxide particles can also be observed on the surface of those micro-voids from the FIB

images. The thickness of oxide scale and the depth of internal damage at 800°C are more than tripled when compared to those at 700°C. Our results suggest the same types of oxidation damage for alloy RR1000 in the presence of fatigue loading. Fatigue tends to increase the thickness of oxide scale and depth of internal damage for temperatures at 750°C and above. For instance, at 750°C, the added measurements of oxide scale and internal damage are 4.1µm, 3.3µm and 3.2µm for Position A, B and C, respectively, as compared to 2.0µm reported in Encinas-Oropesa et al. [24]. At 800°C, the oxidation damage becomes much more severe under fatigue loading. The added measurements of oxide scale and internal damage are 8.7µm, 5.7µm and 4.8µm for Position A, B and C, respectively, as compared to 3.9µm reported in Encinas-Oropesa et al. [24]. The considerably increased oxidation damage for position A at 800°C may be due to the numerous surface cracks formed near the fractured section (Fig.3b) which act as short circuits for oxygen penetration. While, at 700°C, there seems to be no fatigue influence on the oxidation damage, at least for the load level considered in the present work. The measured oxidation damage for the three positions A, B and C is in the range of 1.7~1.9µm, which is comparable to the measurement of 1.5µm in Encinas-Oropesa et al. [24]. It should be pointed out that the specimens used in Encinas-Oropesa et al. [24] has a surface finish of 0.25µm, which is different from the 0.79µm surface finish of the waisted specimen tested in the present work. This might have an influence on the measured oxidation damage.

EDX analyses in Encinas-Oropesa et al. [24] were reported for the surface oxides, which show the enrichment of Cr and Ti as well as low Ni and Co level. This is consistent with the results in the present work. Similar oxides were also found for other types of nickel superalloys. For instance, Chen et al. [35] studied the oxidation behaviour for nickel alloys Wasploy, Astroloy and Udimet 720 after isothermal exposures at temperature between 750°C and 1000°C. It was reported that x-ray diffraction detected Cr₂O₃, TiO₂ and NiCr₂O₄ oxides on the surface of the materials [35]. In addition,

NiO was also found in Astroloy after a 20 hour exposure at 750°C [35]. Surface oxides for nickel alloy 718 was reported to consist of Ni or Nb rich Cr_2O_3 [36, 37]. It was well recognized that the formation of Cr_2O_3 is largely responsible for the superior oxidation resistance of nickel-based superalloys.

During oxidation, outwards diffusion of alloy elements (Cr, Ni and Co) leads to the growth of surface oxides, as well as resulting in the formation of internal voids due to the loss of alloy elements. The voids seem to locate along the grain boundaries, indicating the primary path of alloy elements diffusion. Interestingly, internal oxidation was indentified from the secondary ion imaging (Figs.4b, 5b and 6b), suggesting the simultaneous inwards diffusion of oxygen to form internal oxide particles. Penetration of oxygen was also confirmed by the presence of O element from the EDX analyses at the selected depths into the material (Figs.9 and 10). Internal oxide particles are considered to be a mixture of TiO_2 and Al_2O_3 [24, 35, 36]. Internal oxidation, especially along grain boundaries, may lead to critical embrittlement of the material and results in significantly shortened fatigue life via intergranular fracture process. As shown in Zhao [38], simulation of internal oxidation can be done from oxygen diffusion analyses based on Fick's first and second laws. The depth of oxygen penetration and concentration-depth profile can be predicted from the oxygen diffusion analyses and further utilized to quantify the material embrittlement caused by internal oxidation process.

Investigation of the effect of mechanical stress on oxidation has significant implications for the oxidation-accelerated crack growth and failure behaviour. For polycrystalline nickel superalloys, environmental effects were frequently observed to modify the fracture morphology from transgranular to intergranular during crack growth, as well as to increase the crack growth rates significantly for a given stress intensity factor [4, 5, 9, 39]. Recently, it has been acknowledged and

evidenced that strong environmental effect on crack growth of nickel based superalloy is due to the penetration of oxygen into a crack tip. Under the influence of a tensile stress, oxygen may move into the grain boundaries by diffusion, attacking the grain boundaries by themselves or through chemical reactions with alloy elements. The oxygen diffusion process at a crack tip is a dynamic process, a combined effect of time, temperature, local deformation and microstructure. Oxygen diffusion may be facilitated by high tensile stresses near the crack tip [13-15], especially for superimposed dwell periods at the maximum load where the crack tip is fully open and experiences sustained high tensile stresses. The work of Zhao et al. [40] showed that crack growth rates in air at high-temperature can be well predicted from a dynamic embrittlement hypothesis.

