

Measurement Note

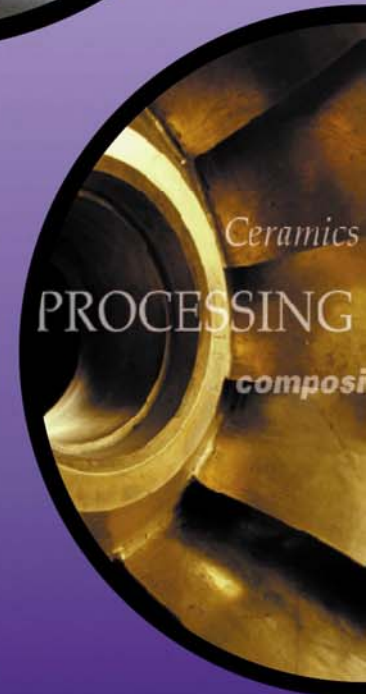
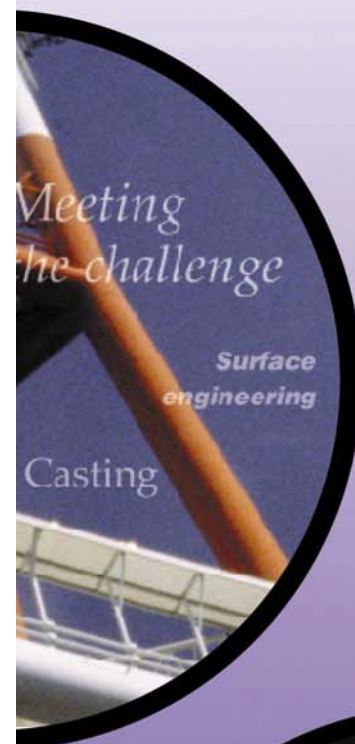
Fractographic Assessment of Powder Metallurgy Steels and Identification of Failure Origins in Notched Transverse Rupture and Notched Bending Fatigue Conditions

Conventional fatigue testing of PM steels, which can be considered to exhibit some ductility on the microscopic scale but can nevertheless be regarded as brittle at the macroscopic level, can be hampered by unpredictability of results due to inappropriate specimen configuration

This Measurement Note demonstrates the use of a V-notch specimen configuration in fatigue which produces fatigue data with a minimum of scatter and to which Linear Elastic Fracture Mechanics (LEFM) can be readily applied to reconcile fatigue behaviour with pre-existing flaws and apparent subcritical flaw growth during fatigue where appropriate.

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Introduction

A common test used in the powder metallurgical (PM) industry for quality control and as a benchmark for mechanical property assessment is the transverse rupture (i.e. bend) test. Although a standard test is available [1] for PM steels, it is not thought appropriate for those PM steels, which although on a microscopic scale can appear to exhibit some ductility, they can be nevertheless regarded as macroscopically brittle. Besides use of the standard test, it is common to find material properties quoted according to an in-house procedure. Most commonly quoted are the results from three-point bend tests with the failure strength (σ_f) defined as the maximum fibre stress at fracture, and calculated from the formula which is based on linear elasticity:

$$\sigma = \frac{3PL}{3BW^2}$$

.....
(1)

Where:

P= rupture load

B=width

W=height

L/2= distance between inner and outer loading points at one end of the test piece.

The formula does not take into account any possible plasticity and that failure of the bend specimen always initiates at the position of maximum stress at the surface. Results of conventional (i.e. un-notched) bend tests on semi-brittle PM steels have been reported for some time [2-4]. However, there are several drawbacks to this apparently simple test. Precautions must be taken to ensure that the test is not influenced by surface conditions such as residual stress or the presence of unintentional stress raisers (e.g. from a poorly prepared metallographic surface). In PM steels exhibiting macroscopic brittle behaviour the volume of the stressed region can also affect the value of rupture strength attained; one of the consequences of brittle fracture from defects outlined originally by Weibull [5], although in recent years it has been shown that this can be

taken into account, with the proviso that the Weibull modulus ("m") is known for at least one specimen configuration. However, this would necessitate conducting enough tests to give a statistically meaningful value of m in the first place. The use of a notched specimen configuration offers the possibility of reduced scatter in both static and fatigue tests due to the relatively small volume of material (i.e. at the notch) subjected to maximum applied bending stress, and also one in which through the use of carefully controlled preparation conditions, the variability in results in un-notched specimens due to defects introduced through sample preparation can be eliminated.

The primary objective of this work was to evaluate the versatility of the notched bend format for performing static and fatigue tests to assess whether static failure and fatigue failure origins could be

- Identified
- Measured
- Modelled

Test System

The test system used was an Instron 8872 rig used in compression mode for the fatigue tests in an Instron 1197 rig for static rupture tests.

Materials, Testpieces and Tests

PM steels

Six grades of PM steel were procured for the test programme as indicated in Table 1. The first two indicated (LGA, LGB) are manufactured from water-atomised powders. The use of water-atomised powders enables production of components by die pressing. After die pressing the present samples were sintered in a mesh belt furnace. The latter four grades (LGC-LGF) were manufactured from gas-atomised powders and then hot isostatically pressed. Some grades were used as HIPed (LGC, LGD); others were tested after being

subjected to post-HIP hot working (LGE, LGF). Two of the grades were tested in the HIPed condition, but also had small quantities of ceramic admixed prior to HIPing.

Testpieces were wire electrodischarge machined (EDM) from larger blocks of material to a nominal, rectangular, 5 mm x 2 mm x 45 mm size. These were then lightly ground using a diamond wheel to remove EDM residues and ensure parallel dimensions (to ± 0.01 mm). A central 45° vee-notch was then ground in the 2 x 45 mm face, being 1mm deep and having a tip radius of 0.1 mm.

The following test types were examined:

- Static load
- Fatigue load

Six sets of 30 specimens were initially tested, the first five of each being tested in static bending. The static tests were undertaken on a Instron 1197 rig with a 100 kN load cell. Transmission of the load from load cell to specimen was by means of compression platens and a four point bending jig with no articulation. The inner span was 10 mm and the outer span 30mm, the bending being imparted to the specimen by means of hardmetal rollers (fixed in the lateral plane, but free to roll). The machine was used under displacement control at a rate of 0.5 mm.min⁻¹ and the specimen loaded until rupture occurred. Precautions were taken to avoid post-rupture damage in the test rig by means of rubber strips under the tensile face of the bending jig.

Fatigue tests were undertaken on an Instron 8872 using the same bending jig as the static tests. Fatigue tests were undertaken using an R ratio equal to 0.1 and at a frequency of 10Hz. The maximum applied tensile bending stress applied was dependent on the mean rupture stress determined from the static tests; initial fatigue tests were conducted using a maximum applied tensile bending stress equal to 95% of mean static rupture stress for the sintered

materials and 90% thereof for the HIPed materials, with decrements of 5% and 10% being applied in subsequent tests in order to build up S-N plots for the respective materials. Up to 25 specimens were used in order to construct the S-N plots. The runout condition was arbitrarily assigned as being 10⁷ cycles.

Results and Discussion

Initial static tests

Five samples of each material were ruptured in four point bending as outlined above. The mean strength data are outlined in Table 3; these were used as a basis for the fatigue loading regimes for each batch of samples and the samples were retained for fractographic examination and flaw size analysis.

