Crack Paths in Friction Stir Welded 5083-H321 and 5383-H321 Aluminium Alloys

G. R. Bradley¹, D. G. Hattingh², T. C. Yio³ and M. N. James³

¹ Now in Department of Mechanical Engineering, University of Sheffield, Sheffield S1 3JD, England <u>gr_Bradley@yahoo.co.uk</u>

² Department of Mechanical Engineering, PE Technikon, Private Bag X6011, Port Elizabeth 6001, South Africa <u>danieh@petech.ac.za</u>

³ Department of Mechanical and Marine Engineering, University of Plymouth, Plymouth PL4 8AA, England <u>mjames@plymouth.ac.uk</u>

ABSTRACT. Friction stir (FS) welding is a relatively new solid-state welding process that offers high levels of joint performance with minimal preparation and little postweld dressing. The high levels of plastic work induced in the weld zone produce a very fine grain size in the stirred region of the weld (e.g. the nugget), while the low heat input limits residual stresses to a low fraction of the proof strength of the weld metal. These effects are generally beneficial to weld dynamic performance. The peculiar thermomechanical history in the FS weld region leads, however, to particular defects with some unusual effects on crack path, whose occurrence partly depends on crack speed, or growth rate. This paper presents observations regarding specific influences of the friction stir welding process on crack paths and dynamic performance for 5083-H321 and 5383-H321 aluminium alloys, and proposes an explanation for the observations in terms of the weld microstructures and thermomechanical history. The insights presented in this paper can be used to inform optimisation of the weld process parameters, through on-line feedback and control of tool geometry, force footprint, torque and temperature.

INTRODUCTION

Crack paths in fatigue and fracture are complex and difficult to predict, despite a significant body of knowledge regarding the mechanics of crack growth. The field of fracture mechanics, which deals with the behaviour of cracked bodies under load, is now widely regarded as 'mature', in the sense that solutions exist to describe the conditions for crack growth and the path that an ideal crack would take [1]. In practice, however, real materials and microstructures are not homogeneous or isotropic, nor are they continua in the manner assumed by mechanics descriptions.

Thus microstructure and mechanical property variation can interact in relatively subtle ways with fluctuations in applied load and environment. The net result of these interactions is a complexity to crack paths that is incompletely understood at the present time, even under straightforward cases of uniaxial loading. The research community in fatigue crack growth has been exposed for long enough to this complexity, that general assumptions of similarity in origin are often imposed in the interpretation of features of crack paths arising from new processes or materials. When this is done, it is important to ensure that proposed mechanisms encompass all the relevant aspects of the process-structure-property interaction. Although fractography has revealed much about the microscopic mechanisms of fatigue, it is most useful as a tool to provide supporting evidence for theories developed from other considerations. Interpretation of fracture surface features, in the absence of full information regarding their possible causes, is known to be fraught with uncertainty.

The present paper will illustrate this complexity in relation to identifying subtleties in crack path mechanisms for the case of friction stir (FS) welded 5083-H321 and 5383-H321 aluminium alloys. FS welding is a relatively new solid-state process that involves plunging a rotating tool into the joint between faying plates. Once the weld zone has reached an appropriate thermo-mechanical state, the tool is traversed along the joint line and the weld is made by pick-up and transfer of material around the tool, to be then deposited behind it. Figure 1 illustrates the main features associated with a single-pass (SP) FS welded joint.

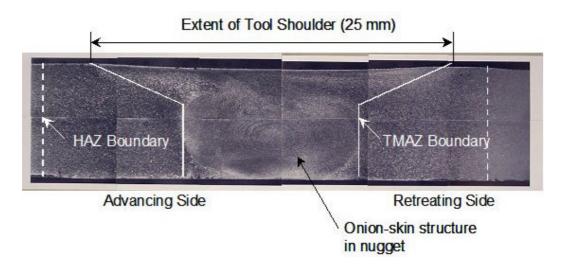


Figure 1. Illustration of the main features associated with a macro-etched SP FS weld.