For modelling of stress-assisted oxygen diffusion near the crack tip, a coupling between the oxygen diffusion and the mechanical deformation is generally required to include the effect of crack tip deformation (stress fields) on oxygen diffusion process [15, 41]. In this case, diffusion of oxygen within the material is controlled by two essential parameters, namely, the oxygen diffusivity and the pressure factor [15, 40, 41]. So far, no direct measurements are available for oxygen diffusion parameters in nickel alloys. In fact, the present work measured the scale of oxidation damage as well as the concentration of O element under varying cyclic stress levels from the fatigue-oxidation tests on waisted specimens, which are useful in the determination of the two essential parameters in oxygen diffusion analyses. Some pilot work has been carried out in Zhao [38], where modelling of oxygen diffusion along grain boundaries was attempted under creep loading condition based on the coupling of crystal plasticity with oxygen diffusion. Further work is currently undergoing to simulate the oxygen diffusion and predict oxygen penetration for waisted specimen under fatigue in order to calibrate the oxygen diffusion parameters and feed the crack growth analyses under fatigue-oxidation conditions.

5. Conclusions

Formation of surface oxide scales (Cr, Ti, Ni and Co oxides) and micro-voids beneath the oxide scales seem to be the major mechanisms for oxidation damage of alloy RR1000 under fatigue loading conditions. Both surface oxides and internal voids could lead to crack initiation and subsequent propagation into the substrate under mechanical loading conditions, resulting in material failure as occurred for the cyclic tests at 750°C and 800°C.

Cyclic stress influenced oxidation damage at 750°C and 800°C, and the thickness of oxide scale and the depth of internal damage were enhanced by the increase of stress level. Both EDX analyses and secondary ion imaging show the penetration of oxygen into the material to form internal oxide particles, which leads to further embrittlement of the material. Measurements of oxidation damage and oxygen concentration have been carried out, and could be utilised to estimate the oxygen diffusivity and the pressure factor which are two essential parameters for modelling of oxidation damage and prediction of crack growth under fatigue-oxidation conditions.

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References

- [1] R.H. Bricknell, D.A. Woodford, *Acta Metall.* 30 (1982) 257-264.
- [2] G. Calvarin-Amiri, A.M. Huntz, R. Molins, *Mater. High Temp.* 18 (2001) 91-99.
- [3] V. Garat, O. Brucelle, J.M. Cloue, V. Rebeyrolle, D. Monceau, B. Viguier, E. Andrieu, *Mater. Sci. Forum* 461-464 (2004) 537-544.
- [4] E. Andrieu, R. Molins, *Mater. Sci. Eng. A* 154 (1992) 21-28.
- [5] E. Andrieu, G. Hoshstetter, R. Molins, R. Pineau, in: T. Magnin (Ed.), *Corrosion-Deformation Interactions* (EFC 21), 1997, pp.461-475.
- [6] C.F. Miller, G.W. Simmons, R.P. Wei, *Scripta Mater.* 44 (2001) 2405-2410.
- [7] C.F. Miller, G.W. Simmons, R.P. Wei, *Scripta Mater.* 48 (2003) 103-108.
- [8] J. Tong, S. Dalby, J. Byrne, *J. Mater. Sci.* 40 (2005) 1237-1243.
- [9] R. Molins, G. Hochestetter, J.C. Chassaigne, E. Andrieu, *Acta Mater.* 45 (1997) 663-674.
- [10] L. Fournier, D. Delafosse, T. Magnin, *Mater. Sci. Eng. A* 316 (2001) 166-173.
- [11] P. Hancock, R. Fletcher, *Metallurgie* 6 (1966) 1-9.
- [12] D.A. Woodford, R.H. Bricknell, *Treatise Mater. Sci. Tech.* 25 (1983) 157-199.
- [13] J.A. Pfaendtner, C.J. McMahon Jr., *Acta Mater.* 49 (2001) 3369-3377.
- [14] U. Krupp, W.M. Kane, C. Laird, C.J. McMahon Jr., *Mater. Sci. Eng. A* 387-389 (2004) 409-413.
- [15] U. Krupp, *Int. Mater. Rev.* 50 (2005) 83-97.
- [16] D. Bika, C.J. McMahon Jr., *Acta Metall. Mater.* 43 (1995) 1909-1916.
- [17] C. Stöcker, M. Zimmermann, H.-J. Christ, Z.L. Zhan, C. Cornet, L.G. Zhao, M.C. Hardy, J. Tong, *Mater. Sci. Eng. A* 518 (2009) 27-34.
- [18] J. Tong, S. Dalby, J. Byrne, M. Henderson, M.C. Hardy, *Int. J. Fatigue* 23 (2001) 897-902 .
- [19] L.G. Zhao, J. Tong, B. Vermeulen, J. Byrne, *Mech. Mater.* 33 (2001) 593-600.
- [20] S. Dalby, J. Tong, *J. Mater. Sci.* 40 (2005) 1217-1228.