Fatigue tests

It can be seen from Figure 2 that all the materials under investigation exhibited S-N behaviour with little scatter and that the S-N plots fall into two broad bands; the upper band corresponding to the materials which were processed from gas atomised powders and the lower band corresponding to the water atomised powder materials. Also illustrated are the static results (arbitrarily assigned a value of 1 cycle on the x-axis due to the log scale). Fractography was conducted on the specimens initially by optical microscopy to note position of the failure-initiating region. Grazing incidence illumination [6] was also used where necessary to aid identification. Detailed fractographic examination was conducted using a Hitachi 6400 field emission scanning electron microscope (FESEM). In the materials produced from gas-atomised powders, identification of failure initiating regions was relatively straightforward, being characterised by radial fracture markings pointing back to the origin. Microstructural features at the centre of the initiating region were identified easily, being pores, carbides, sulphides or ceramic particles. In some cases a relatively smooth area around the initiating feature was also seen (a good example is illustrated in Figure 7), akin to

the fracture mirrors seen in certain ceramic fractures [6] though as with some ceramic fracture mirrors, the boundaries of the peripheral regions seen in these PM materials were often not very distinct. Dimensions of both the fracture-initiating feature and of this peripheral region were noted for future reference in conjunction with modelling based on linear elastic fracture mechanics (LEFM). In the case of the steels manufactured from water-atomised powders, identification of the initiating feature was hampered by the lack of radiating fracture markings seen in their HIPed counterparts; thought to be a consequence of the lower fatigue strengths exhibited by these materials. Failure appeared to have initiated at carbides and pores (in LGA) and additionally from sulphides (in LGB only). The dimensions of possible initiating features were noted as with the gas-atomised based materials for use in conjunction with LEFM.

Application of Linear Elastic Fracture Mechanics to Notched Fatigue

The stress at the notch root in both the static and the fatigue tests is given by multiplying that calculated according to equation (1) by a stress concentration factor, k_t . In the static tests no deviations from linearity were found in the load-deflection traces and hence it was felt justified in using the linear elastic bending formula in calculating the local applied stress at the notch root.

From linear elastic fracture mechanics, the critical stress intensity factor, K_{IC} , is related to the size of the initiating defect (a) by:

$$K_{IC} = Y\sigma\sqrt{\pi a} \dots\dots\dots (2)$$

Where:

Y = a flaw shape factor.

In mode I loading, K_{IC} can be considered to be equal K_{IC} , plane strain fracture toughness if plane strain conditions are attained; this is a material property.

In the case of fracture initiating defects in semi-brittle PM steels, previous studies have taken into account the variation in applied stress intensity factor (calculated assuming penny-shaped crack geometries) with ellipticity of the defect (the c/a ratio). However, previous fractographic evidence has indicated that the eventual failure initiating zones are in general semicircular. In the present study predominately subsurface defects have been found, and has enabled a defect size analysis to be undertaken without resorting to the use of various flaw shape factors. Using the theoretical LEFM Model a plot showing the predicted size of subsurface circular critical flaws is shown in Figure 3. Defect size analysis has been taken using K_{IC} data from edge toughness experiments [7] and published data in conjunction with dimensions of the observed fracture initiating features and regions. Fractographs illustrating both actual initiating features and dimensions of theoretical critical defects for the appropriate maximum applied stress are shown in Figures 4 to 8. Defect size analyses are shown in Figures 9 to 16 as plots of the square root of defect area against local rupture stress at the notch root for the theoretical critical size defects from LEFM, the actual origin and the apparent growth region (the latter where identifiable). It can be seen that in almost all cases that the apparent origin size, from fractography, is almost always smaller than that predicted by LEFM. This implies that the steels do not behave in a classical (i.e. Griffith) manner where fast fracture occurs from a critical sized flaw and there must be another catastrophic failure criterion; hence the measurement of the zones peripheral to the initiating defect. It can be seen that in the case of LGE and LGF there are good correlations between theoretical defect size and that of the observed peripheral region, which gives credence to it being a possible zone of (sub-critical) crack growth prior to failure. Within the measured populations of initiating defects it can be seen that some materials appear to possess a narrow band of defect sizes (LGB, LGC, LGE and LGF); namely in materials where failure occurred from an added sulphide in a sintered material (LGB) or from a fairly uniformly sized population of carbides in HIPed material. Identification the apparent growth region in the HIPed material without subsequent forging (LGC) was hampered by the presence of prior particle boundaries which tended to obscure the

peripheral regions around the initiating features. The (fast) fracture surfaces in this material also tended to show a preference for fracture along prior particle boundaries. Metallography revealed that the inter carbide spacings in these regions to be apparently smaller than the main bulk of the material; this may account for preferential fracture and the possibility of a local fracture criterion during growth from prior particle boundary region which is yet further removed from KIC due not only to crack length (i.e. R-curve) considerations but also to a local microstructure which is somewhat different to that found in the forged materials. With added ceramic (LGD) there was a good correlation between the size of the initiating feature and the theoretical critical defect size. Either instantaneous fracture of the ceramic (and associated microporosity where present) and the main body of the surrounding steel occurred simultaneously or subcritical crack growth occurred within the ceramic until a (local) fast fracture criterion was established. The correlations between theoretical conditions for unstable crack propagation (from LEFM) and observed microstructural features were not found as frequently for the materials based on water-atomised powders (LGA and LGB). This may have been due to the difficulty in identifying initiating features and possible (subcritical) growth regions.

Further static tests

The PM steel grades which were found to possess (relatively) good correlations between (a) initiating features and unstable crack propagation criteria and (b) between apparent growth regions and unstable crack propagation criteria, namely LGD and LGE respectively, were selected for further investigation into correlations between observed fracture initiation and/or growth features and LEFM. To this end a further 25 specimens of LGD and LGE were tested under the same static bending conditions as the initial five used to establish initial conditions for the fatigue test programme. Plots of square root of defect area against local rupture stress (again from linear elastic calculations) detailing theoretical critical defect size, actual origins and actual apparent growth

regions were made. In this case fracture-initiating features were readily identifiable as was the case with the fatigue specimens, but where the initiating features appeared to be subcritical (from LEFM), identification of growth regions was more problematic than with the fatigue specimens. Initiating features were of the same species as the respective fatigue specimens; overwhelmingly of subcritical size in the case of LGE, but in the case of LGD, in five out of the 30 tests, critical size defects (ceramic particles) were found. To put the utility of the notch bend configuration to test, Weibull analysis was conducted on LGD to establish whether in fact the apparent presence of the subcritical and critical defects would be reflected in the Weibull plot; there being effectively two populations of defects; both discrete ceramic particles and (ceramic+matrix) defects. As can be seen in Figure 17 this does indeed to be the case; evidenced by the “knee” in the plot, indicating a bimodal flaw population. The points above the knee in Figure 17 are those which correspond to theoretical fast fracture from a combined ceramic/matrix region, and those below the knee correspond to theoretical fast fracture from a discrete ceramic particle. A Weibull plot for LGE is shown in Figure 18 and is also bimodal, but again it appears from the defect size analysis to be due to failure which in some cases may occur subsequent to subcritical crack growth, whilst in others this appeared not to be a necessary precursor to (fast) fracture.

Code	Supplier	Supplier Code	Notes
LGA	Federal Mogul Sintered Products Ltd	36xxPH Single Temper	
LGB	Federal Mogul Sintered Products Ltd	3610	Added Sulphide*
LGC	Saveker Steels Ltd	30	HIPed
LGD	Saveker Steels Ltd	30Z	Added Ceramic*
LGE	Saveker Steels Ltd	30L	Forged: Longitudinal
LGF	Saveker Steels Ltd	30T	Forged: Transverse

Table 1: Steels tested and suppliers. Note that the compositions of the steels, the nature and percentage addition of the added species (*) have not been revealed by the suppliers.

Test Type	Load Rate	Frequency	R ratio	Load
Static	50Ns ⁻¹			To rupture
Fatigue	According to regime	10Hz	0.1	Peak load: 90% of static, in decrements of either 5*or 10% [†]

Table 2: Outline of test regimes. * Sintered materials; [†]HIPed materials.

Steel	Mean (Nominal) Rupture Strength (GPa)	Notch Root Stress at Rupture (GPa)
LGA	0.61	2.32
LGB	0.57	2.16
LGC	1.09	4.15
LGD	0.85	3.25
LGE	1.14	4.36
LGF	1.34	5.11

Table 3: 4-point mean nominal and notch root rupture stresses used to determine stress regimes in fatigue.

