CRYSTALLOGRAPHIC ANALYSIS OF SURFACES AFTER BRITTLE FRACTURE IN FERRITIC STEELS

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ABSTRACT

Recently considerable advances have been made in devising robust experimental methodologies for crystallographic analysis of fracture surfaces, and applying them to materials such as steels. There are two main practical thrusts: precision sectioning through the polished side of a specimen, perpendicular to the average fracture surface, and quantitative photogrammetry directly from the fracture surface. The second of these approaches is particularly novel since it allows electron back-scatter diffraction (EBSD) data to be obtained concurrently with the spatial coordinates, both in a scanning electron microscope. In this paper examples of some preliminary investigations in which these procedures have been used to produce valuable results will be described.

KEYWORDS

Electron back-scatter diffraction, quantitative fractography, fracture facet, stereo-photogrammetry.

INTRODUCTION

Brittle fracture in ferritic steels can occur intergranularly, especially in the presence of embrittling species such as phosphorus, or transgranularly on cleavage planes of low surface energy in the lattice. Unambiguous identification of which of these two processes is operating, and detailed analysis of the characteristics of the fracture phenomenon, relies crucially on knowledge of the local crystallography. There are two primary and separate requirements for the experimental measurement of facet crystallography on the fracture surface of polycrystalline materials [1]:

- the *positional coordinates* of a fracture facet in space
- the *crystallographic orientation* of the facet

both measured relative to the *same* reference axes. Electron back-scatter diffraction (EBSD) in a scanning electron microscope (SEM) is the forefront technique to provide crystallographic information [2], although there are several challenges regarding its accurate application to fracture surfaces. Equally, obtaining precise spatial coordinates for the positional orientation of a facet is taxing.

Recently considerable advances have been made in devising robust experimental methodologies in this area and applying them to real materials, namely steels. There are two main practical thrusts: precision sectioning through the polished side of a specimen, perpendicular to the average fracture surface, and quantitative photogrammetry directly from the fracture surface. The second of these approaches is particularly novel since it allows EBSD data to be obtained concurrently with the spatial coordinates. In this paper examples of some preliminary investigations in which these procedures have been used to produce valuable results will be described.

EXPERIMENTAL METHODOLOGIES

Essentially the experimental strategies divide into two approaches: some or all of the measurements are taken *directly* from the fracture surface, or some or all of the measurements are taken from a surface adjacent to the fracture surface, i.e. *indirectly*.

The crystallographic orientations of facets - or any other planar surface such as some internal interfaces - may be determined by a totally indirect approach. Orientations are obtained by EBSD from a polished section perpendicular to the overall fracture surface, coupled with fracture surface profile analysis from optical or SEM images of at least two serial sections through a plane perpendicular to the fracture. This information defines the crystallographic facet orientation. The procedures and applications are discussed in detail elsewhere [3]. Indirect crystallographic analysis of facets in this manner from serial sections has the distinct advantage that the orientation data are straightforward to obtain by EBSD, because they are taken from a flat polished section. Drawbacks are that it is an inherently destructive technique, it is restricted to materials with average grain size greater than approximately 100μm, and the procedures are very labour-intensive.

A variant of the indirect approach is to obtain data from a single polished section only. Useful information can be gleaned by performing EBSD on a 'matched fracture' specimen. Here the specimen is fractured into two halves, then the two halves are realigned, mounted together as they were prior to fracture, and polished as if they were a single specimen. The alignment and mounting procedure requires considerable care to maintain the specimen geometry. The merit of this procedure is that intergranular and transgranular fracture surfaces can be immediately identified from evidence of colour matching across the fractured interface in the crystal orientation map. Subsequently, proportions of intergranular and transgranular (cleavage) fracture can be quantified. Another piece of information which can be obtained from a single section is the crystallographic trace vector which defines the fracture edge. This direction must lie in the fracture plane, and so can be used to test the probability that, for bcc steel, the plane is a {001} cleavage plane.