The advancing side of the weld is defined as existing where the translational velocity along the weld line is additive with the rotational velocity of the tool. The retreating side is the opposite, and the rotational velocity is hence subtractive from the translation. The extensive mechanical work put into the thermomechanically affected weld zone (TMAZ) leads to a fine-grained structure in the weld nugget, which usually etches up as an 'onionskin' structure both in cross-section and along the length of the weld.

It might be expected that this structure would be reflected in crack paths and, as will be detailed in this paper, this occurs to a certain extent. The more interesting aspects relate to the subtlety of the underlying mechanism through which the structure/path interaction is evidenced in, at least, certain FS welded alloys, and the ways in which this affects fatigue and fracture performance. The fractographic evidence offers little insight into the mechanism of the structure/path interaction and can be readily interpreted in a misleading way. To illustrate this, consider the fracture surface shown in Figure 2. The fracture surface shows a fatigue crack in the right of the figure, with fast fracture to the left of the fatigue region. A large planar facet, some 1.5 mm in length (marked with the

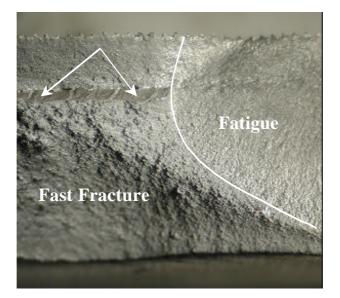


Figure 2. Optical fractograph of an 8 mm thick SP FSW fatigue specimen.

arrows) can be seen in the fast fracture region. Such apparent defect indications are of significant concern, as a major advantage of the FSW process is the absence of cast material in the weld zone and an associated reduction in potential defect population.

Considering the nature of the FSW deformation processes, which involve material entrainment around the tool and subsequent deposition in its wake, this apparent defect seems likely to reflect some microstructure-related aspect of the necessary deformation and forging processes. Indeed, such an explanation was proposed when these features were first observed by the present authors [2] and they were then referred to as 'partial-forging defects'. Subsequent work has led to a refinement of ideas regarding their formation, the conclusions of which are supported by related work from other researchers.

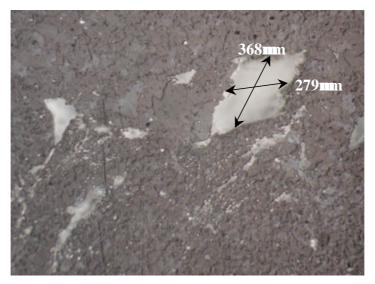
Thus the intention in this paper is to discuss potential FSW defects in general terms, and then address specific fractographic defect 'indications' for FS welded fatigue specimens in 5383-H321 and 5083-H321 aluminium alloys. It will examine the role of process-structure interactions in generating such indications, and their influences on crack paths, and hence on dynamic performance of the welds. This leads to some general conclusions as to the manner in which FSW process parameters might be optimised so as to minimise any detrimental influence on fatigue and fracture.

DEFECTS IN FRICTION STIR WELDS

As stated above, defect levels are generally low in FSW, compared with typical fusion welds. A number of types of defect are known to occur, however, and because they can occur in any orientation and at any angle, may be difficult to detect with directionally specific techniques such as radiography and ultrasonics [3]. The known defects in FS butt welds include lack of penetration (tool length too small for the plate thickness), voids and root defects, which are also known as 'kissing bonds'. Tool penetration is generally around 90% of the plate thickness, and can therefore lead to defective welds if, for example, plate thickness is variable along the weld line. This is essentially a process control problem that can be resolved by appropriate seam tracking devices.