- [21] Z.L. Zhan, J. Tong, *Mech. Mater.* 39 (2007) 64-72.
- [22] Z.L. Zhan, J. Tong, *Mech. Mater.* 39 (2007) 73-80.
- [23] L.G. Zhao, J. Tong, *J. Mech. Phys. Solids* 56 (2008) 3363-3378.
- [24] A. Encina-Oropesa, G.L. Drew, M.C. Hardy, A.J. Leggett, J.R. Nicholls, N.J. Simms, Effects of oxidation and hot corrosion in a nickel disc alloy, the 11th International Symposium on Superalloys, Champion, Pennsylvania, USA, 14~18 September 2008.
- [25] G. Moulin, P. Arevalo, A. Salleo, *Oxid. Met.* 45 (1996) 153-181.
- [26] P. Berger, G. Moulin, M. Viennot, *Nucl. Instrum. Meth. B* 130 (1997) 717-721.
- [27] P. Berger, L. Gaillet, R. El Tahhann, G. Moulin, M. Viennot, *Nucl. Instrum. Meth. B* 181 (2001) 382-388.
- [28] C.H. Zhou, H.T. Ma, L. Wang, *Corros. Sci.* 52 (2010) 210-215.
- [29] M. Reger, L. Remy, *Metall. Trans. A* 19 (1988) 2259-2268.
- [30] U. Krupp, *Shreir's Corrosion* 1 (2010) 304-315.
- [31] Z. Azari, M. Abbadi, H. Moustabchir, M. Lebienvén, *Int. J. Fatigue* 30 (2008) 517-527.
- [32] B. Fournier, M. Sauzay, C. Caës, M. Noblecourt, M. Mottot, A. Bougault, V. Rabeau, A. Pineau, *Int. J. Fatigue* 30 (2008) 649-662.
- [33] Fibics. Using FIB Secondary Ion Images to Observe the Presence of Corrosion. Retrieved from: <http://www.fibics.com/fib/application/using-fib-secondary-ion-images-to-observe-the-presence-of-corrosion-/22/>, 2009.
- [34] ABAQUS, Version 6.8, Dassault Systemes Simulia Corp., Providence, USA, 2009.
- [35] J.H. Chen, P.M. Rogers, J.A. Little, *Oxid. Met.* 47 (1997) 381-410.
- [36] V.B. Trindade, U. Krupp, Ph.E.-G. Wagenhuber, Y.M. Virkar, H.-J. Christ, *Mater. High Temp.* 22 (2005) 207-212.
- [37] L. Geng, Y.S. Na, N.K. Park, *Mater. Design* 28 (2007) 978-981.

- [38] L.G. Zhao, ASME J. Eng. Mater. Tech. 2011, in press.
- [39] H. Ghonem, D. Zheng, Mater. Sci. Eng. A 150 (1992) 151-160.
- [40] L.G. Zhao, J. Tong, M.C. Hardy, Eng. Fract. Mech. 77 (2010) 925-938.
- [41] F.L. Carranza, R.B. Haber, J. Mech. Phys. Solids 47 (1999) 27-58.

Figure captions

Figure 1, Waisted specimen geometry and the three positions “A”, “B” and “C” selected for FIB examination.

Figure 2(a), Contour plot of the normal stress in the y direction for the waisted specimen under an applied load of 10kN, where d is the distance from the narrowest section of the specimen.

Figure 2(b), Stress concentration factor, defined as the ratio of the normal stress in the y-direction (σ_{yy}) to the applied stress ($\sigma_{app}=39.0\text{MPa}$), against the distance from the narrowest section of the specimen.

Figure 3(a), The fracture surface of the specimen fatigue tested at 750°C.

Figure 3(b), Multiple surface cracks on the side surface of the specimen fatigue tested at 800°C.

Figure 4, FIB images for position "A" at 700°C (a) secondary electron and (b) secondary ion, where the dashed line in red indicates the original specimen surface.

Figure 5, FIB images for position "A" at 750°C (a) secondary electron and (b) secondary ion, where the dashed line in red indicates the original specimen surface.

Figure 6, FIB images for position "A" at 800°C (a) secondary electron and (b) secondary ion, where the dashed line in red indicates the original specimen surface.

Figure 7, Comparison of oxidation damage at position (a) "B" and (b) "C" for 800°C, where the dashed line in red indicates the original specimen surface.

Figure 8(a), Comparison of thickness of oxide scale and depth of internal damage for positions A, B and C at 700°C.

Figure 8(b), Comparison of thickness of oxide scale and depth of internal damage for positions A, B and C at 750°C.

Figure 8(c), Comparison of thickness of oxide scale and depth of internal damage for positions A, B and C at 800°C.

Figure 9(a), EDX element profiles showing weight% of Cr, Ni, Ti and Co against the distance from the oxide scale surface (750°C).

Figure 9(b), EDX element profiles showing weight% of O, Mo, Al and Ta against the distance from the oxide scale surface (750°C).

Figure 10(a), EDX element profiles showing weight% of Cr, Ni, Ti and Co against the distance from the oxide scale surface (800°C).

Figure 10(b), EDX element profiles showing weight% of O, Mo, Al and Ta against the distance from the oxide scale surface (800°C).

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