The Influence of Grain Size on Brittle Crack Propagation

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Abstract

Ferritic steels undergo a ductile to brittle transition as the temperature is progressively reduced. The strain annealing method has been developed to produce a microstructure in EN1A ferritic steel that contains a transition from a coarser grain size, \( \sim 40 \mu m \), to a finer grain size, \( \sim 20 \mu m \). Small pre-notched bend geometry specimens have been tested at \(-196^\circ C\) to investigate crack propagation from the coarser to finer grain region and vice-versa. Details of the fracture path have been investigated using a combination of high resolution scanning electron microscopy, electron back scattered diffraction and ion milling and imaging. The results are considered with respect to the transgranular and intergranular modes observed.

Introduction

The fracture of a solid arises from the formation of new surfaces in a body created by thermodynamically irreversible processes. The failure mechanisms including crack initiation and propagation may change with temperature. For example, polycrystalline ferritic steels are ductile at room temperature but when temperature is reduced they undergo brittle failure. The ferritic steels fail by transgranular cleavage at low temperatures on \{100\} planes, but encounter obstacles such as grain boundaries or sub-boundaries as the crack propagates. Hence there is a need to accommodate grain orientation mismatch as the cleavage crack propagates from one grain to another [1]. Crack arrest at the grain boundaries has been studied by various workers [2]. The recent advancement in high resolution electron and ion beam techniques has allowed the local grain boundary failures as well as general fractographic details to be considered in more detail. In this paper, brittle crack propagation from a coarser to finer and a finer to coarser grain size in ferritic steel is analysed.

Crack Plane Determination

Two surface analysis is a recognised technique for determining the transgranular fracture planes in polycrystalline materials. As described by Davies and Randle [3] this is usually achieved by providing two macro surfaces at right angles to intersect the fracture plane to be evaluated. However, as the grain size decreases it becomes increasingly difficult to maintain the accuracy of the determination using this method of preparation. Moreover, to select the specific feature to be measured often requires significant destruction of the specimen containing the fracture surface. As shown by Hughes et al. [4] focussed ion beam milling provides a tool for interrogating 3-D crack morphology. When combined with electron backscattered diffraction, EBSD, it becomes a powerful tool for undertaking two surface analyses of fracture planes. A difficulty is that the surface to be oriented by EBSD has to be inclined at an angle of 20° to the incident electron beam, 70° to the horizontal, and there has to be an unimpeded path for the diffracted beam to reach the detector. Two approaches can be adopted to achieve these requirements: (i) FIB milling to introduce two surfaces intersecting the crack surface at 90° at a position on a pre-cut edge of the specimen, Figure 1(a) and (ii) Initially FIB milling a single trench parallel to the incident ion beam, Figure 1 (b), stage (i), and then rotating the specimen, Figure 1(b), stage (ii), through 45° and creating a new surface, surface 3, that intersects the fracture surface. The specimen is then rotated by 25° as shown in Figure 1(b), stage (iii) to provide a free path to the detector for the diffracted beam. Hence a pre-selected feature can be evaluated at any position on a fracture surface.
Experimental

A section of a 16 mm square EN1A ferritic steel bar was heat treated at the temperature of 920°C for 1h and air cooled to room temperature. The chemical analysis, (wt%) for this EN1A steel gives 0.06% C, 0.30% Si, 0.78% Mn, 0.015% P, 0.02% Ni, 0.01% Mo, 0.05% S as main elements plus Fe. These, specimens were compressed using a Zwick tensometer to a strain level of 2.2% and annealed at 690°C for 3hours. As a result of greater near surface strains, this heat treatment resulted in layered grain size microstructure which is used to propagate cracks from coarser to finer grains and vice-versa; Coarser grain size 40 µm and finer grain size 20 µm (mean linear intercept).

An unique three point bend test has been developed to study the crack propagation in this ferritic steel. Specimens with dimensions of 20 mm × 5 mm × 2.7 mm were extracted from the bar by electric discharge machining (EDM) after the heat treatment. The notches were cut to a depth of ~ one third the width (a/w = 1/3) by electric discharging machining with a wire of diameter 0.1 mm. At a temperature of -196°C, the specimens were loaded with a load rate of 3Ns⁻¹ and three repeated tests were undertaken. Load - displacement was acquired using the embedded software in the system. Fracture surfaces of the failed specimens were examined using the SEM (Hitachi S-2300) at an operating voltage of 25 kV. The high resolution FEI Dual Beam (Helios) work station was used for milling and imaging; a high beam current (11 500pA) to mill and a low beam current (70 pA) to polish specimens. Cross-sectioning, as described in the above section was used to establish the cleavage planes using two surface analyses to provide unique solutions for the orientation of the fracture facets and cleavage planes.