Voids

Voids have been proposed to occur as consequence of the fluid dynamics associated with the plastic flow in the weld zone [4]. Numerical 3-dimensional modelling of the flow dynamics in the weld region has indicated that there is a zone on the advancing side of the weld where chaotic flow occurs. There is a location within this zone above and below which the flow is in opposite directions, creating a vortex [4]. Such vortices could lead to the generation of a series of voids in the weld zone, potentially with significant sizes. Voids do occur in FS welds, and sometimes can be seen on the surface of fatigue specimens machined from welded plates, i.e. that occurred in the interior of the original weld. Figure 3 shows such voids observed metallographically on the surface of a reversed bend fatigue specimen. The largest void is some 368 im by 279 im, and such large defects would be expected to have a very significant effect on crack initiation and/or crack paths and hence on fatigue life.



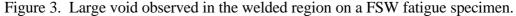


Figure 4 provides supporting evidence for this effect on fatigue life and shows the crack initiation region on the fracture surface for the specimen seen in Figure 3. This

specimen was tested at a peak surface stress of 196 MPa and returned a life of 50 723 cycles, a very much lower value than those pertaining to specimens tested at stresses either side of 196 MPa (see Table 1). Void assisted crack initiation has also been observed by the present authors at the surface of full-plate-depth fatigue specimens, and it therefore seems likely that voiding can occur at all depths in FSW joints.

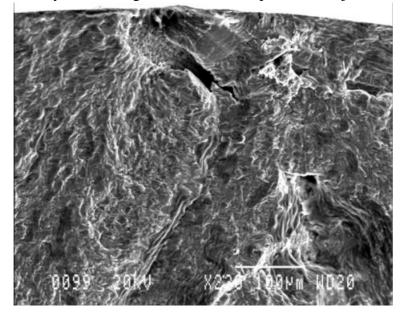


Figure 4. Void assisted crack initiation in SP FSW reversed bend fatigue specimen.

		Failure distance from
Stress amplitude	Cycle life	weld centreline
(MPa)	N_{f}	(mm)
261	15 873	5.5
240	36 332	5.5
213	192 167	0.5
196	50 723	2.5
176	185 000	4.5
173	673 509	6
169	299 784	0
136	285 024	1.5
130	1 204 153	3.5

Table 1.	Fatigue 1	life for 6	5 mm	diameter	reversed	bend I	FSW	specimens
----------	-----------	------------	------	----------	----------	--------	-----	-----------

This then leads to doubts that all voids arise from fluid dynamic processes. There is still argument in the FSW research community regarding the kinetics and dynamics of the process, and fractographic information may assist in interpretation of the underlying mechanisms. Equally, a thorough understanding of the process-microstructureperformance characteristics of FS welds will contribute to crack path analysis and the improved understanding will contribute to eventual process-performance optimisation. As an example of this, which is related to the mechanisms of void formation, consider the magnified view of the crack initiation region in Figure 4, which is given as Figure 5.

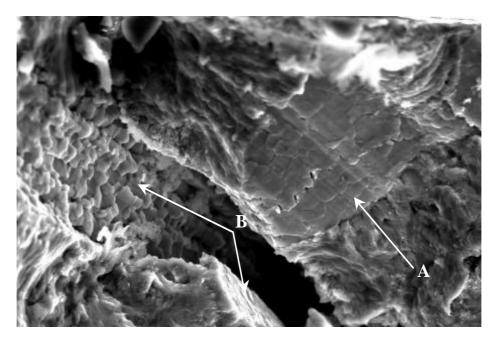


Figure 5. Fractographic evidence of dynamic recrystallisation in FSW.

Dynamic recrystallisation is still a topic of controversy amongst FSW researchers, although substantial evidence exists for continuous dynamic recrystallisation in FSW of aluminium alloys [5]. Figure 5 shows clear evidence of dynamic recrystallisation in the fine (10ì m average diameter) polygonal grains, which are marked with arrow A. Grain growth is indicated in the variety of grain sizes present. Interestingly, this mechanism of grain formation is shown up because some of these regions (arrows labelled A and B) represent crack path defects and are here associated with voids in the material. The polygonal shape of the voids seen in Figure 3 raises the question of their provenance. Voids associated with a vortex generation mechanism would be expected to be circular or elliptical in shape, and such voids are sometimes found on the fracture surfaces of FSW specimens (Figure 6). In contrast, it can be speculated that the voids seen in Figure 3 have their origin and/or shape linked in some way to the occurrence of dynamic recrystallisation, rather than vortex shedding.