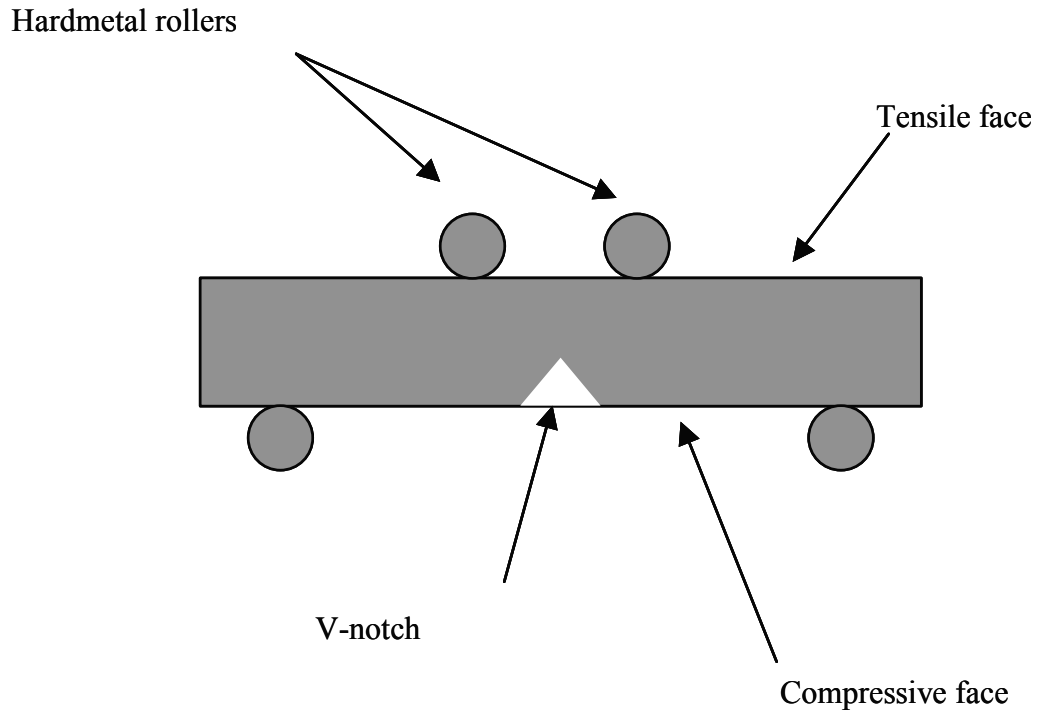


Fig 1 Schematic diagram of specimen configuration (both for static and fatigue tests).

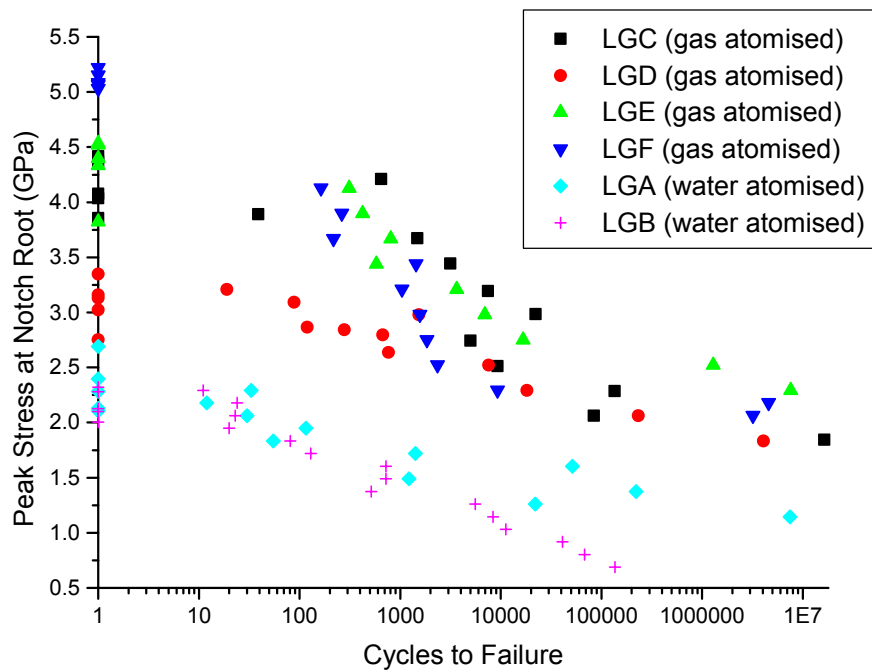


Fig 2 Summary S-N plot for all PM steels investigated

Fracture Mechanics; Subsurface Defect Initiated Failure

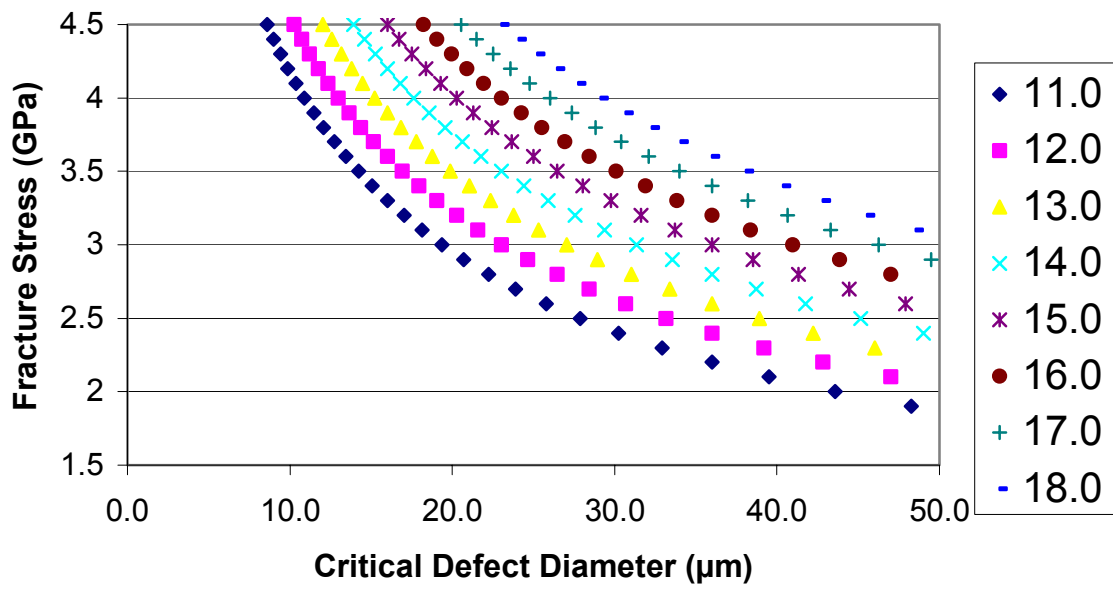


Fig 3: LEFM Model; prediction of subsurface critical defect sizes from K_{IC} values in the range 11.0-18. Fracture stress is that at the notch root.

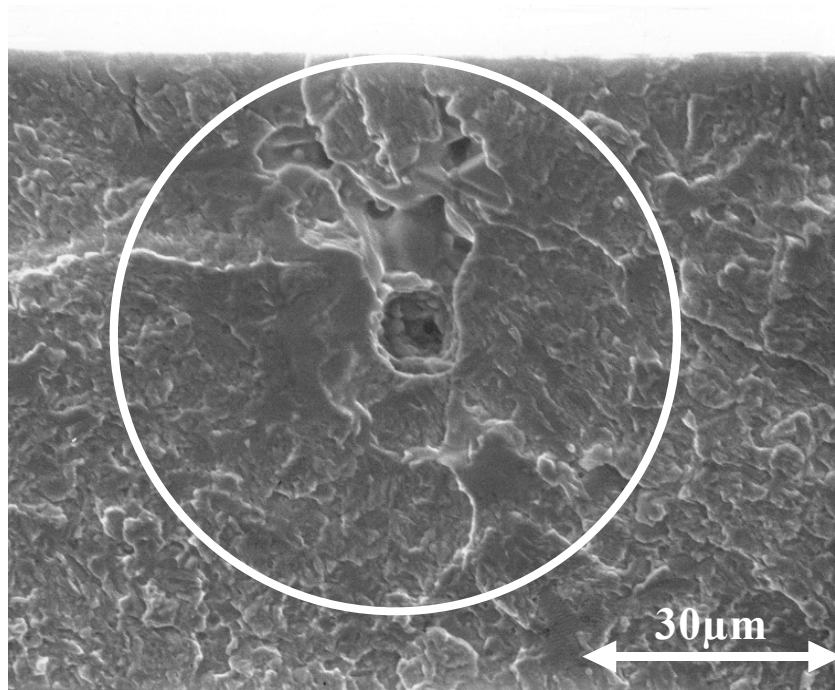


Fig 4 Example of fracture origin in LGA; (maximum local stress=1.15 GPa) showing failure initiating from apparent pore and size of theoretical critical flaw (from LEFM).