In contrast to the indirect techniques, direct techniques have the advantage of being non-destructive although they still generally require accurate correlation between the crystallographic and macroscopic orientation of the facet. Probably the first experiment which combined photogrammetry and EBSD was employed by Slavik and coworkers [4] to determine the fracture facet crystallography using quantitative tilt fractography of an Al-Li-Cu (AA2090) alloy. The grain orientation was measured by EBSD from a polished surface, perpendicular to the average fracture plane, and combined with the facet orientation, which was aligned such that the co-ordinate system was identical for the tilt fractography and the EBSD analysis. A series of SEM fractographs (i.e. images of the fracture surface) were acquired at different tilts and a series of measurements made of projected lengths between features on the fracture surface. The data were then combined to determine the fracture facet crystallography.

An extension of photogrammetry is stereo-photogrammetry, where images of a rough surface, taken at least two different tilt angles, are combined to produce a three-dimensional (3D) reconstruction of the surface. In the last few years computer assisted stereo-photogrammetry, in real time in an SEM, has greatly expanded the potential of this technique to facet analysis, and there have been some pilot experiments to couple stereo-photogrammetry with EBSD [5,6] and, recently, those reported in this paper.

EXAMPLES OF CRYSTALLOGRAPHIC ANALYSIS OF SURFACES

Here we will present an example of both the indirect and direct techniques for facet analysis which have recently been performed on two steels as part of ongoing investigations..

Samples of an alloy having composition Fe-0.06wt%P-0.002wt%C were cut to 5mm × 5mm × 30mm and a notch of ¼-thickness depth was cut into one of the faces. The sample was held under liquid nitrogen and fractured by an impact on the face opposite the notched face. After fracture the two halves were realigned and mounted. An EBSD map was then obtained from the fractured region as if it were still a single specimen. Figure 1 shows an example of a 'matched fracture' specimen. In this case the crystallographic orientation map has been superimposed on the secondary electron image. It can be seen that for much of the map the colours match across the fracture surface, indicating cleavage fracture. There are also some cases where the colours do not match across the fracture surface, indicating an intergranular, accommodation fracture facet. As the ageing temperature increased so the proportion of facets on the brittle fracture surface showing colour match across the fracture (i.e. cleavage facets) increased.

Measurements directly from the fracture surface will be illustrated here by reference to recent investigations on a C-Mn alloy which had fractured predominantly in the transgranular mode. A previous investigation, using single crystals, into the accuracy of EBSD measurements obtained directly from fracture surfaces had revealed a large associated error, >25°. This is because although EBSD is calibrated for parallelism between the camera screen and the specimen surface (an individual fracture facet in this case), a large deviation away from the parallel condition can be tolerated before the diffraction pattern becomes unindexable by the software. This difficulty can be overcome somewhat by searching for 'local minima', but this technique is tedious and only suitable for small sample populations [6].

A more promising approach to direct crystallographic analysis of fracture surfaces than EBSD alone is computer-assisted stereo-photogrammetry, which was carried out in the present ongoing research using 'Stereo Facet' software, commercially available from Oxford Instruments. Validation of the stereo-photogrammetry procedure was achieved by a series of tests on a surface feature of known geometry, namely a Vickers hardness indent where the angle between the faces of the pyramidal indent is 136°. Stereo images were acquired separately, tilted at +5° and -5° with respect to the primary beam direction. The parallax shift data for each pixel is calculated using a digital-image correlation analysis routine provided by the software, which was subsequently used to construct a '3D-elevation model' of the indent.

Having validated the stereo-photogrammetry procedure, it was then used to determine the positional orientation of fracture planes in space and correlate the measurement with the crystallographic orientation obtained by EBSD. A 3D-elevation model of the fractured facet was produced using the stereo images and measurements of the cleavage plane orientation were then correlated with the crystallographic information determined using the automated EBSD application of Crystal Orientation Mapping (COM). The region of fracture surface, which is shown in figure 2a, was used to produce an 'anaglyph', i.e. a 3D visual representation of the surface that combines two stereo-images. A selected region of fracture surface was then successfully modelled using the stereo-photogrammetry software to produced a 3D-elevation model of the selected area enclosed by the rectangle on figure 2a (figure 2b) and a line profile of surface heights was acquired along the line bisecting the rectangle (figure 2c). Transferring the EBSD-measured crystal axes to the 3D-elevation model, where the reference plane is positioned perpendicular the normal direction, the deviation from the exact [001] direction correlates with the positional co-ordinates of the plane, indicating that the actual crystallographic orientation of the cleavage facet is exactly [001].

