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Influence of shot peening on high-temperature corrosion and corrosion-fatigue of nickel based superalloy 720Li

G. J. Gibson[1*], K. M. Perkins[2], S. Gray[3] and A. J. Leggett[1]

High-temperature corrosion fatigue, a combination of corrosion with a fatigue cycle, is an emerging generic issue affecting power generation and aero gas turbine engines and has the potential to limit component life. Historically, surface treatments, such as shot peening have been used to improve component life and have been optimised for fatigue response. Research into optimisation of shot peening techniques for hot corrosion and high-temperature corrosion fatigue has shown 6–8A 230H 200% coverage to provide overall optimum performance for nickel-based superalloy 720Li based on the limited data within this study. Utilisation of electron backscatter diffraction techniques, in combination with detailed assessment of corrosion products have been undertaken as part of this work. The resultant cold-work visualisation technique provides a novel method of determining the variation in material properties due to the shot peening process and the interaction with hot corrosion. Through this work it has been shown that all three shot peening outputs must be considered to minimise the effect of corrosion fatigue, the cold work, residual stress and surface roughness. Further opportunity for optimisation has also been identified based on this work.

Keywords: Nickel, Corrosion fatigue, Hot corrosion, Sulphidation

Introduction

The drive towards more efficient methods of power generation and the associated reductions in greenhouse gas emissions require existing gas turbine engines to operate under ever-increasing service temperatures. To meet these demands, future designs may utilise novel or improved alloy systems with an enhanced temperature capability and employ a range of thermal barrier coatings.[1] Irrespective of such advances, the safe and efficient operation of new and existing plant will, to an ever-increasing degree, rely on a fundamental understanding of rotor and blade materials. In particular, the mechanical response during prolonged exposure to hot corrosive conditions is an emerging generic issue facing the power generation industry.[2]

Interactions between the environment and component substrate can lead to corrosion pitting. Fundamental studies have identified the detailed mechanistic drivers of such damage. Under high-temperature exposure, various authors have identified the propensity for Type II sulphidation in a number of different nickel-based systems. Nicholls et al. have investigated the controlling parameters behind such damage, in particular, the roles played by surface condition, local *in situ* gaseous chemistry and surface residues.[3,4]

In-service conditions for these components dictate that they will also experience cyclic and/or static stress conditions. Under the combined influence of both environment and stress, a modified Type II sulphidation mechanism has been identified.[5] The mechanism is associated with the formation of sulphides in the alloy ahead of the corrosion front, at the alloy–oxide interface, commonly observed in Type I hot corrosion. The oxide formed also contains a sulphide band, similar to that observed in Type II hot corrosion. This mechanism results in the localised attack in the substrate ahead of any physical damage, increasing susceptibility to crack initiation. This is in contrast to what is traditionally seen from Type II stress-free corrosion, where damage is typically shallow and broad in nature.[6,7] The formation of wide shallow pits from a fatigue perspective will not increase the local surface stress significantly.

In order to minimise the effect of surface damage on fatigue life, many components routinely use shot peening to provide life benefit.[8–10] The shot peening parameters are traditionally assessed via a series of fatigue experiments performed in laboratory air, which is unrepresentative of service conditions. Previous testing on pre-pitted specimens under Type II corrosion, then subsequently tested in air, had minimal effect on life provided the features remained within the peened layer. However, little work has examined the combined effect of surface finish and/or peening, in combination with a corrosion fatigue mechanism. This shortfall is a direct result of, until recently, having no capability for examining fatigue under such harsh environmental conditions. The aim of the current work is to therefore assess current peening parameters with respect to corrosion and corrosion fatigue by performing a systematic

[1]Rolls-Royce PLC, P.O. Box 31, Derby DE24 8BJ, UK
[2]School of Engineering, Materials Research Centre, Rolls-Royce University Technology Centre, Swansea University, Swansea SA2 8PP, UK
[3]Surface Engineering & Nanotechnology Institute, Building 57, Cranfield University, Bedfordshire MK43 0AL, UK

*Corresponding author, email grant.gibson@rolls-royce.com

Table 1 Chemical composition of 720Li[11]

	Cr	Co	Mo	W	Ti	Al	C	Zr	Ni
Amount wt %	16	14.8	3	1.25	5	2.5	0.015	0.035	Bal

study. The work attempts to assess the relationship between peening parameters and their effects on surface finish, corrosion and finally that of corrosion fatigue in order to obtain optimum protection from corrosion in a cyclic/static stress environment.

Experimental procedures

All tests were undertaken on a nickel-based superalloy commonly used for aerospace high pressure turbine disc applications, 720Li, composition given in Table 1.

Role of shot peening on surface condition

Two identical sets of round 720Li specimens, 5 mm in diameter and 10-mm long, were shot peened, Fig. 1. One set was used to perform electron backscatter diffraction (EBSD) depth and surface roughness measurements and the other to investigate the role of shot peening on pure corrosion resistance of the nickel alloy.