The discussion, thus far, does not make clear why regions such as the one indicated with the arrow marked A, apparently form weak interfaces in the TMAZ of the weld. A hypothesis is advanced later in the paper regarding strain localisation effects in the onionskin structure that may, however, offer an explanation of some of the features associated with crack paths in both Figures 2 and 5.

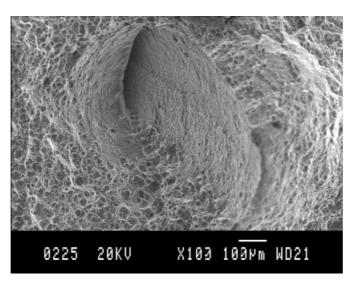


Figure 6. Elliptical void on the fracture surface of an FSW specimen.

Root Defects

Root defects or 'kissing bonds' occur when the root of a single pass weld achieves only partial bonding and their effect on fatigue strength is covered in a paper by Dickerson and Pryzdatek [6]. The occurrence of kissing bonds appears to be alloy specific and in particular, in the limited range of alloys considered, 5038-H321 is known to be more susceptible to these defects than either 5083-O or 6082-T6 alloys [6]. They are difficult to detect using either radiography or dye penetrant techniques [6] but can have an effect on fatigue performance if the root flaw is > 0.35 mm in depth.

The relative difficulty of detecting defects in FS welds makes it imperative to fully understand their influence on fatigue crack initiation and total life. It would also be advantageous to know their dependence on process parameters such as tool travel speed, rotational speed and geometry. As yet, however, there is an absence of detailed information particularly regarding internal defects, their mechanism of origin and influence on fatigue crack initiation and life. The few reported studies in this area are often preliminary in nature, which cover only limited ranges of controlling parameters [6, 7]. These studies have tended to find minimal effect of defects on the fatigue performance of FSW joints, but work by the present authors has indicated more serious consequences both of voids and of crack path defects associated with the onionskin structure. These aspects will be considered in the following sections.

INFLUENCE OF FSW DEFECTS ON CRACK PATHS AND FATIGUE PERFORMANCE

Material and Experimental Conditions

The work reported in this paper was carried out on fatigue specimens machined from FSW butt joints made between 8 mm thick plates of 5083-H321 or 5383-H321 All welds were made at TWI under empirically determined aluminium alloys. conditions that gave sound welds, at least as judged by the absence of root defects and any significant radiographic defect indications. These conditions can be summarised as using a Type 5651 tool, which had a 25 mm shoulder and a pin with a diameter of 10 mm and a length of 7.9 mm, a tool rotational speed of 500 rpm clockwise, looking at the plate, with a forwards tilt in the direction of travel of 2.5° and a heel plunge depth of 0.2 mm. Weld travel speed was kept constant for a particular weld, but varied between 60 mm/min and 200 mm/min. This variation had the aim of examining whether certain crack path defects associated with the onionskin structure increased in frequency as travel speed increased. In the event, no systematic variation in their occurrence was noticed and welds made under this range of speeds are comparable in fatigue terms. The chemical composition and mechanical property data for the two alloys are given in Table 2

Weight %	Mg	Mn	Si	Fe	Cr	Zn	Cu	Ti	Ó _{0.2%}	Ó _{TS}
									MPa	MPa
5083-H321	4.20	0.60	0.25	0.15	0.09	0.09	0.06	0.02	264	350
5383-H321	4.75	0.82	0.11	0.12	0.08	0.22	0.08	0.03	275	370