The principal advantage of direct techniques for crystallographic analysis of fracture surfaces is that the fracture surface remains intact, and so is available for further investigation. [7]. Computer assisted stereo-photogrammetry can yield accurate crystallographic data which, combined with its powerful imaging and visualisation capabilities, proves it to be a powerful technique for fracture surface analysis. However, only certain facets, where the image contrast is excellent, are candidates for analysis and the analysis procedure is quite lengthy.

CONCLUSIONS

There are several approaches to crystallographic analysis of fracture surfaces. These are:

- Indirect techniques: 'matched fracture' specimens where a fractured specimen was reassembled and EBSD performed across the fracture surface; serial sectioning; a single-surface section to obtain the trace vector of the fracture edge.
- Direct techniques: EBSD from the untreated fracture surface itself; computer-assisted stereo-photogrammetry combined with EBSD of the fracture surface.

There are merits and drawbacks for both the direct and indirect methods. A common feature is that both methods for crystallographic fracture analysis are experimentally difficult and challenging, although they are worthwhile to pursue because they yield valuable information about the fracture process.

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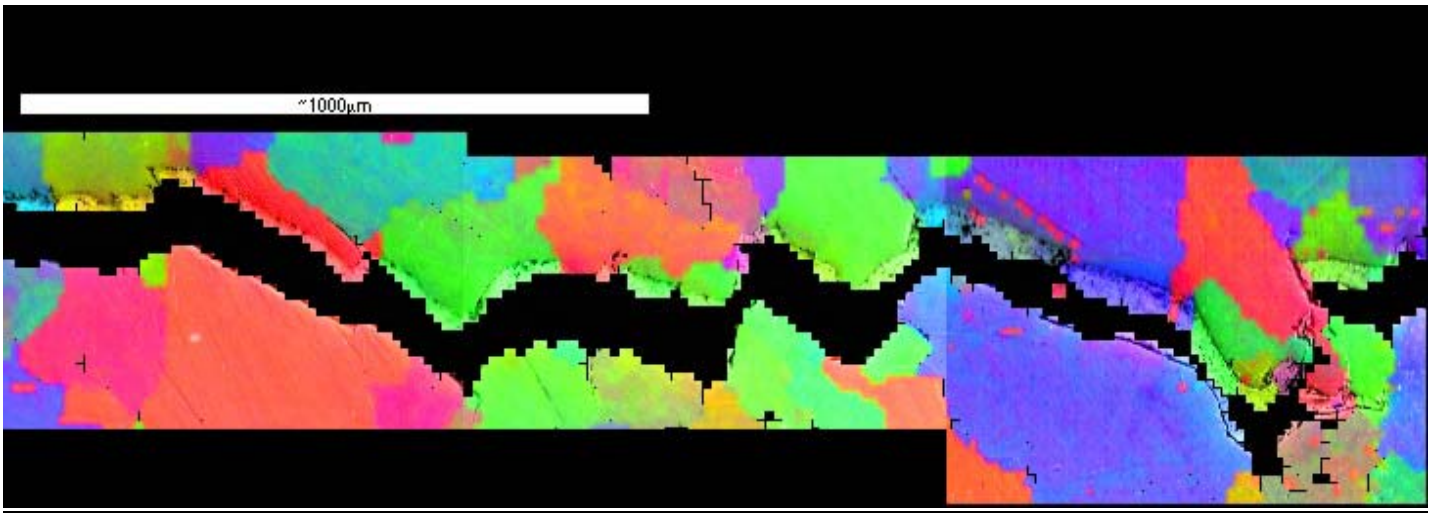
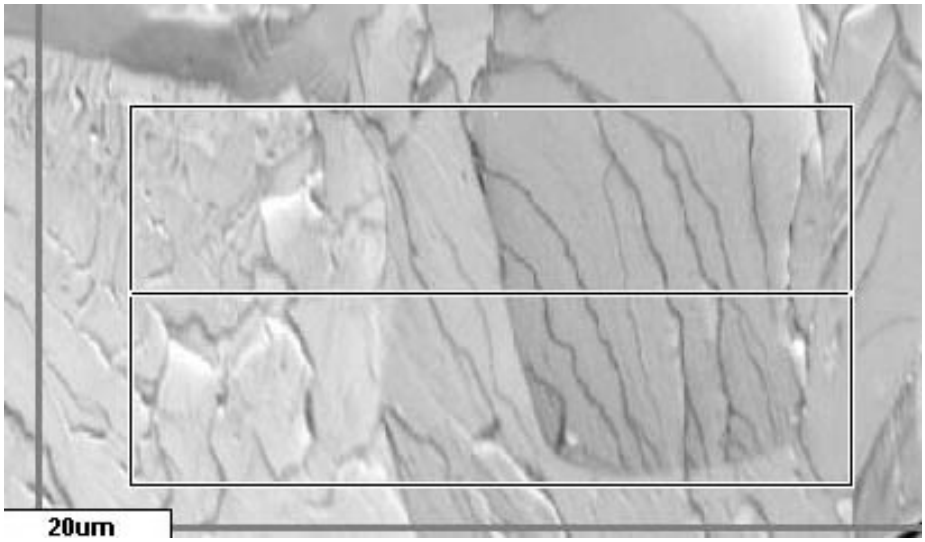
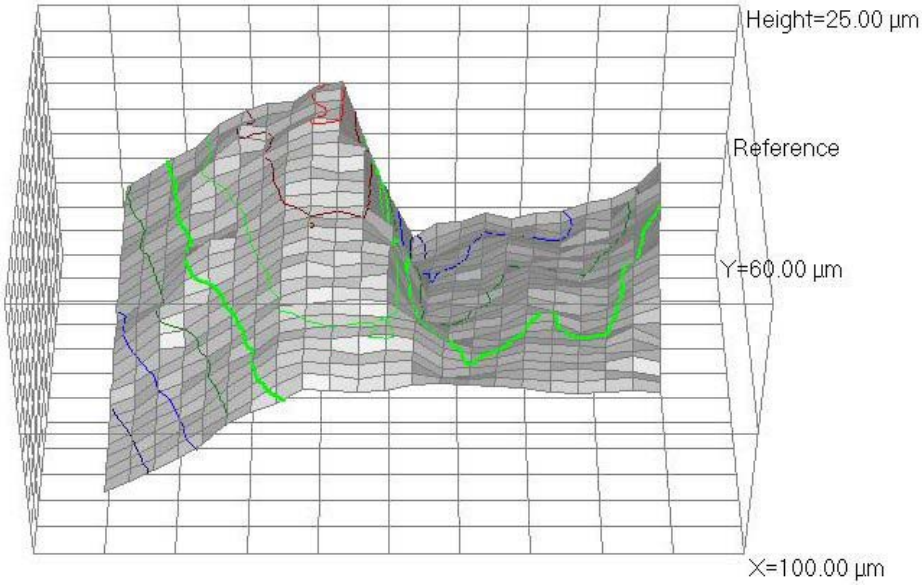