Table 2 shows the matrix of peening conditions applied to the small round specimens.
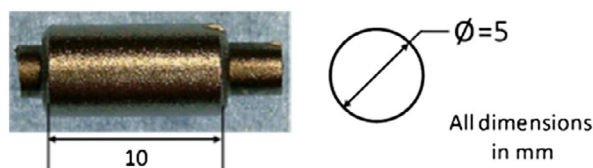
The peening condition of 6–8A, 110H, 200% coverage was provided by Rolls-Royce to be used as a reference. The nomenclature utilised is consistent with AMS 2431[12] where intensity is denoted in imperial Almen units, shot size is given in inches where 110H is equivalent to 0.011″ and H denotes hard shot type. Coverage is given as a percentage which relates to the surface coverage of the sample, i.e. 100% coverage equates to one complete surface coverage of shot peening indents. Two off specimens were produced for each condition and additional two control specimens were also peened using a standard peening condition of 6–8A, 110H, 200% coverage as a reference. Two-hundred per cent coverage is stipulated in order to ensure areas on parts achieve at least 100% coverage in all areas. Complex geometries often

1 Shot peening cotton reel sample schematic

Table 2 Matrix of shot peening conditions used

Notation	Intensity/A	Shot size/H	Coverage/%
GA0	–	–	–
GA1-3	1–3	110	200
GA3-5	3–5	110	200
GA6-8	6–8	110	200
GA8-10	8–10	110	200
GB70	6–8	70	200
GB110	6–8	110	200
GB170	6–8	170	200
GB230	6–8	230	200
GB330	6–8	330	200

hinder access to peening media and the coverage level of 200% also accounts for process variability.

Batch 1 was initially used for surface roughness measurements. Subsequently, EBSD analysis was performed to determine the level of strain hardening imparted to the specimens during each peen condition. EBSD will provide grain misorientation at the surface of each specimen relative to remainder of the section. The level of this misorientation is directly proportional to the level of strain hardening[13] which has been shown to be the main contributing factor to fatigue resistance rather than the resulting residual stress. Batch 2 was utilised in the static corrosion trials described in Section 2.3.

Classical assessment of compressive residual stress utilises X-ray diffraction to measure the stress as a function of distance from the material surface.[14–16] Despite numerous measurements of residual stress to assess the effects of compressive residual stress, a recent study by Guechichi and Castex[17] provides evidence that strain hardening and not residual stress is the primary contributor to fatigue resistance. Such claims are supported if residual stress relaxation is considered, as shown by Evans et al.[18], who demonstrated that one cycle of high-temperature isothermal fatigue is enough to reduce residual stress levels in a Ni-based superalloy by more than 50%. More recent work has highlighted the ability to utilise EBSD tools to produce a map of the grain to grain misorientation near the surface. The EBSD data is used to produce a grain orientation spread (GOS) map, a measurement which is directly proportional to the level of strain hardening.[13]

The technique uses post-processing software to define a region of permanently strain-hardened material. Unlike peak compressive depth measurements, which decay during thermal or service exposure, the layer defined using the EBSD tool can be assessed pre- or post-service. The method defines a threshold limit of grain boundary angle relative to the neighbouring grains. Software is then used to calculate those grains which have a high misorientation, currently set at one standard deviation above the average, Fig. 2.

Static corrosion trials

To assess the hot corrosion resistance of the shot-peened samples, batch 2 was sprayed with approximately 0.16 mg of a 98% Na_2SO_4 – 2% NaCl salt mixture and placed in a corrosion furnace containing air + 300 vppm SO_2 at 700 °C for 300 h with salt re-coats applied every 50 h to ensure full coverage of the specimens. The flux level used in the testing is calculated based on the salt dosage over the area over the 50 h of the exposure. Samples were salted via a hot sample spray process whereby the specimens are placed on a hot plate, heated to around 200 °C at which point the salt mixture, suspended in water, is sprayed on the specimen surface. The heated surface causes flash evaporation leaving localised salt deposits on the surface.

Static corrosion trials also included a datum, un-peened sample to enable a direct comparison with the matrix of peened conditions. Specimens were then assessed using the 4% exceedance method, a procedure first proposed by Nicholls and Hancock.[4] The 4% exceedance method provides a maximum, median and minimum pit depth generated after
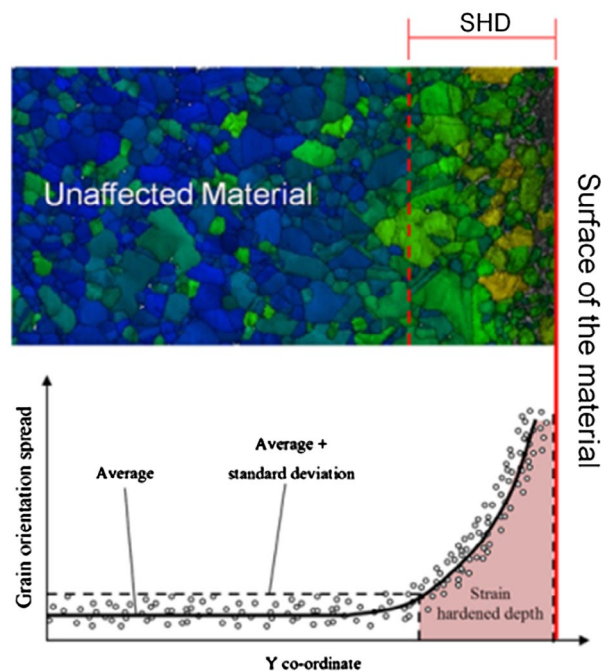
2 Top image shows a GOS map produced by utilising the technique shown in the bottom image. Grains highlighted in green have high misorientation relative to the grains coloured in blue. The image is read from right to left with the surface of the material indicated[13]

a fixed time interval. The maximum depth provides a 96% level of confidence that all pits will be less than this value. Alternatively, only 4% of pits will exceed this value.