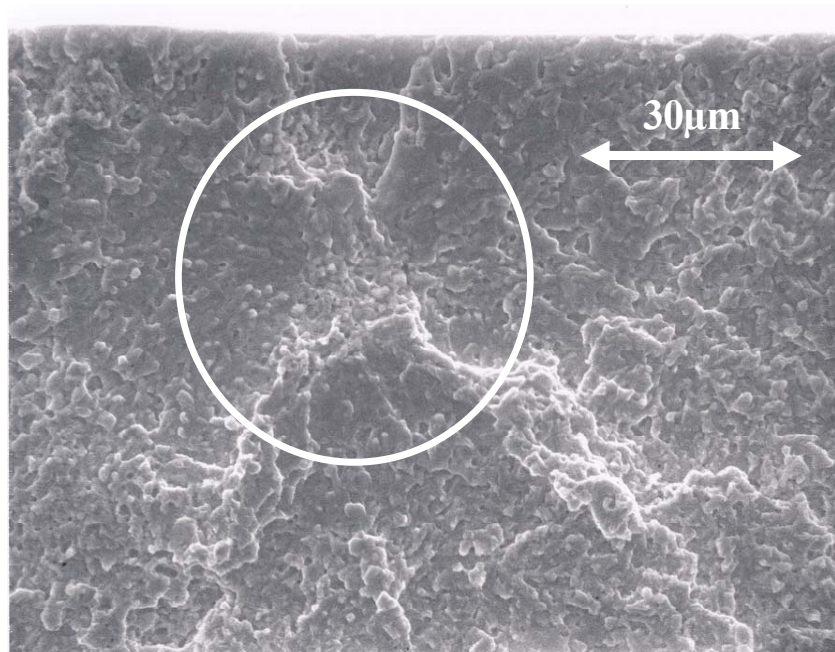


Fig 5: Example of fatigue failure origin: LGD (maximum local stress = 1.83 GPa), showing size of theoretical critical flaw (from LEFM); prior particle triple point with ceramic and microporosity.

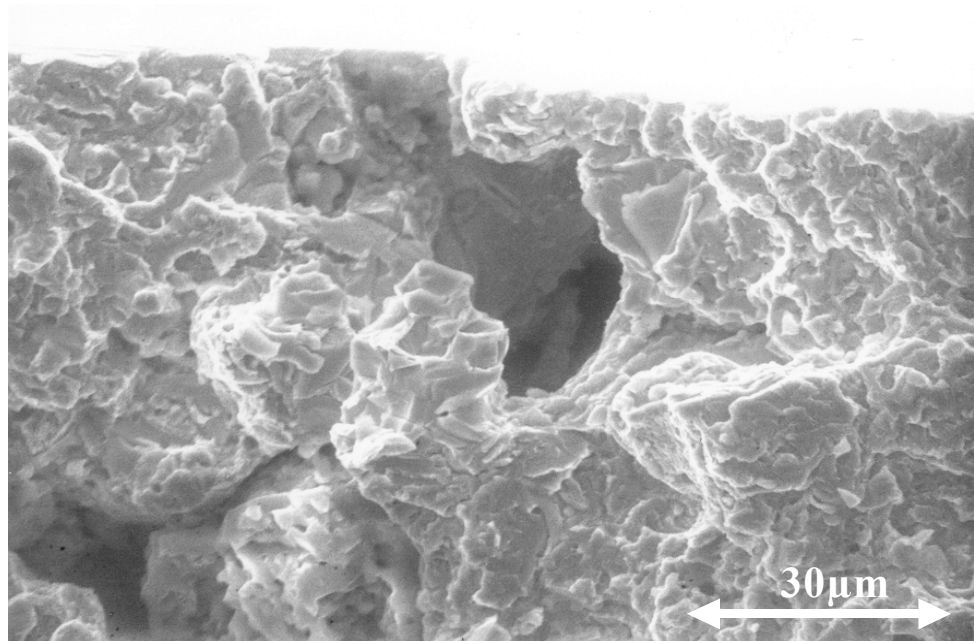


Fig 6 Example of fatigue failure origin in LGB; sulphide particle + apparent pore (maximum local stress = 2.292 GPa)

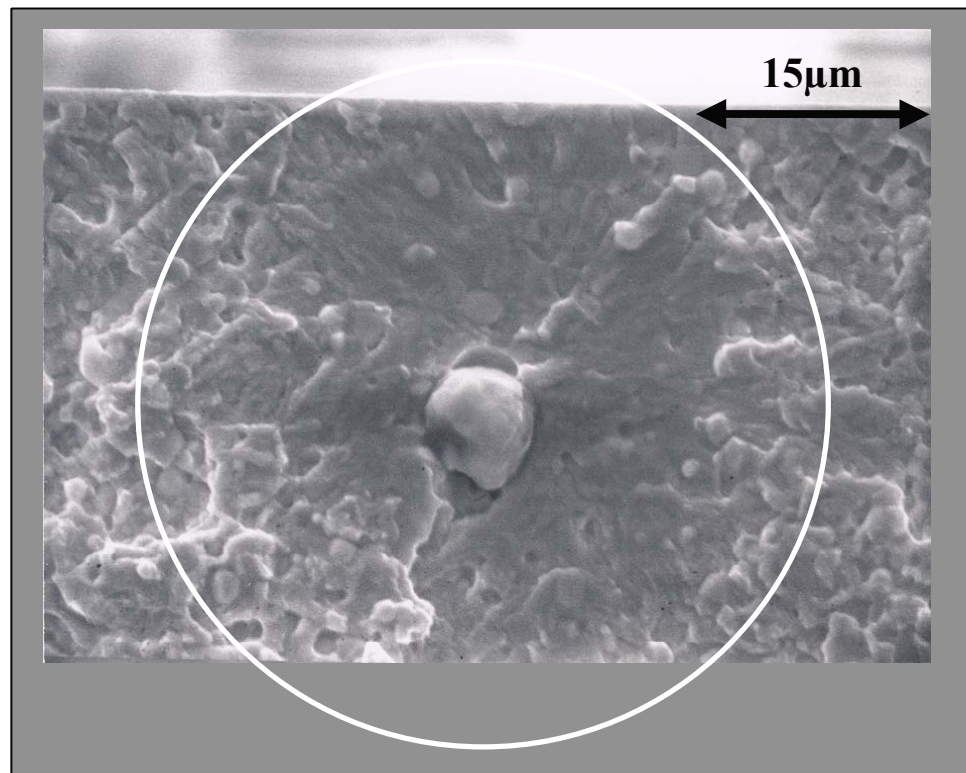


Fig 7: Example of fatigue failure origin: LGE ; (maximum local stress = 2.06 GPa) showing failure from initiating at sulphide + pore and size of theoretical critical flaw (from LEFM).