Table 2. Chemical composition and mechanical property data

Friction stir welding reduces the 0.2% proof strength in the weld region to around 160 MPa. Vickers hardness values (HVN) under a 200 gf load drop from around 105 in the parent plate to 81-85 within the TMAZ, which extends out to about 15 mm either side of the weld centre-line. These hardness values are little affected by travel speed, as indicated in Figure 7 for 5383-H321 plate welded at 80 mm/min and for 5083-H321 welded at 200 mm/min. The mean hardness value in the weld metal with the faster travel speed is perhaps 90 Vickers, while that for the 80 mm/min travel speed is around 87.5 Vickers. Local variation in hardness values can be seen in the weld zone, perhaps averaging around 5 HVN, with occasional spikes > 10 HVN. It is possible that this is related to the existence of the onionskin structure in the weld TMAZ, in line with the discussion later in the paper, and further work on interpreting TMAZ microstructures is under progress.

Several different types of fatigue load and specimen have been considered in this work. That part primarily dealing with travel speed effects used rectangular section, hourglass-shaped fatigue specimens. Gauge length of the specimens was 40 mm, width 16 mm and thickness was kept as close as possible to the original plate size of approximately 8 mm. A 100 mm radius was used to connect the gauge length to the grip sections, and the edges were slightly rounded to prevent crack initiation occurring there. S-N testing was performed in tension at 112 Hz and R = -1 (fully reversed loading). Two specimen surface conditions were used; as-welded, with small burrs at the edges of the weld region removed, but the tool shoulder ledges (~ 0.2 mm) remaining, and machined, where both burrs and ledges had been removed, leaving a smooth surface free of stress concentrations (net thickness about 7.4 mm). This was done because there was interest in both the fatigue performance of as-welded samples, representing general engineering usage, and in the inherent fatigue properties of the welding process.

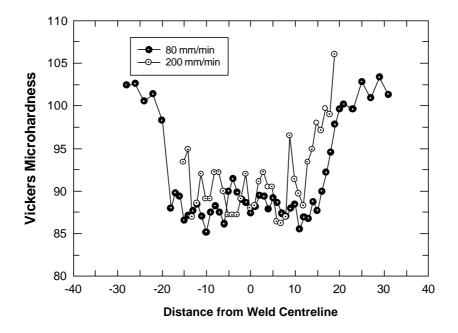


Figure 7. Microhardness variation across typical FSW joints.

Further work on the influence of defects on crack paths was performed using 6 mm diameter hourglass specimens tested at R = -1 in reversed bend. These specimens had a polished surface and therefore also bypass crack initiation from artefacts of the welding process (i.e. ledges at weld edge and tool travel marks). The gauge section in these specimens was only 8 mm and the weld and specimen centrelines were arranged to coincide. This confined crack initiation largely to the weld nugget region.

Fatigue Performance

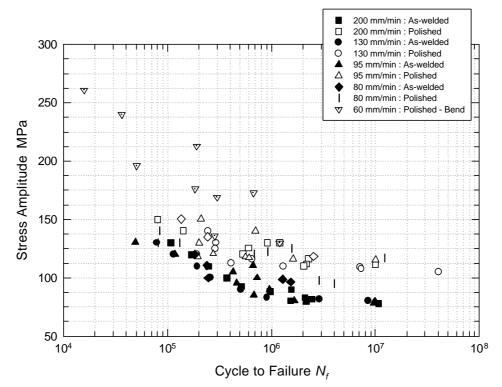


Figure 8. S-N data for reversed tension and bend fatigue is SP FSW specimens.