Mechanical assessment

Optimisation of a new peening routine needs to satisfy several criteria, namely those of static corrosion, mechanical performance and the combined effect of corrosion and mechanical conditions. Optimal peening parameters under static corrosion may not necessarily offer the required resistance to fatigue cracking. Subsequent to static corrosion testing, specific peened conditions were down selected for mechanical assessment, Table 3.

Cylindrical plain LCF specimens with a nominal diameter of 4.5 mm and gauge length of 12 mm were used for all high-temperature fatigue and corrosion fatigue trials. The specimen is shown schematically in Fig. 3.

All specimens were tested at 700 °C for both air and corrosion fatigue conditions under load control utilising a fully reversed, $R = -1$, sawtooth waveform of 1.5–1.5–1.5–1.5 s at a single stress level. These specific test conditions were chosen to minimise creep effects, whilst maximising the time at temperature exposure of the corrosion fatigue specimens.

Table 3 Down-selected matrix of shot peening parameters

Peening condition	Intensity/A	Short size/H	Coverage/%
1–3A 110H 200%	1–3	110	200
8–10A 110H 200%	8–10	110	200
6–8A 70H 200%	6–8	70	200
6–8A 230H 200%	6–8	230	200
6–8A 330H 200%	6–8	330	200
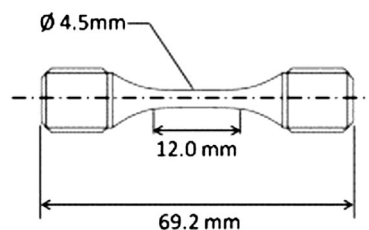
3 Round bar low cycle fatigue test specimen schematic

Complete specimen rupture was chosen as test termination criteria.

Specimens for the corrosion fatigue testing were pre-sprayed prior to testing with a 98% Na_2SO_4 – 2% NaCl salt mixture and tested in an environmental chamber containing an air + 300 vpm SO_2 atmosphere. Complete specimen rupture was again chosen as the failure criteria.

Results

Role of shot peening on surface condition

Fatigue requirements are that a material's surface be as smooth as possible to ensure surface-dominated features do not develop into fatigue cracks. Moreover, a rough surface will locally be able to trap more corrosive species. Thus, it should be expected that an optimum condition may exist where the surface roughness is acceptably low and the strain-hardened depth (SHD) is sufficient to retard micro-cracking. Table 4 summarises the results of roughness and SHD layer for a given shot peening condition.

Plotting the interaction of surface roughness with shot peening parameter allows two general trends to be observed. The relationship between surface roughness and intensity is linear and the relationship between roughness and shot size is in general, relatively flat, (Fig. 4).

The relationship between intensity and roughness follows a logical argument in that for the equivalent coverage condition, the impacts will be of deeper depths due to the increased kinetic energy of the shot stream. The relationship shown for shot size is less well understood as an increasing shot size for hard materials such as nickel should improve as the shot size increases. This is because the area of contact between the shot and the surface will increase, leading to a reduced crater depth. One explanation for the results would be that a critical coverage level exists above which surface roughness values become homogenous. Therefore, the argument of reduced roughness with increasing shot size may be more applicable to low coverage levels where the number of impacts for a given coverage level will depend on the shot size.

Utilisation of EBSD allows for the measurement of SHD layer for the different shot peening parameters. In order to maximise fatigue performance, it is desirable to have a deep SHD layer with the lowest roughness. By correlating the two data-sets, it is possible to form a correlation between roughness and SHD and therefore by inference intensity and shot size (Fig. 5).

Moving from top left to bottom right indicates the progressively desirable condition, a deep strain-hardened region and a low surface roughness. The circled condition in Fig. 5 suggests the best shot peening condition to be 6–8A, 330H, 200%. It should be noted, however, that this assumes the role of surface roughness is as important as the depth of strain-hardened material. In the case of corrosion fatigue, where features will develop irrespective of the

Table 4 Summary of surface roughness and SHD layer for the different shot peening parameters