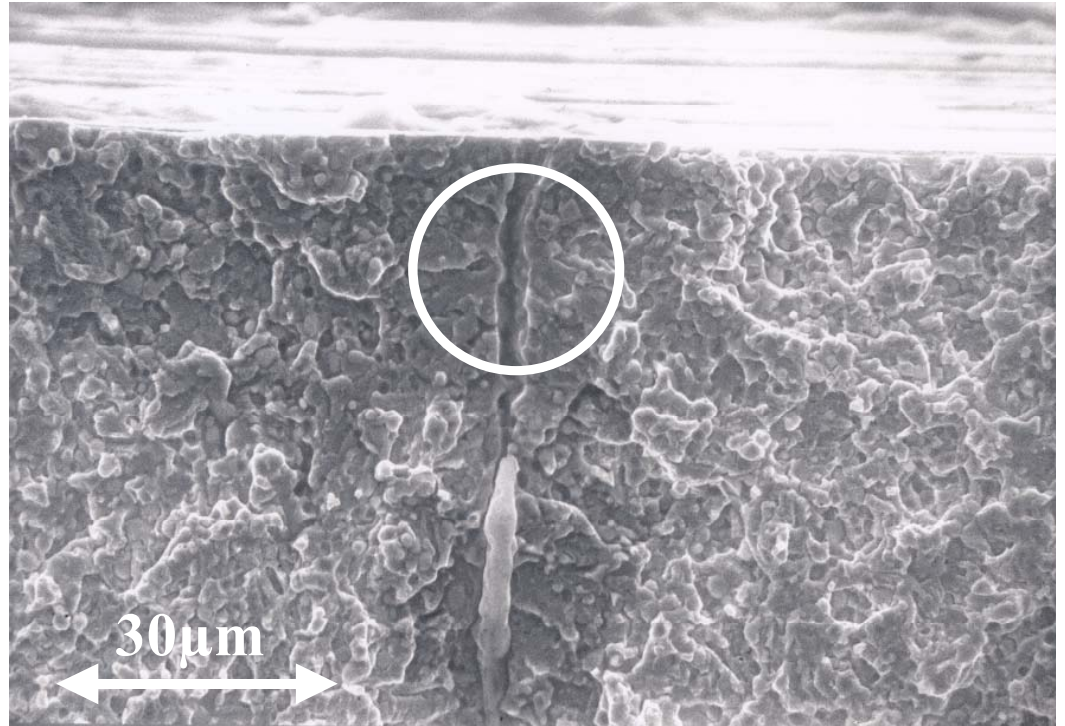


Fig 8: Example of fatigue failure origin: LGF; (maximum local stress = 2.98 GPa) showing size of theoretical critical flaw (from LEFM).

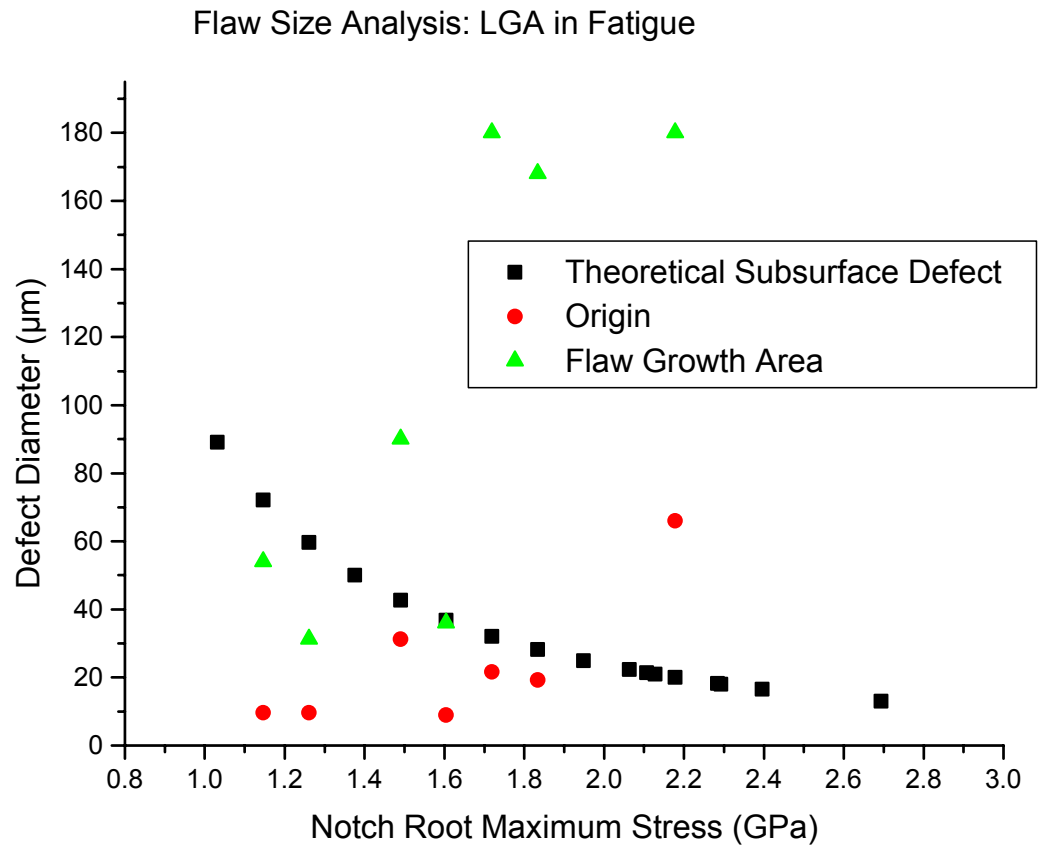


Fig 9: Flaw size analysis of LGA series in fatigue; actual origin size and growth areas (where identifiable) compared with theoretical origin (from LEFM).

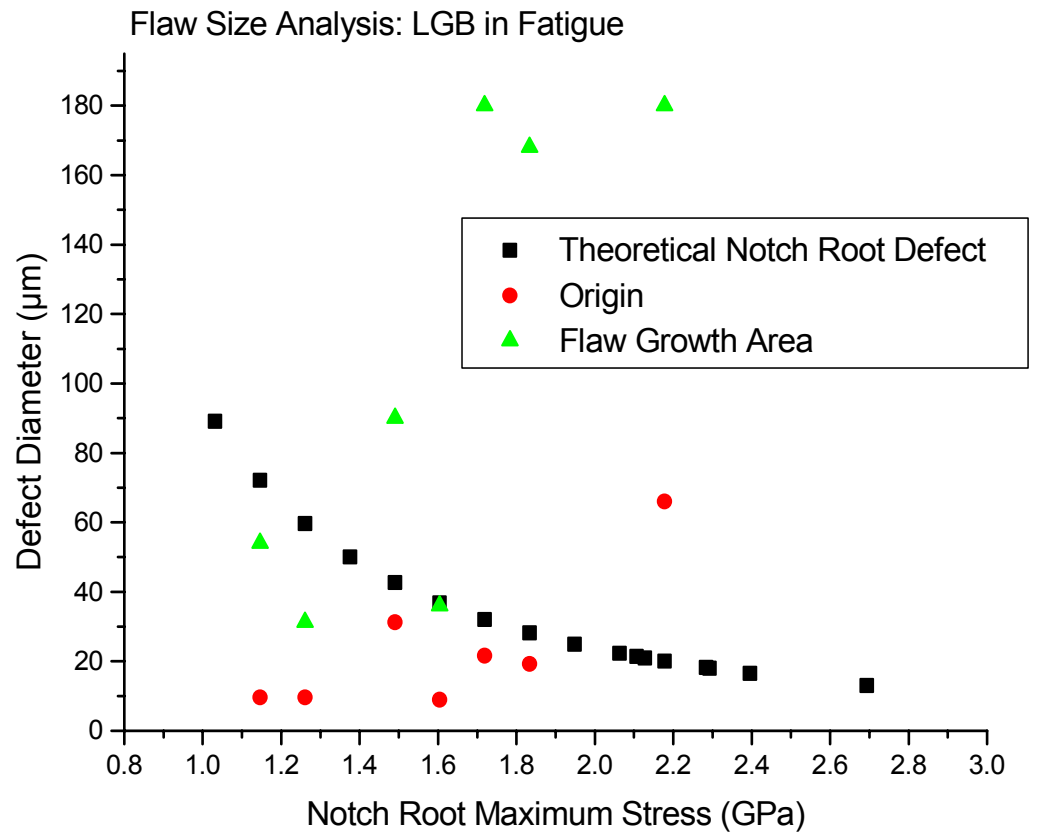


Fig 10: Flaw size analysis of LGB series in fatigue; actual origin size and growth areas (where identifiable) compared with theoretical origin (from LEFM).

