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Initiation sites for cracks developed from pits in a shot-peened 12Cr martensitic stainless steel

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A R T I C L E I N F O

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A serial sectioning procedure was carried out on a shot-peened 12Cr martensitic stainless steel to determine the location of fatigue crack initiation sites from pits of varying depth. Multiple cracks, including fully-developed cracks (surface-breaking cracks extending beyond the pit base) and non-fully-developed cracks, were observed for pits of depth greater than 70 μm. If the longest crack at each side of the pit was assumed to be the dominant crack, most dominant cracks did not initiate from the pit base despite a propensity for macro-stress and strain localisation to the pit base. These results indicate that common assumptions about crack initiation from pits in shot-peened steel can be misleading. Knowledge of the location of crack initiation sites and how the crack develops is required to characterise the mechanical driving force for the crack prior to attainment of a fully-developed crack.

1. Introduction

Shot peening is commonly applied to engineering components to induce a residual compressive stress field into the surface of the metal and thereby increase the fatigue life. Most research has focused on the beneficial effect of the shot-peened surface [1–3] including testing with mechanical notches [4]. However, where cracks develop from corrosion pits there is significant uncertainty as to the residual benefit of shot peening, especially when the pit depth approaches that of the compressive stress layer. While there is awareness of this possibility, there has been surprisingly little research to investigate the effect of pitting corrosion on the fatigue life of shot-peened components. In previous fatigue studies, notches/artificial pits were introduced by electrical discharge machining [5,6], or by drilling to a certain size followed by a corroding treatment with nitric acid [7] and deductions made about the location of crack initiation. However, artificial pits generated by these methods may not be representative of the macro-geometry of real pits. Also, a real pit will exhibit microtopographical features that would be expected to affect the stress/strain distribution at a local level [8,9] and consequently the crack initiation site. Hence, simple fracture mechanics based approaches assuming the initial crack depth is that of the pit depth, or projections based on the pit depth, may not be reliable.

In this report, the site(s) of fatigue crack initiation from "real" pits was investigated in a shot-peened 12Cr martensitic stainless steel, typical of that used for steam turbine blades. Pits with a range of depths on the same specimen were generated electrochemically. Fatigue tests were then conducted but interrupted in order to capture the early stages of crack development before the cracks were fully developed and information about the initiation location lost.

2. Experimental procedure

The shot-peening was undertaken on all faces in the gauge length of flat dog-bone tensile specimens (gauge length 20 mm, thickness 4 mm and width 6 mm) of FV566 steel by Metal Improvement Company (MIC, UK) using the procedure S170 8-12A [10], typical of industry practice for this steam turbine application. The Almen intensity was 8-12A, the shot diameter was 0.432 mm; shot hardness (HRC) was 48–52, coverage was 200% and the shot velocity 56 m/s.

X-ray diffraction (XRD) combined with layer removal by electropolishing was used to determine the residual stress distribution. The measurements were performed using a Pulstec μ-X360 diffractometer using Cr Ka radiation (u-X360, Pulstec Industrial Co., Ltd, Japan). Electro polishing was performed using a standard electrolytic solution supplied by Pulstec and the hand held Pulstec electropolishing unit. Each polishing step was conducted for 30 s after which the polishing depth was measured. Polishing was

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repeated until the desired depth was reached. The residual stress was then re-measured and the process repeated.

The surface roughness were measured using a "PGI 1000" stylus profilometer (Taylor Hobson, UK) with a 2 μm diameter stylus tip, a traversing speed of 0.25 mm per second and a data spacing of 0.125 μm.

Eight pits (positioned in a line, parallel to the stress axis and positioned about 1–2 mm apart) with depths ranging from 31 μm to 238 μm were produced on the shot-peened dog-bone tensile specimen using a galvanostatic droplet technique [11], which had been shown to replicate well the geometrical features of pits in service. A single droplet of 0.1 M NaCl solution (volume = 2 ml) from a capillary tip with outer diameter of 0.47 mm and inner diameter of 0.13 mm is placed near the centre of the gauge length and the specimen polarised anodically using a galvanostatic current of 20 μA supplied by a computer controlled potentiostat (Gill AC, ACM Instruments, UK). A thin platinum wire of diameter 0.1 mm is used as the counter electrode. The depth of the pit is controlled by the duration of the anodic polarisation. Pits could be produced with a variation in depth less than 10% of the target value. The composition of FV566 steel is shown in Table 1.