Notation	Intensity/A	Shot type/H	Coverage/%	Sa (µm)	Strain-hardened layer depth (µm)
GA0	–	–	–	0.32	–
GA1-3	1–3	110	200	0.66	42
GA3-5	3–5	110	200	0.96	50
GA6-8	6–8	110	200	1.83	91
GA8-10	8–10	110	200	2.13	103
GB70	6–8	70	200	1.89	63
GB110	6–8	110	200	1.96	77
GB170	6–8	170	200	1.25	62
GB230	6–8	230	200	1.41	75
GB330	6–8	330	200	1.38	83
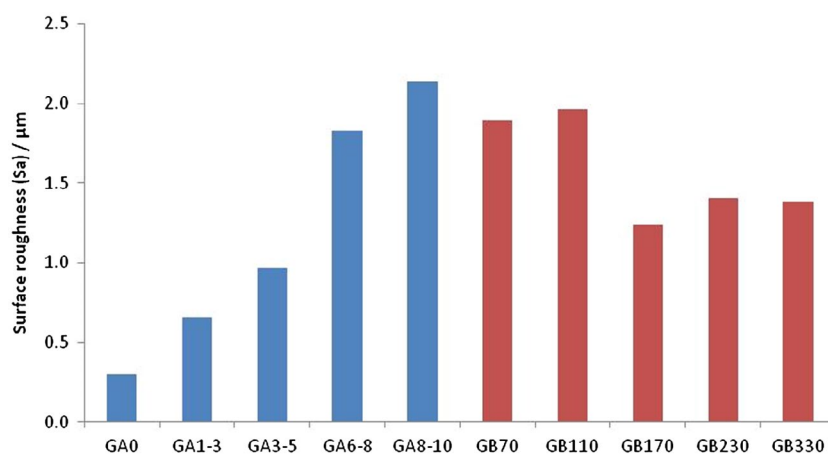
4 Surface roughness in µm against shot peening condition
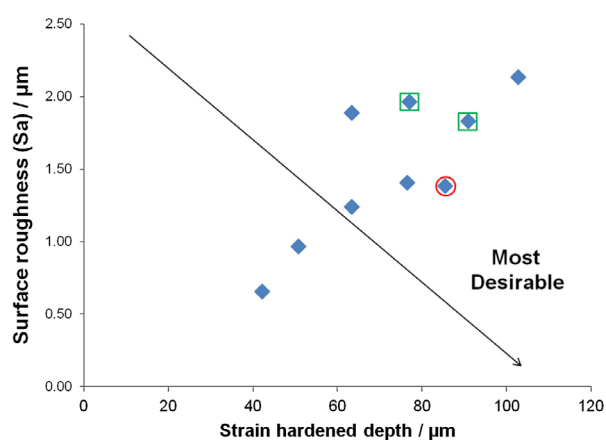
5 Surface roughness against SHD layer – boxes represent the datum reference condition and the ringed data point is 6–8A, 330H, 200% coverage

surface condition, it may be more beneficial to increase the depth of strain-hardened layer to ensure micro-cracks are retarded for longer time.
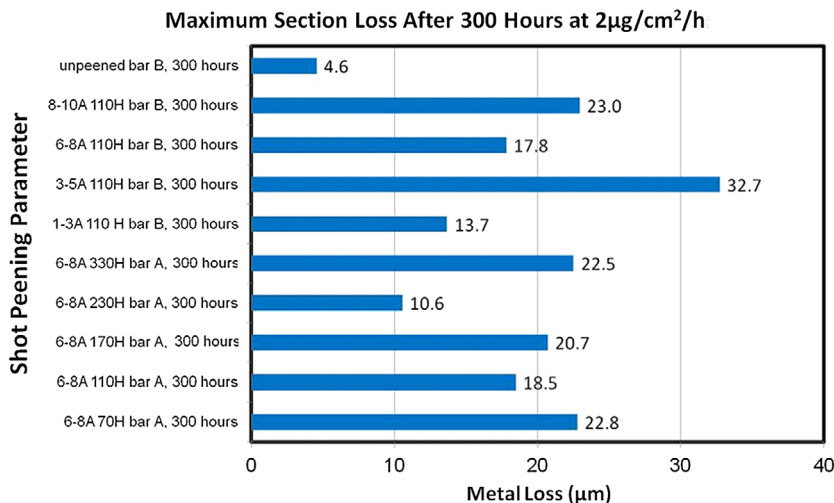
Static corrosion trials

Maximum and median metrology results are presented for the matrix of shot peening conditions listed in Table 2. The results show that the un-peened specimen gave the best hot corrosion performance as compared to the shot-peened samples. The datum shot peening condition performed poorly as did the initially identified most desirable shot peening parameter,