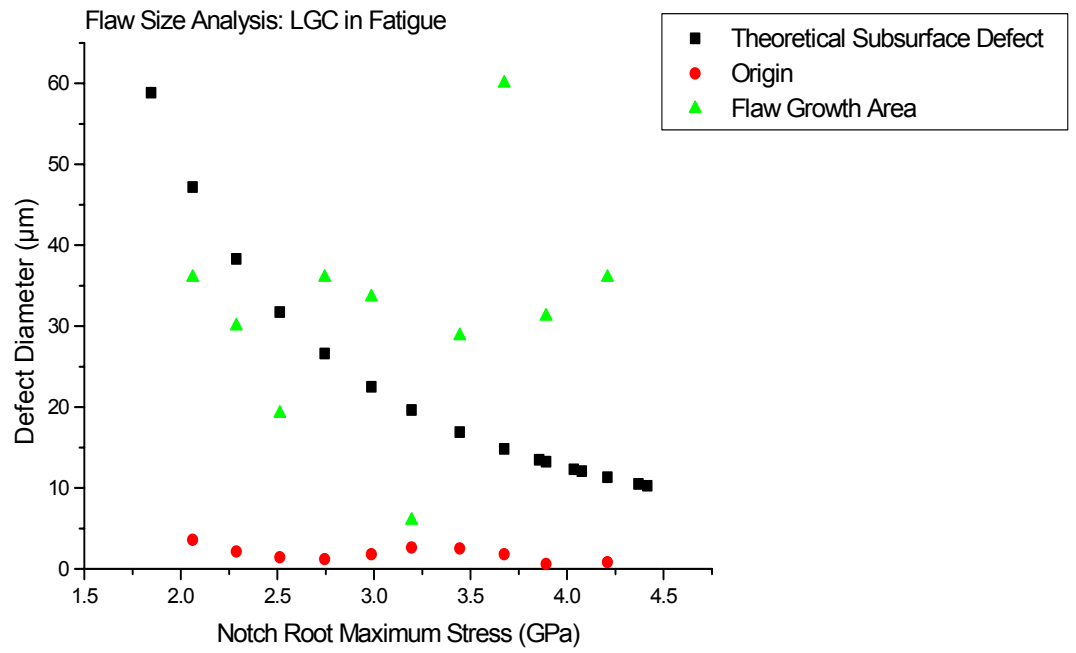


Fig 11: Flaw size analysis of LGC series in fatigue; actual origin size and growth areas (where identifiable) compared with theoretical origin (from LEFM).

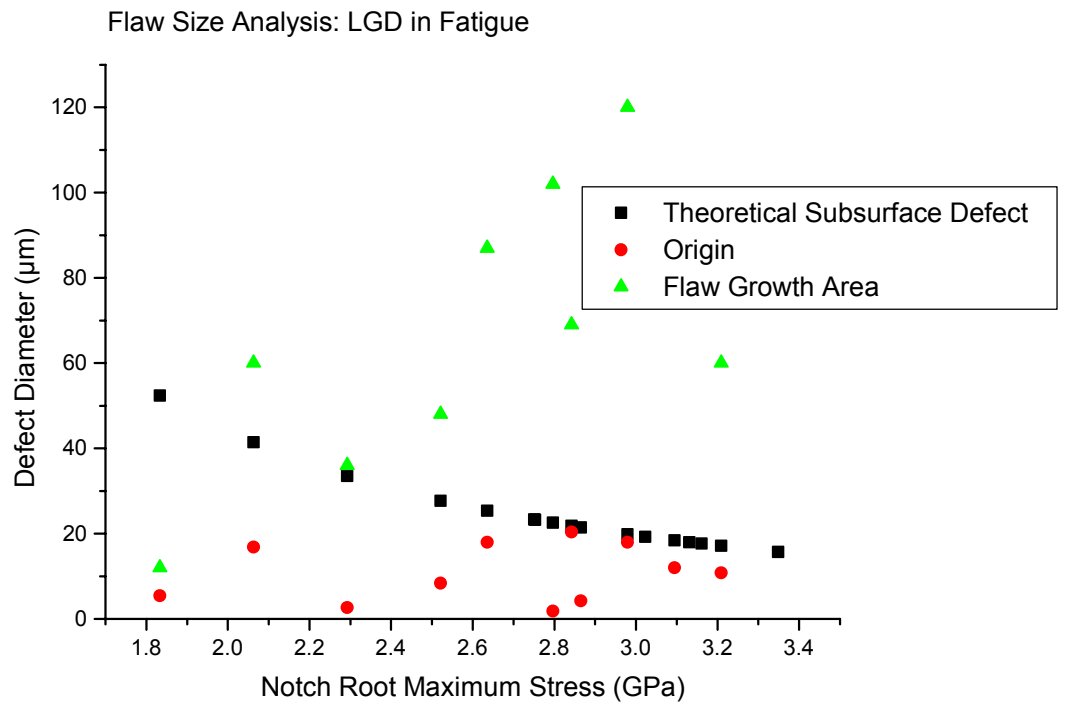


Fig 12: Flaw size analysis of LGD series in fatigue; actual origin size and growth areas (where identifiable) compared with theoretical origin (from LEFM).

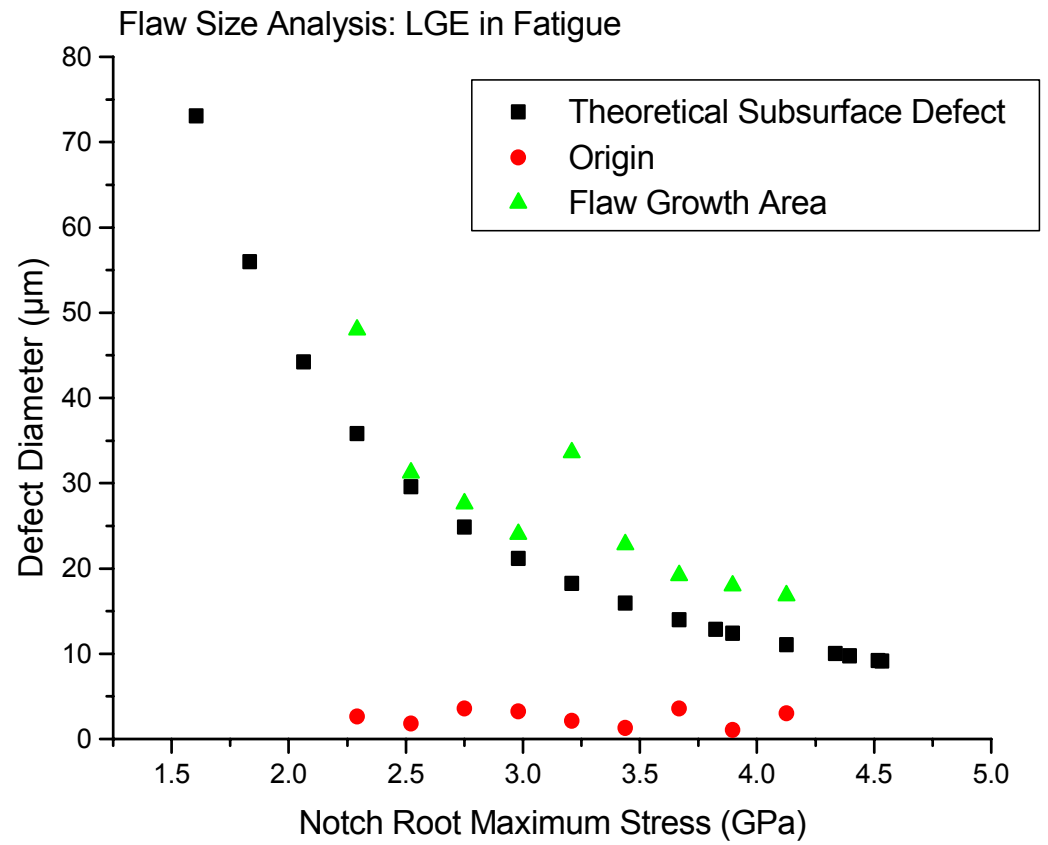


Fig 13: Flaw size analysis of LGE series in fatigue; actual origin size and growth areas (where identifiable) compared with theoretical origin (from LEFM).