Results

The load-displacement characteristic for the finer to coarser microstructure is shown in Figure 2a. The curve shows two prominent regions before the final fracture. The similar behaviour was observed for all the samples tested. The load-displacement curves for coarser to finer microstructure showed three discrete regions before the final fracture, Figure 2b. There is linearity up to ~180 N and thereafter an increase in energy corresponding to crack arrest and then a sudden drop in load before proceeding to the final fracture. The work of fracture is calculated from the area under the curve, Table 1.

Figure 3a shows the low magnification image of the fracture surface obtained at -196 °C for the coarser to finer region microstructure. The corresponding fracture features seen from the notch root are illustrated schematically, Figure 3b. From the notch root to ~ 0.2 mm, in the coarse grain region the cleavage planes are observed, Figure 3c - region X. At this point there is a crack arrest at the finer grain region consistent with the load-displacement curve and this is a region, Y, of intergranular fracture. Figure 3d reveals a cleavage plane meeting at an interface (region Y) where the intergranular failure dominates. The crack then propagates in a cleavage mode until the final failure, Figure 3e-region Z. The crack propagation from finer to coarser grain show continuous cleavage fracture.

FIB cross-sections were milled at the edge of the specimen for cleavage plane evaluation in the coarser grain region and two surface analysis was undertaken as Figure 1 (a). The surfaces were carefully milled using a low beam current to obtain clear bright EBSD patterns. The pole figure for surface 1 is shown in Figure 4(a). The two milled surfaces lie close to [001] and the cleavage plane was established to be [001]. Figure 4(b) shows the EBSD pattern obtained for the finer grain region using the general preparation method. Figure 1(b). Here a cut was made on the surface at 45°, surface 3, to achieve stage (ii), and then the specimen was transferred to the SEM and aligned as shown in stage (iii). In this way clear EBSD patterns were obtained and the main pattern showing the [100] normal, Figure 4(b). The subsequent two surface analysis showed that the cleavage facets in this finer grain region were [001].

Concluding Comment

Two preparation methods for two surface analyses have been adopted which provide an accurate evaluation for fracture surfaces at smaller grain sizes than the macro-sectioning method as described by, for example, by Davies and Randle [3]. Although more convenient it is limited since macro-sections have to be prepared close to the fracture surface feature to be analysed. The new method described in Figure 1(b) provides a general approach. In particular a fracture facet of interest can be selected at any position.
on the surface and by adopting the more complex FIB milling sequence described in Figure 1(b) it is possible to undertake a two surface analyses to provide a unique solution. Indeed this can be applied to fracture facets over a wide range of sizes from about one micrometer up to tens of micrometers. In the present case for both the coarser and finer grain size regions the cleavage fracture is \{100\}.

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Table 1 Average values for work of fracture (Nmm) calculated from area under load-displacement curves

| Work of fracture (Nmm) | Fine to coarse | Coarse to fine |
|------------------------|---------------|---------------|
| Total                  | 91±10         | 106±10        |
| Coarser region         | 80            | 21            |
| Fine region            | 11            | 85            |

Figure 1 Schematic drawing showing the FIB milled surfaces for EBSD analyses; (a) surface, created at the prior edge of the specimen, (b) the general method, stage (i) surfaces created within the specimen at 90° to each other, stage (ii) showing inclined angle surface 3 prepared at 45° and stage (iii) subsequent rotation to allow EBSD detection.
Figure 2 Load-displacement characteristics of three point bend tests at -196 °C (a) finer to coarser grain structure (b) coarser to finer grain structure

Figure 3 Fractography of coarser to finer grain structure; (a) low magnification SEM image from the notch root, (b) schematic drawing of the microstructural regions from the notch root, (c) transgranular cleavage at region X, (d) intergranular fracture at region Y, and (e) transgranular cleavage at region Z

Figure 4 (a) An Inverse pole figures for the surface1, Figure 1(a); (b) electron back scattered pattern of the surface 3, Figure 1(b) shown [001] normal