6–8A, 330H, 200% coverage. Based on the results of the hot corrosion testing, 6–8A, 230H, 200% coverage provides the best balance between surface roughness, SHD layer and hot corrosion performance (Figs. 6 and 7).
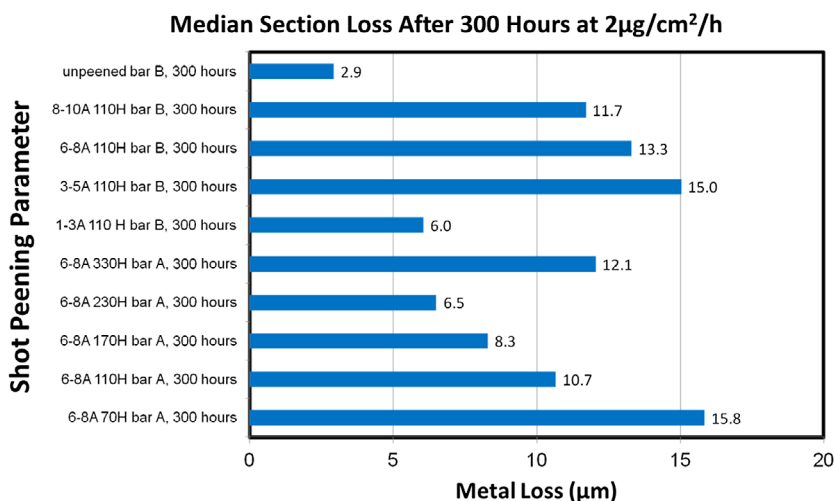
Mechanical assessment

Air fatigue testing was undertaken on the down-selected shot peening conditions listed in Table 3 based on the hot corrosion test results. Due to the size and scope of this work, the decision was made to investigate both intensity and shot size. This meant that the number of tests carried out at each condition had to be limited. Results are presented in Figure 8 plotted against the standard shot peening condition baseline data for 720Li. Testing was performed at a single stress to attempt to maximise the validity of conclusions with a limited number of tests. The results have been normalised with respect to the highest test stress used in the programme, to protect the absolute data.

The results highlight the important balance between surface roughness and SHD layer. The high-temperature air fatigue result of 1–3A, 110H, 200% coverage is poor due to a low SHD layer in combination with a relatively poor surface roughness. These in combination lead to early onset of fatigue failure. High intensity shot peening provides the deepest SHD layer, which would be predicted to give a life benefit. However, the high energy associated with the shot stream during peening leads to an increased risk of detrimental shot peening indents leading to a life debit. The other conditions investigated all sit within scatter of the baseline datum air fatigue result.

Maximum Section Loss After 300 Hours at 2µg/cm²/h

Shot Peening Parameter	Metal Loss (µm)
unpeened bar B, 300 hours	4.6
8-10A 110H bar B, 300 hours	23.0
6-8A 110H bar B, 300 hours	17.8
3-5A 110H bar B, 300 hours	32.7
1-3A 110 H bar B, 300 hours	13.7
6-8A 330H bar A, 300 hours	22.5
6-8A 230H bar A, 300 hours	10.6
6-8A 170H bar A, 300 hours	20.7
6-8A 110H bar A, 300 hours	18.5
6-8A 70H bar A, 300 hours	22.8

6   Ninety-six percentile corrosion metal loss data for the different shot peening parameters after 300 h of exposure to salt and SO$_2$

Median Section Loss After 300 Hours at 2µg/cm²/h

Shot Peening Parameter	Metal Loss (µm)
unpeened bar B, 300 hours	2.9
8-10A 110H bar B, 300 hours	11.7
6-8A 110H bar B, 300 hours	13.3
3-5A 110H bar B, 300 hours	15.0
1-3A 110 H bar B, 300 hours	6.0
6-8A 330H bar A, 300 hours	12.1
6-8A 230H bar A, 300 hours	6.5
6-8A 170H bar A, 300 hours	8.3
6-8A 110H bar A, 300 hours	10.7
6-8A 70H bar A, 300 hours	15.8

7   Median (50 percentile) corrosion metal loss data for the different shot peening parameters after 300 h of exposure to salt and SO$_2$
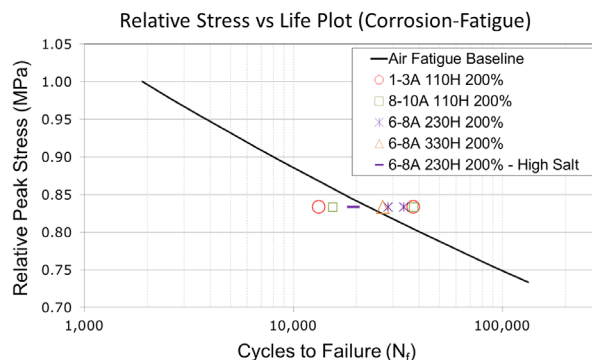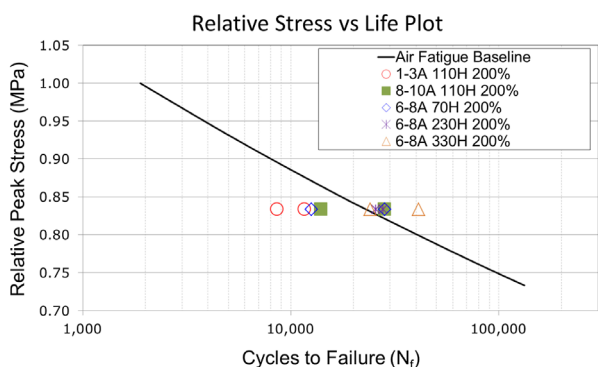
8   Normalised air fatigue data for the down selected matrix of shot peening conditions

9   Relative stress vs. life plot for specimens testing in corrosion fatigue

A second set of plain LCF 720Li specimens, peened at conditions described in Table 3 was sprayed with approximately 0.15 mg/cm² of a 98% Na$_2$SO$_4$ – 2% NaCl salt mixture and placed in a high temperature corrosion fatigue rig. The specimens were tested to failure in an air + 300 vppm SO$_2$

atmosphere again at a single stress to maximise the usefulness of limited testing (Fig. 9).