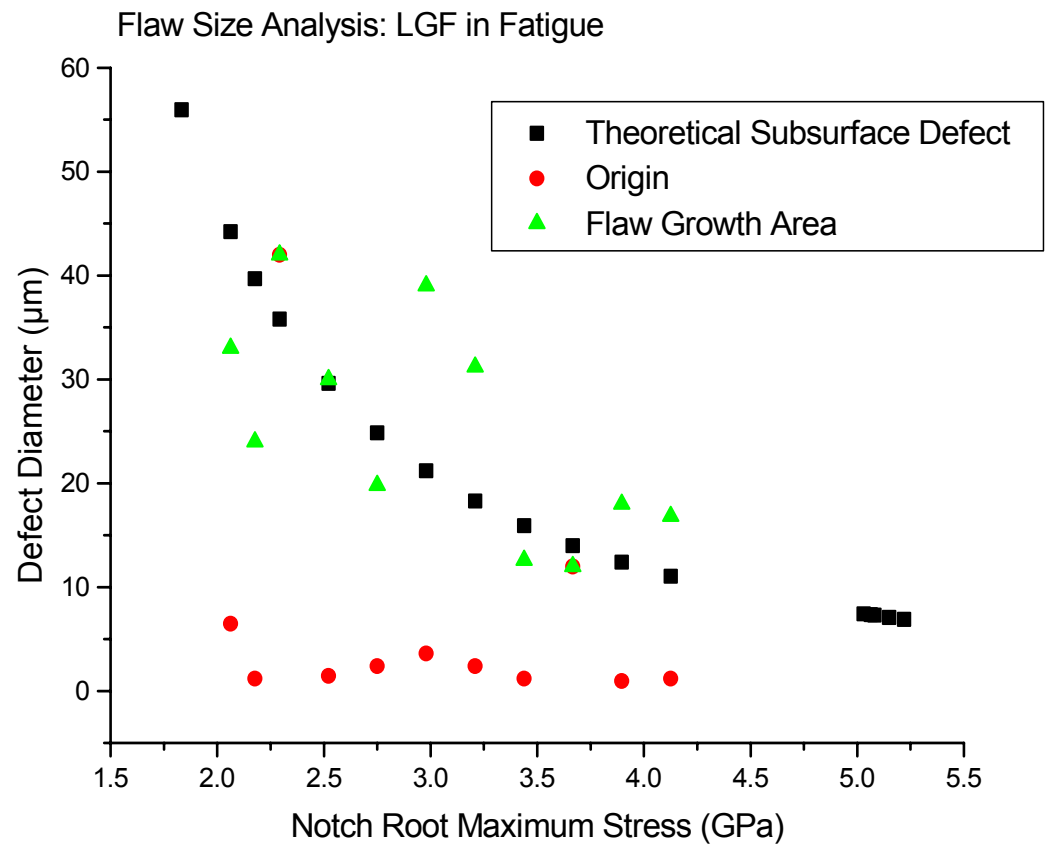


Fig 14: Flaw size analysis of LGF series in fatigue; actual origin size and growth areas (where identifiable) compared with theoretical origin (from LEFM).

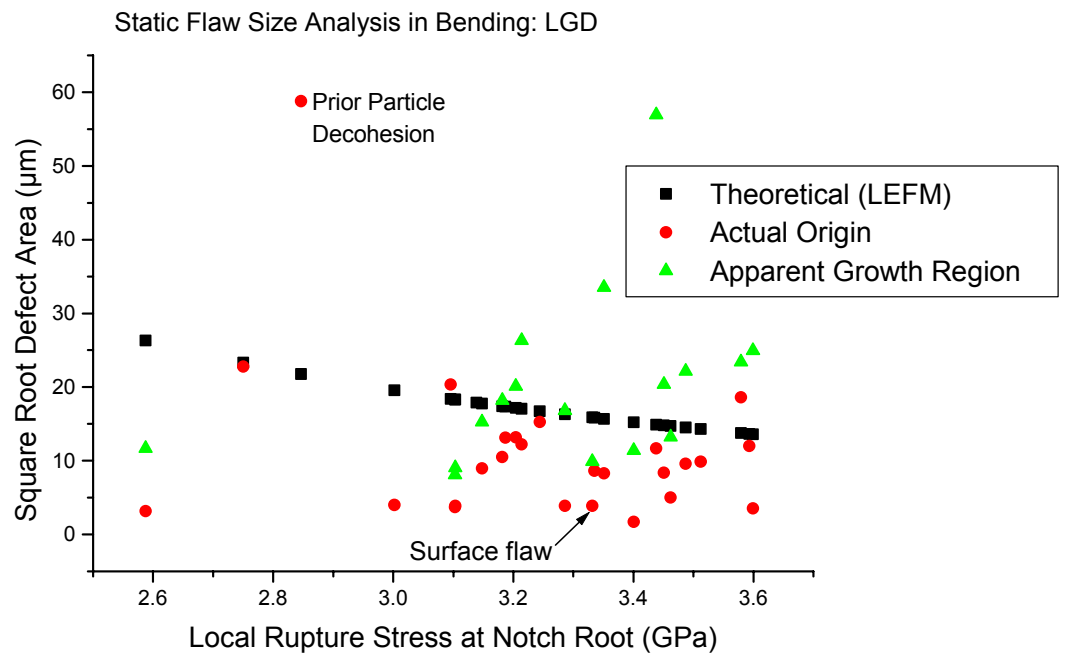


Fig 15: Flaw size analysis of LGD series in static bending; actual origin size and growth areas (where identifiable) compared with theoretical origin (from LEFM).

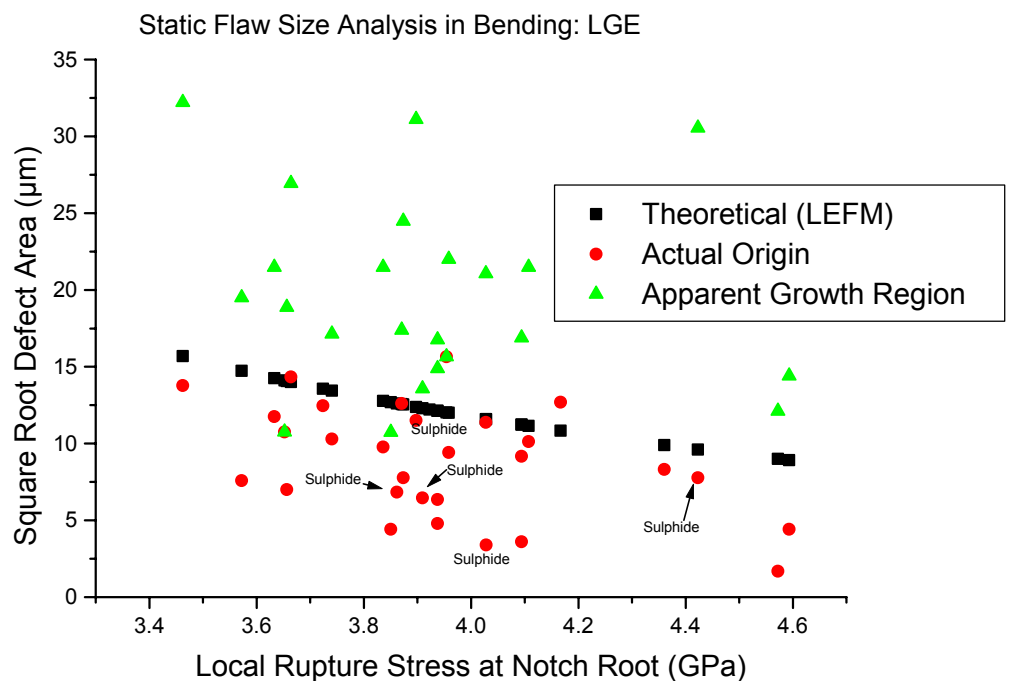


Fig 16: Flaw size analysis of LGE series in static bending; actual origin size and growth areas (where identifiable) compared with theoretical origin (from LEFM). Note that where failure is indicated