Fatigue testing, with a sinusoidal wave form, a frequency of 1 Hz and a stress ratio of 0.1 was carried out in air on the multi-pitted specimen using an 8801 Fatigue Testing Machine (Instron, UK). The challenge is knowing when to stop the test to capture the early stages of crack initiation for the different pit depths on the specimen. The approach made was to stop the test when a crack was clearly observable for the deepest pit with the expectation that embryonic cracks would then be evident for the smaller pits. The initial maximum stress (σ_{max}) was 622 MPa (75% $\sigma_{0.2}$); no cracks were observed after 112,000 cycles. The maximum stress was subsequently increased to 663 MPa (80% $\sigma_{0.2}$). Fatigue testing was undertaken until a surface crack of ~2.5 mm in length was detected at the deepest pit, at which point the testing was stopped. After testing, the specimen was ultrasonically cleaned with Super Clarke's solution (5 g/l of 1,3-di-n-butyl-2-thiourea in 18.9% HCl) for 1 h, followed by ultrasonic cleaning with a solution of 32 g/l of $KMnO_4$ and 100 g/l of NaOH at 80 °C for 15 min. The depth of the pits on the specimen was measured by a Nikon Measuring Microscope (MM-60, Nickon, Japan) before and after chemical cleaning and there was no significant change.

Serial sectioning was used to study the crack initiation sites. One of the fundamental issues in layer removal is to check with precision the amount and uniformity of material removed. The four ball method provides such reassurance. The specimen and four metal balls with a well-defined diameter of 2500 μm as reference were mounted in bakelite. The specimen was then polished layer by layer with the final surface polished with a 1 μm diamond suspension at the end of each serial sectioning step. The diameters of the four metal balls were measured after each serial sectioning step to calculate the depth of material removed from the specimen and the average of the values obtained used to define the average depth removed (after each step, the maximum difference in the values obtained from four balls was usually less than 5 μm but increasing to 15 μm at pit depths in excess of 150 μm). Since the metal balls were not revealed until the first serial sectioning step, the removed depth of the first step was estimated from the depth difference of one pit before and after the serial sectioning process.

The average depth of the material removed each step was about 16 μm with smaller increments initially and larger increments at greater depths. At each stage, images of all eight pits and cracks were captured in the scanning electron microscope (SUPRA 40, SEISS, Germany). The sequence of polishing and subsequent examination was continued until the base of the pit was no longer observed.

3. Results and discussion

3.1. Characterisation of pit depth

Table 2 compares the pit depths obtained via the serial sectioning process with those determined by optical microscopy (OM). For each pit, the corresponding depth of material removed at which the pit was last observed (the second column in the table) and first absent (the third column) during the serial sectioning process is listed. The fourth column shows the pit depth estimated from the mean of these two values. The fifth column shows the depth of pits, measured by optical microscopy. For a few pits there was some discrepancy between the estimates from microscopy and those derived from serial sectioning. For example, Pit 6 was estimated to have a depth of 185 μm according to microscopy measurements but the pit was still observed after a depth of removal of 201 μm. There is some contribution to this difference from non-uniform polishing; also local variations in geometry of the pit base may not have been picked up readily in microscopic measurement (the uncertainty of OM measurement is ±0.5 μm). In this report, the depth of pits refers to the mean value from sectioning.

3.2. Characterisation of cracking from pits

Cracks were observed for pits with depths varying from 70 μm to 240 μm but no crack was observed for the pit of 31 μm depth. Fig. 1 shows a series of section images for a pit of 74 μm depth with a surface-breaking crack on the right of the pit. A surface crack was first observed after fatigue testing, before serial sectioning. The crack can also be seen after layer removal steps of 20 μm, 27 μm and 36 μm, while it was absent after a further serial sectioning step. By implication the cracks initiated nearer the pit surface and not at the pit base.

Fig. 2 provides an example of a crack that initiated near the pit base, in this case for a pit of 209 μm depth. After a layer removal of 133 μm, a crack can be detected that gets progressively larger with depth of material removed.

The question then posed concerns the propensity for cracks to initiate near the base as opposed to near the surface of the pit. To address that question we need to quantify the location of initiated cracks as a function of pit depth. This can be challenging as our observations show that while significant cracking can be observed near surface, for example, cracks from the pit base were not absent in every case but the cracks were smaller. This raises the issue in defining the significance of a crack relative to its size. Fig. 3 shows a pit of 209 μm depth with a layer removal of 51 μm (with irregular shape and microtopographical features) and the corresponding multiple cracks at the left side of the pit. Crack length refers to the length from the edge of the pit to the crack tip perpendicular to the stress axis, as shown in Fig. 3. In this study, a crack is defined as such only when the crack length is greater than 5 μm to

Table 1
Composition of FV566 steel (in mass %).

Steel	C	Si	Mn	P	S	Cr	Mo	Ni	V	N_b	Fe
Composition	0.12	0.30	0.86	0.013	0.002	11.73	1.64	2.59	0.28	0.13	Bal