The results show a similar trend to the air fatigue data. Early failure occurred in 60% of tests due to the poor hot corrosion performance observed in static no-stress hot

corrosion trials. The response for the reference shot peening condition under corrosion fatigue is not shown on the plot but has found to give around a 50% life debit. The best shot peening condition under corrosion fatigue testing is 6–8A, 230H, 200% coverage. The combination of good hot corrosion resistance and no worse air fatigue performance combines to produce an optimised corrosion fatigue shot peening parameter for 720Li. Even under aggressive high salt conditions, the life debit is no worse than would be expected for a normal salt load for the datum shot-peened parameter.

EBSD and SEM analysis

Type II hot corrosion testing suggests shot peening of nickel superalloys is detrimental to their hot corrosion performance. Due to the low salt levels used in testing, the form of corrosion is not a classical Type II morphology. At low fluxes, the mechanism is dominated by accelerated oxidation rather than broad front pitting attack. The combination of low grain misorientation and good surface roughness in the un-peened sample reduces inward diffusion of sulphur and enhances adhesion of the protective chromium oxide scale forming on the surface. In the shot-peened specimens, the roughness can cause oxide cracking, due to bending stresses generated on the surface of the sample from the shot peening dimples. Once the oxide scale is ruptured, this allows corrosive species to attack the substrate below. This will enhance the rate of sulphur diffusion down the grain boundaries due to high level of grain misorientation in the near surface region. Figure 10 shows the typical corrosion depth and EDX map of specimen GA0. For comparison, Fig. 11 shows the typical corrosion depth and EDX map of specimen GA8-10.