The reversed bend fatigue data given in Table 1 are plotted in Figure 8, together with data obtained from the tension tests. It is immediately clear from the figure hat the smaller polished tension specimens show much higher fatigue strengths than the larger Tension tests are known to give lower fatigue strengths than tension specimens. equivalent bend tests, because the whole cross-sectional area experiences peak stresses in the fatigue loading. The difference would not be expected to be this marked, however, and these data are therefore likely to reflect defect influences in the larger cross-section of the tension specimens (128 mm² compared with 28 mm² for the bend specimens). In support of this argument, the poorer performing bend specimens, where large defects influenced crack initiation, fall into the same scatter band as the polished tension specimens (tension data at stress amplitudes of 196 MPa, 136 MPa and 130 MPa). Figure 9 compares typical low magnification fractographs for reversed bend specimens in cases where defects have influenced crack initiation and crack paths, as well as where they have not had such a role. The defects shown in Figure 9a (left side of the picture with $N_f = 50~732$ cycles at $\sigma_{amplitude} = 196$ MPa) have reduced the fatigue performance of the specimen by a factor of around 2.6 on life (equivalent to a reduction in fatigue strength of perhaps 15%), compared with that of the specimen shown in Figure 9b (right side of the picture with $N_f = 185\ 000$ cycles at $\sigma_{amplitude} = 176\ MPa$).

Similar observations can be drawn regarding the tension specimens. Close analysis of the fatigue data at various travel speeds [2] demonstrated that defects could influence

fatigue performance in these specimens, and that the onionskin TMAZ structure manifested itself in at least two different effects on the crack path, namely large planar facets and banding on the fracture surface. The banding is very likely to be related to the recrystallisation structure shown in Figure 5, and these observations allow some useful conclusions to be drawn in the next section, regarding the origin of, and mechanism behind, planar regions seen on the fracture surfaces of FSW specimens. In addition, polygonal voids did occur and were occasionally observed influencing crack initiation.

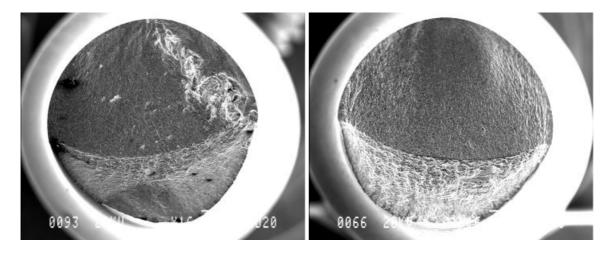


Figure 9. Low magnification fractographs of reversed bend fatigue specimens with: a) (LHS) defect-influenced crack initiation and growth; b) (RHS) initiation uninfluenced by defects.

The combined effects of voids and planar facets (which provide, for example, easy fracture paths linking two small fatigue cracks) led to reductions in fatigue strength of around 15-20%, compared with specimens where such defects did not occur. These observations indicate that the issue of defects in FS welds, their origins and effects on initiation and crack paths would clearly benefit from further attention by the research community.

Crack Path Defects

FSW crack path defects in 5083-H321 and 5383-H321 include the flat regions seen in Figure 5, which reflect details of the recrystallised grain structure and are probably related to banding observed on other fracture surfaces (see Figure 11 below). They also include much larger planar facets on the fracture surface, which occur more frequently when the crack growth rates are higher and when the cracks are larger [2]. These are crack growth conditions where crack tip plastic zones are larger, and the strain rates in the plastic zones are higher. The defects are particularly prevalent in the fast fracture region.

It seems reasonable that crack path defect behaviours in FS welds that are triggered by crack plasticity effects, reflect the mechanisms of thermo-mechanical deformation that lead to the onionskin structure observed in the TMAZ region. As not all FS welds show such a structure in the TMAZ, proving this linkage would be of benefit in identifying process optimisation routes to minimise the thermo-mechanically induced layered structure and its effects on dynamic performance.