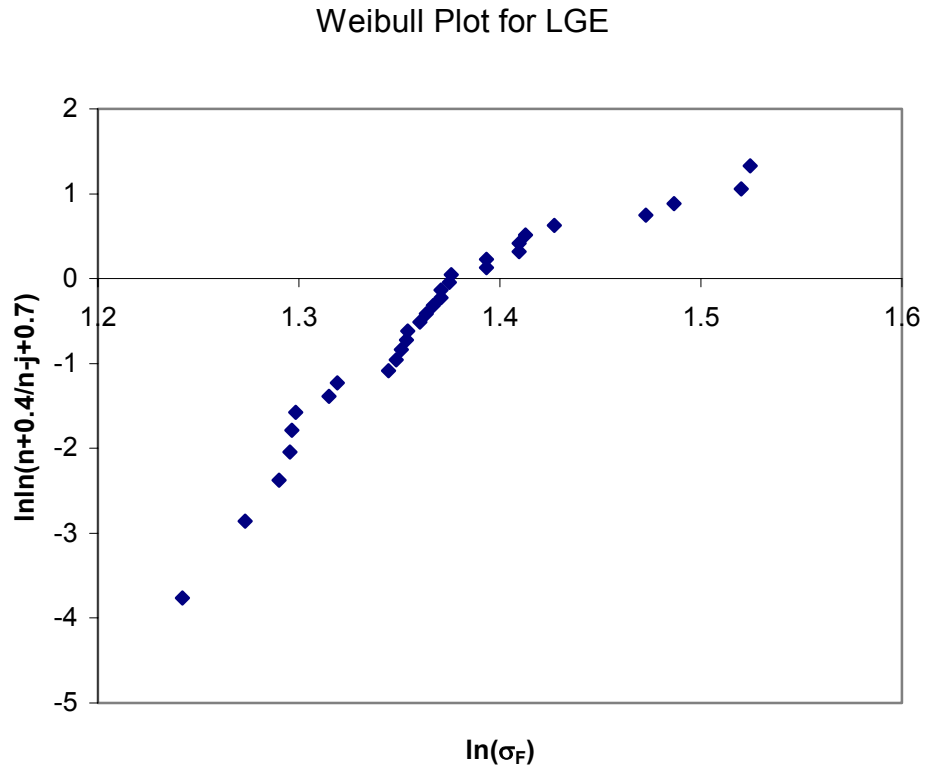


Fig 17: Weibull plot for LGE; vee-notch specimens in 4-point bending.

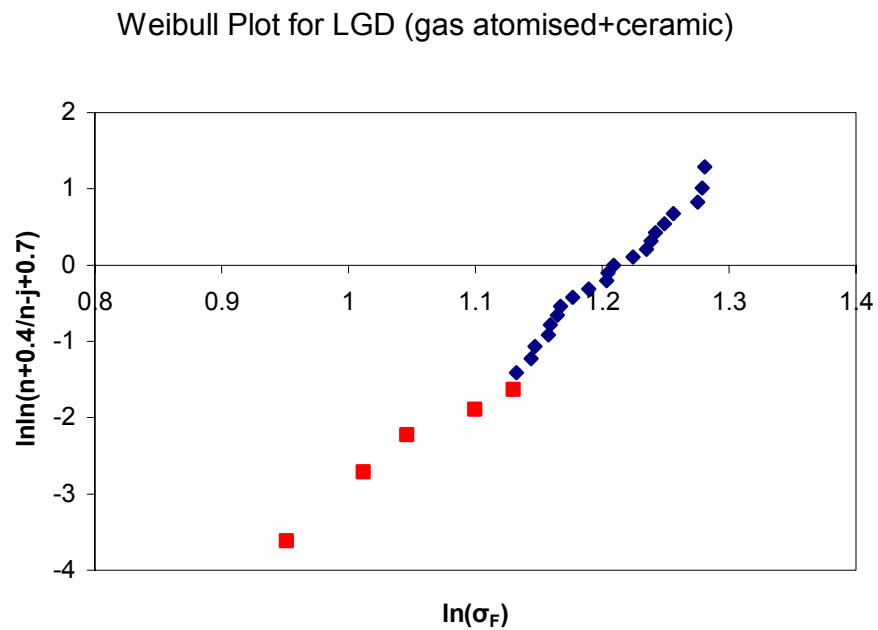


Fig 18: Weibull plot for LGD; vee-notch specimens in 4-point bending. Note that the lowest five points on the plot (i.e. below the knee) correspond to failure initiated by a ceramic particle or cluster without any apparent growth region.

CONCLUSIONS

Fatigue Behaviour:

a) All materials show S-N behaviour, which has considerably less scatter than other methods; thought due to microstructure-initiated failure at or below the notch root.

b) In terms of the S-N behaviour, two generic families of curves were generated which corresponded with the processing routes for the steels studied; namely gas atomisation followed by hot isostatic pressing (in some cases with additional post-HIP working HIPed/(HIPed+forged) and (pressed+sintered)

c) Some show possible endurance limit in fatigue

d) In the HIPed and forged grades (LGE, LGF) a good correlation between size of theoretical defect size and observed growth region (regardless of orientation) was found.

e) Flaw size analysis for HIPed grade with added ceramic seems to indicate fast fracture can occur from single ceramic particles origins; in other cases subcritical crack growth would appear to occur prior to catastrophic failure.

f) Poor correlation in the LGC grade between size of theoretical defect size and fracture surface features maybe due to difficulty of discerning size of features due to interaction of cracks with prior particle boundaries; this problem was not found in LGE and LGF which are of near-identical composition, but have been subjected to post-HIP working. There may also be a local fracture criterion in this material which is

modified due to the presence of more closely spaced carbides in the initiating regions (prior particle boundaries and boundary triple points).

g) Flaw size analysis in pressed and sintered grades (LGA, LGB) did not correlate with either fast fracture or (growth + fast fracture) from pre-existent defects.

Static Behaviour:

a) Apparent measured origins are in nearly all cases smaller than or equal to predictions from LEFM.

b) Subcritical crack growth can occur, but is not always present. Previous studies by others could not report with certainty on this issue due to insufficient results.

c) Carbides, carbide clusters, zirconia, zirconia clusters, zirconia/carbide clusters range in size from subcritical to almost critical boundary condition.

d) Monotonic s.c.c. growth region boundaries less distinct than those for s.c.c. growth in fatigue.

e) Observed regions attributed to subcritical crack growth are in some cases seen to give reasonable agreement with size of critical initiating feature from LEFM. However, in most cases, boundary between observed "growth" region and bulk of fracture surface may be simply a demarcation in surface roughness resulting from increasing crack velocity.

f) In LGE failure initiates from carbides, carbides+ matrix or sulphides. LEFM in conjunction with fractography would seem to indicate that single carbides are more commonly of subcritical size.

g) Subcritical crack growth in LGE may explain difficulty in reconciling Weibull plot with observed flaws (bimodal plot)

h) In LGD failure occurs from single ZrO_2 particles, clusters thereof or ZrO_2 /carbide clusters. All failures below the “knee” in the Weibull plot are of single ZrO_2 particles. Cross-referencing back to the plot of calculated and actual defect areas reveals that these failures initiated from origins whose size is almost exactly as predicted by LEFM; i.e. they are critical.

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