Detailed SEM analysis highlights the differences in oxide thickness between the datum un-peened sample and the highest intensity shot peening parameter, 8–10A. Sample GA8-10 also shows a greater density of sulphur in the substrate suggesting that the high angle grain boundaries may reduce the required amount of energy for diffusion.
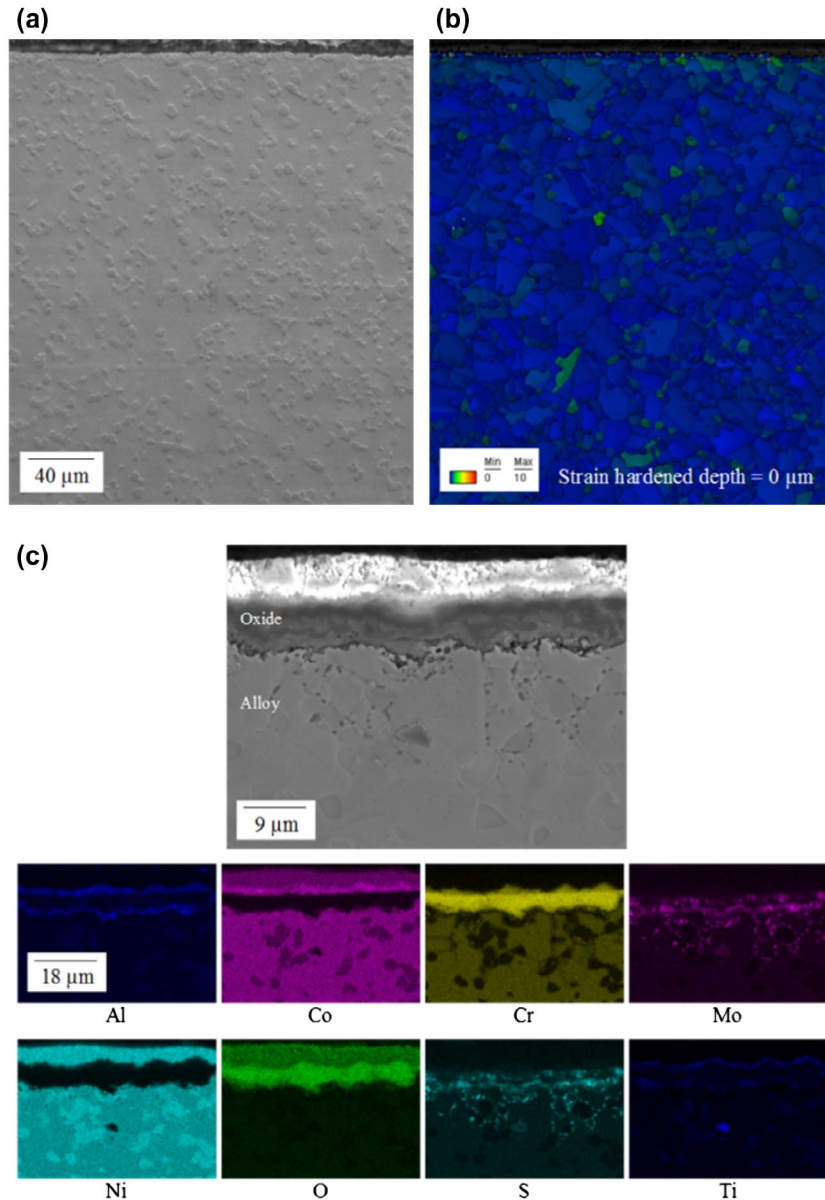
10   *a* Secondary electron image of specimen GA0; *b* EBSD-generated SHD layer for specimen GA0 and *c* secondary electron and associated EDX maps for the typical corrosion depth in specimen GA0
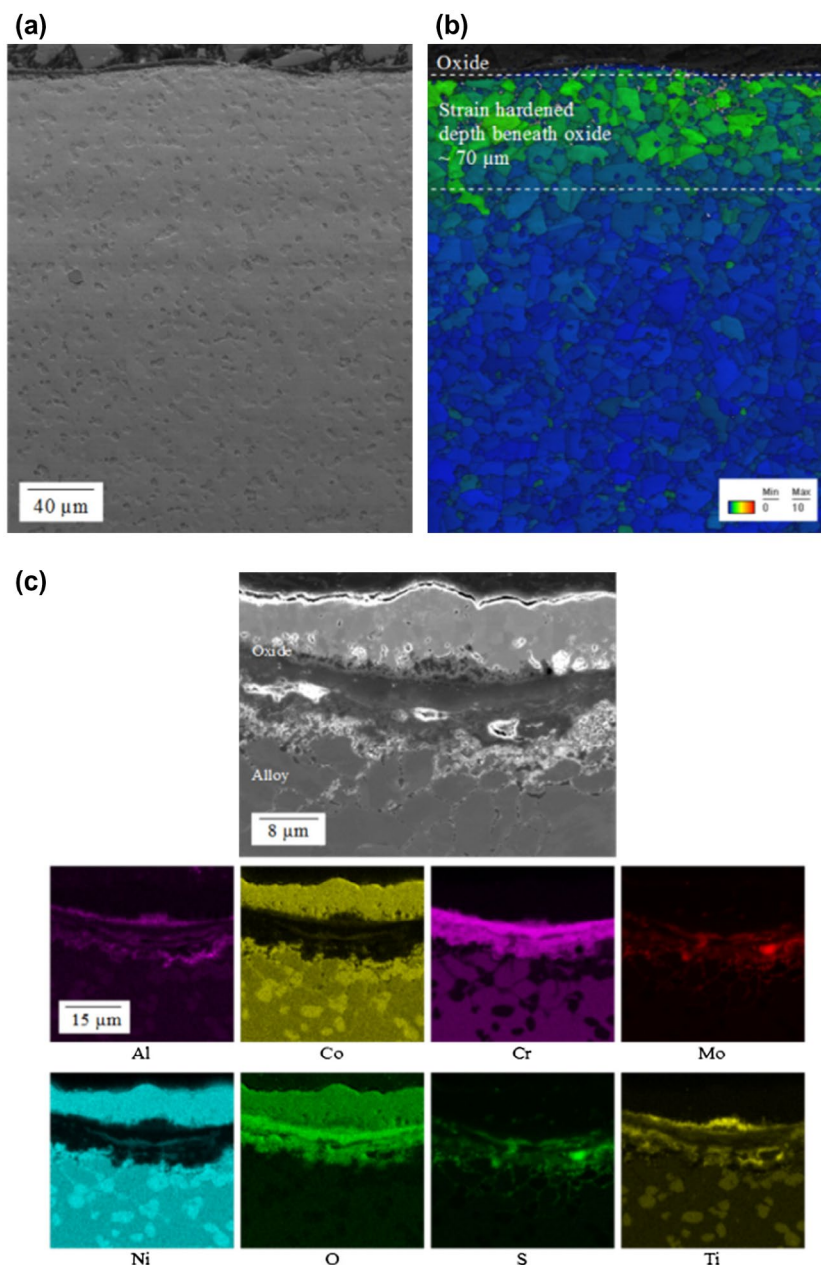
11 *a* Secondary electron image of specimen GA8-10; *b* EBSD-generated SHD layer for specimen GA8-10; and *c* secondary electron and associated EDX maps for the typical corrosion depth in specimen GA8-10

Discussion

The testing undertaken and described in this paper represents the culmination of a significant package of work. For the first time, air fatigue, hot corrosion and high-temperature corrosion fatigue have all been assessed with regards to the interaction with shot peening on 720Li. From the testing undertaken within this research, it is clear that the datum shot peening condition of 6–8A 110H 200% coverage is not optimised fully for any of the damage mechanisms assessed.