The results summarised in this paper, and those reported in reference 2, therefore lead to the proposal that a particular class of planar crack path defects in FSW arise from the plastic flow processes involved in generating the layers in the onionskin structure. As seen in Figure 10, the large planar facets can occur in sequences reflecting the tool advance per revolution, around 0.16 mm in the case shown, and hence they occur at layer interfaces. Whilst the defects can unequivocally be shown to arise in this layered structure, the mechanism behind them is more difficult to identify. Until recently, information on flow processes in FSW has been lacking in the open literature. Thus the large planar fracture surface facets reported by James et al in reference 2 were identified in that paper as 'partial-forging' defects. There may well be some truth in

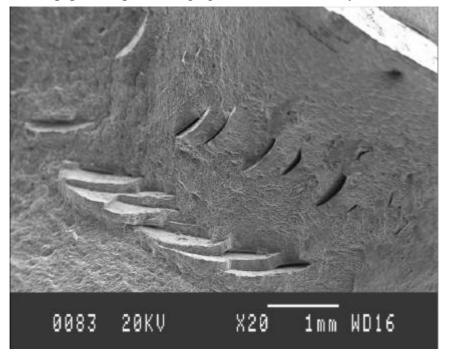


Figure 10. Planar facets on the fast fracture region of a tension fatigue specimen.

that descriptor, as the pressure and temperature conditions may vary sufficiently in parts of an FS weld to lead to such partial bonds between deposited layers.

However, a recent paper by Guerra et al [8] has provided a description of the formation of the onionskin layers, which provides a framework to explain a number of important observations related to the microstructure in the TMAZ of an FS weld and their dynamic performance. Reference 8 indicates that the flow of metal during FSW occurs by two main processes. The first involves 'wiping' of material from the

advancing front side of the tool onto a zone of metal that rotates and advances with the tool. The material undergoes a helical motion within the rotational zone. After one or more rotations, this zone of metal is sloughed off in the wake of the tool, primarily on the advancing side. The second process is an entrainment of material from the front retreating side of the tool that 'fills in' between the sloughed off pieces. In essence, as proposed by these authors [8], the metal in the FSW TMAZ consists of two streams of material with different thermo-mechanical histories and mechanical properties. These constitute the layers in the onionskin structure.

This process explains why the layers etch differently, as the different thermomechanical histories would lead to different dislocation densities and distributions. It would also be expected that adjacent layers would show different strain hardening exponents and microhardness values. This would lead to scatter in microhardness values in the TMAZ and, more importantly, could also lead to strain-partitioning effects occurring during deformation processes. Results on strain measurements during tensile testing, presented by Reynolds at an international workshop [9], clearly indicated that such strain-partitioning did occur between adjacent layers in the TMAZ structure.

Any propensity towards strain-partitioning would be exacerbated by high strain rates during deformation, such as would occur as fatigue crack velocities increase, or under fast fracture. Strain-partitioning mechanisms are often associated with the occurrence of ductility-related cracking problems; two well-known examples are strain-age embrittlement and reheat cracking. It is proposed that the planar defects observed in this work on the fracture surfaces of the specimens represent the operation of a strainpartitioning induced ductility drop at layer interfaces, possibly sometimes coupled with partial forging during welding. Activation of such a mechanism during fatigue or fracture would depend, amongst other things, on the crack orientation with respect to the interface, the strain rate and the relative differences in mechanical properties and strain hardening exponent of adjacent layers in the onionskin structure.

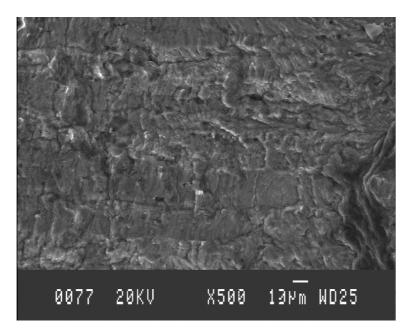


Figure 11. Banding texture on a FSW fracture surface near the crack initiation site.

An effect of orientation of the layers, relative to crack path can be illustrated by comparing Figure 5 with Figure 11. The latter figure shows banded texture on the fracture surface near the crack initiation site in a tensile fatigue specimen. The width of these bands is around 10-20 i m, much smaller than the tool advance per revolution, and more on a scale with the recrystallised grain size. The details of the structure provide an indication that if the crack had crossed these layers at a glancing angle, their appearance might then mirror that of the flat region marked A in Figure 5. The significance of this structure is not yet clear.