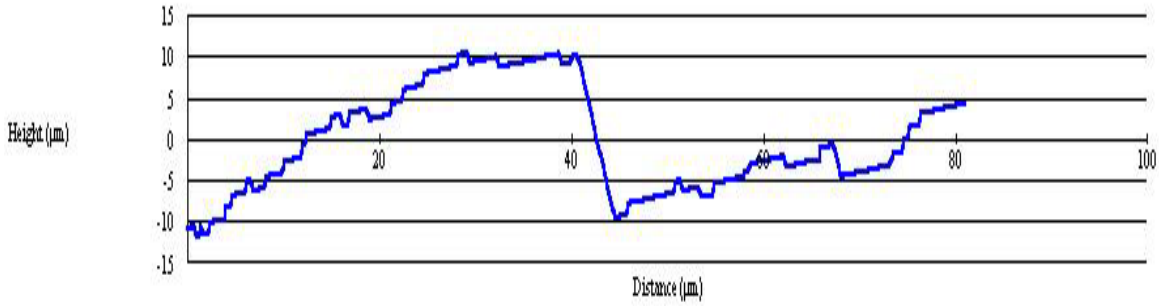
Figure 1 A 'matched fracture' specimen from an Fe-P-C alloy. The colours correspond to crystallographic orientation and are superimposed on a secondary electron image.



(a)



(b)



(c)

Figure 2 (a) Micrograph of a region on a fracture surface of a C-Mn steel. The region enclosed by the rectangle was selected to produce a 3-D elevation model and a horizontal line, bisecting the rectangle, was selected to produce a height profile. (b) 3-D elevation model of the region shown in (a). (c) Height profile along the line shown in (a).