Stress-free hot corrosion trials showed that for low flux Type II hot corrosion, where the mechanism of attack is diffusion dominated, shot peening a surface increases its susceptibility to corrosion. It appears there is some contribution of surface roughness as well as cold work depth to the results. It is proposed that surface roughness contributes to a pooling effect whereby larger valleys are able to trap more salt and this acts to locally increase the concentration. Cold work is

present from the process and manifests itself in the form of highly misorientated grains near the surface. The high level of misorientation creates a dislocated network of high angle boundaries which appear to have a reduced resistance to diffusion. Indeed, for machined samples which contain little or no cold work and have relatively good roughness, the hot corrosion performance, under low fluxes, outperforms all the shot-peened surfaces.

Testing in air fatigue, albeit with limited data, shows some difference in the lives achieved for the same peak stress value. Given that testing has been undertaken at 700 °C, it is likely that residual stress relaxation has taken place. It is therefore the cold work, which has been shown to remain post-test and the surface roughness, which is inherent in the surface to control the specimen life. Samples peened with low intensity generally produced low lives. This is backed up by the fact that there will be a reduced cold work depth and the roughness imparted by peening increases the probability of

surface-initiated cracks, which then cannot be retarded by the beneficial cold work layer. For the higher intensity peening conditions of 8–10A, the level of cold work is much deeper than 1–3A; however, this comes at a cost of the surface roughness and risk of a peening indent. Peening indents can occur during the shot peening process as a result of an oblique or unusually high velocity impact. They occur at all intensities; however, higher intensity processes provide more energy to the shot stream and therefore the likelihood of a relatively deep crater forming is higher than for low-intensity processes.

In the case of corrosion fatigue testing, the relaxation of residual stresses at temperature suggests that compressive residual stress does not control specimen life. The formation of micro-cracks within the strain-hardened layer is proposed as the controlling mechanism. The high grain to grain misorientation in the strain-hardened layer induces greater micro-crack deviation, thus the system requires more energy to continue to drive the micro-cracks as compared to an un-peened material. Fatigue is far more dominant than the corrosion for nickel superalloys exposed to representative corrosion fatigue environments. Moreover, the mechanism and rate of corrosion are much lower in flux than classical broad front Type II pitting. Whilst hot corrosion performance is detrimentally affected by shot peening, the life benefit in fatigue due to strain hardening provides a significant relative life improvement as compared to un-peened material exposed to high-temperature corrosion fatigue.

Whilst the number of tests carried out in this programme is acknowledged as being limited using the hot corrosion performance in combination with the hot corrosion results, it is possible to predict the behaviour under corrosion fatigue. Fundamentally, what has been shown in this work is that increasing shot size is beneficial for minimising the effect of corrosion fatigue. The results suggest that 6–8A 230H 200% coverage is the best overall combination, followed by 330H at the same coverage and intensity. The formation of a deep strain-hardened layer in combination with low surface roughness is deemed to be giving rise to this response. Based on the work within this programme, three fundamental output parameters from shot peening have been identified: SHD layer, surface roughness and depth of compressive residual stress. The latter is controlled through intensity and to some degree shot size, the surface roughness is dictated by the shot size, media type, incident angle of the shot stream on the part, intensity and coverage. The strain-hardened layer is controlled through shot size and intensity.

The conclusions obtained from this work have enabled to further optimisation to continue with the aim of minimising the impact of corrosion fatigue on life.

Due to the low surface roughness and relatively deep layer of strain-hardened material, three controlling parameters for further shot peening optimisation have been identified: SHD layer, surface roughness and depth of compressive residual stress.

Conclusions

- High-temperature corrosion and the associated mechanism of high-temperature corrosion fatigue are likely to affect component lives if temperatures and stresses continue to increase in the development of the gas turbine engine.
- Suitable optimisation of the cold work process may provide valuable life benefit.

- Further work is required to fully understand the nature of the mechanism of high-temperature corrosion fatigue and the controlling parameters affecting specimen lives.
- Compressive residual stress is not a controlling parameter for hot corrosion and high-temperature corrosion fatigue due to the relaxation of stresses at temperature.
- Higher intensity shot peening parameters lead to greater sulphide diffusion thought to be a result of increased cold work and dislocations in the microstructure.
- Shot peening enhances the rate of diffusion-based hot corrosion mechanisms when the material is not subjected to mechanical stress.
- Shot peening does however provide an effective barrier for high-temperature corrosion fatigue in nickel-based superalloys.
- A suitable balance of surface roughness, SHD layer and cold work is required to provide optimum system performance.
- 6–8A 230H 200% coverage has been shown to provide optimum hot corrosion and high temperature corrosion fatigue performance within this study, although any increase in shot size is also deemed to give benefit.

Notes on contributors

Karen Perkins is an associate professor at Swansea University working in the Materials Research Centre on all aspects of fatigue in both nickel and steel alloys.

Simon Gray works at Cranfield University in the Surface Science Department on high-temperature corrosion, corrosion fatigue and coatings for the aerospace and power generation industry.

Andrew Leggett is a sulphidation and corrosion specialist and technical lead on sulphidation within Rolls-Royce. He works on a variety of coating programmes particularly targeted at REACH as well as high-temperature turbine applications.

Grant Gibson is a materials technologist currently working on high-temperature nickel-based alloys suitable for direct laser deposition. He has a background in nickel-based super alloys and helping to support the team investigating DLD.

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