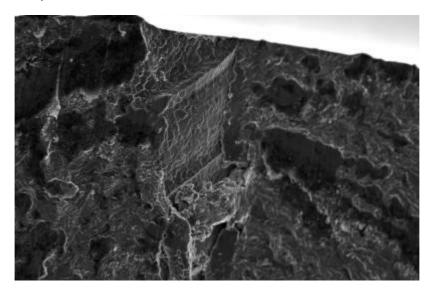


Figure 12. Layer interface linking two small fatigue cracks.

Figure 12 shows similar fracture surface marks to those seen in Figure 5, but here directly associated with crack initiation and occurring at an intermediate angle to the surface. This is a particularly interesting example, because the layer interface defect is acting as a path linking two smaller fatigue cracks near their initiation sites, and because its shape is reminiscent of a threaded helical profile. The sharp protrusions have an apparent spacing of around 0.1 mm. This specimen was tested in reversed bend with $\sigma_{amplitude} = 130$ MPa, giving a life of 1 204 153 cycles and, as seen in Figure 8, has a low fatigue performance.

CONCLUSIONS

This paper has presented some observations and discussion centred around the interaction between the dynamics of the friction stir welding process, its influence on crack path defects and their significance for fatigue and fracture performance. In particular, an explanation for the origins of planar facets on the fracture surface and their effect on fatigue life has been proposed. This explanation draws on the work of other research groups [8, 9] regarding the plastic flow processes that occur during FSW

and the strain-partitioning during loading that occurs in adjacent layers of the TMAZ that have different thermo-mechanical histories.

The main conclusions can be summarised as:

1. Planar facets occur on the fracture surfaces of FSW welds with sizes in a range from 0.1-1.5 mm.

2. These facets arise from an interface ductility drop induced by strain-partitioning that occurs in adjacent layers in the TMAZ during loading. Their occurrence is enhanced by higher strain rates, but they also occur near crack initiation sites.

3. These interfacial strain-partitioning defects can reduce fatigue strength of FSW specimens by up to 20%.

4. The study of crack path subtleties through fractography forms an indispensable part of the overall analysis of fatigue crack paths.

If strain-partitioning defects arise from the thermo-mechanical history of the weld, optimisation of the FSW process clearly requires some analysis of the force footprint, torque and temperature experienced by the tool during welding. On-line monitoring of these parameters, and their relationship to weld defects and performance is the subject of on-going work by the present authors.

REFERENCES

1. Pook, L.P. (2002) Crack Paths, WIT Press, Southampton.

- 2. James, M.N., Hattingh, D.G. and Bradley, G.R. (2003) in press Int. J. Fatigue 25.
- Lamarre, A. and Moles, M. (2000) Ultrasound phased array inspection technology for the evaluation of friction stir welds, In: *Procs 15th World Conf. on Nondestructive Testing*, Rome, Italy, October 2000. http://www.ndt.net/article/wcndt00/index.htm
- 4. Bendzsak, G.J., North, T.H. and Smith, C.B. (2000) In: *Procs* 2nd Friction Stir Welding Symp., Gothenburg, Sweden, June 2000, TWI Cambridge.
- 5. Lienert, T.J., Stellwag, W.L., Grimmett, B.B. and Warke, R.W. (2003) *Welding Journal*, January 2003, 1S-9S.
- 6. Dickerson, T.L. and Przydatek, J. (2003) in press Int. J. Fatigue 25.
- 7. Dalle Donne, C., Biallas, G., Ghidini, T. and Raimbeaux, G. (2000) In: *Procs* 2nd *Friction Stir Welding Symp.*, Gothenburg, Sweden, June 2000, TWI Cambridge.
- 8. Guerra, M., Schmidt, C., McClure, J.C., Murr, L.E. and Nunes, A.C. (2003) *Mats Characterisation*, **49**, 95-101.
- 9. Reynolds, A.P. (2003) presentation at *Int. Workshop on FSW*, Port Elizabeth, April 2003.