AN INVESTIGATION OF THE PROPAGATION OF BRITTLE FRACTURE IN POLYCRYSTALLINE ZINC

G.M. Hughes[1], G.E.Smith[2], P.E.J.Flewitt[1] and A.G.Crocker[2]
[1] Interface Analysis Centre, University of Bristol, Bristol, BS2 8BS, UK
[2] Department of Physics, University of Surrey, Guildford, GU2 7XH, UK

ABSTRACT

The initiation and growth of brittle cleavage fracture in hcp polycrystalline materials is considered. In this study polycrystalline zinc was fractured at a temperature of 77K. High resolution scanning electron microscopy has been carried out to characterise fracture modes and propagation of cleavage. We also describe the novel use of focussed ion beam techniques for analysis of fracture surfaces. These techniques provide a powerful means of investigating the path of cleavage cracks. Cleavage in the polycrystalline zinc is observed on the basal (0001) plane, and also on {11-20} planes. Additionally, the mechanisms for propagation of cleavage from grain to grain are considered. Twins are observed to accompany the overall deformation and fracture of the zinc. The experimental results are compared with the predictions of crack initiation and propagation in polycrystalline hcp materials based upon a previously described geometrical model.

1 INTRODUCTION

Two and three dimensional geometrical models have been developed to describe fracture in brittle polycrystalline metals and alloys (Crocker et al.[1]). One application of these models has been to describe the initiation and propagation of brittle fracture in bcc materials such as α-iron (Smith et al. [2]). This model takes into account accommodation at grain boundaries as the cleavage crack propagates from one grain to the next. Three possible accommodation mechanisms have been identified: brittle intergranular fracture, ductile intergranular fracture and multiple stepped cleavage. (Crocker et al [1],Qiao and Argon [3]) Cleavage within the individual grains is on {100} planes, so there are three possible planes per grain. In the case of hcp metals and alloys, cleavage has been recognised to occur in single crystals on the (0001) basal plane (Schmid and Boas[4]). Therefore, in the case of polycrystalline zinc, there is only one cleavage plane per grain and this implies there is need for significant accommodation when a crack propagates from one grain to the next. In the case of polycrystalline hcp zinc, Greenwood and Quarrell [5] examined the tensile brittle fracture of zinc at 77K and also the variation of fracture energy with various microstructural parameters such as grain size. However, the detailed fractography was limited. Nevertheless, it is recognised that the brittle fracture of zinc is accompanied by deformation processes including deformation twinning on {10-12} planes, kinking and limited slip (Moore [6], Pratt and Pugh[7]).

In this paper, we examine the fracture of polycrystalline zinc at a temperature of 77K using novel focussed ion beam techniques and scanning electron microscopy combined with electron backscattered diffraction. In addition, geometrical modelling is used to predict the initiation and propagation of cleavage failure in a polycrystalline hcp material. The experimental results are discussed with respect to the predictions of the geometrical modelling.

2 GEOMETRICAL MODELLING

In order to obtain an enhanced understanding of the mechanisms associated with brittle fracture processes in polycrystalline zinc, a preliminary computer simulation investigation has been carried out. The results presented in this paper have been obtained using a two-dimensional (2-d) model containing 500 grains (Smith et al. [2]). This has been generated by placing grain nuclei randomly within the defined model space. The grains grow outwards from these nuclei until they meet neighbouring grains in straight lines (flat faces in 3-d). The crystallographic orientation of each grain is then assigned randomly. Physical characteristics that are relevant to the simulation are allocated using a graphical interface. These include relative fracture energies for different fracture modes, the number of cleavage planes available in each grain and the stress direction. Fracture is initiated at a weak point and tracked across the model by choosing the most favoured mechanism at each decision point.

3 EXPERIMENTAL METHODS

Specimens taken from 3mm thick sheet zinc were heat treated at 453K for 2hrs (HT1) and at 353K for 1hr (HT2), allowing the formation of large grains with little residual stress. After heat treatment, the samples were cut to a size of 5x20x3mm and notch-fractured in 3 point bend under liquid nitrogen parallel to the short axis.

Fractographic examination was performed on the specimens using a Philips 535 scanning electron microscope fitted with an electron backscattered diffraction system (TSL), which was used to orient the cleavage planes.

An FEI Strata 201 focussed ion beam (FIB) workstation was used for high resolution fractography. The images were obtained from electrons released as a focussed beam of Ga^+ ions is rastered across a surface, giving spatial resolutions of ~7nm. In addition to imaging, the ion beam can be used for milling a pit into the surface (~10 μm deep), which can then be finished using the beam to polish one edge of the etch pit, which can then be imaged. When the specimen is tilted to 45° to the incident beam, imaging of the cross-section reveals sub-surface features.

Modern instrumentation using charged particle beams can provide crystallographic contrast through channelling effects(Goldstein and Yakowitz[8]); variations in crystal orientation are shown by a change in contrast. Electron channelling produces a certain level of crystallographic contrast. However, it is one of the weakest contrast effects. The complex interactions at the specimen surface give rise to a more pronounced contrast, resulting in a large effect on ion induced electron channelling contrast. Once hampered by low spatial resolution, focussed ion beam based instruments were used mainly for mass spectrometry applications. Modern developments have given the instruments resolution comparable to scanning electron microscopes.

4 RESULTS

4.1 Geometric Modelling

A preliminary set of computer simulation results was obtained using the 2-d model assuming that only two brittle fracture mechanisms were available. These were cleavage on a single crystallographic line (plane in 3-d), corresponding to the (0001) basal plane in zinc, and grain boundary fracture. The fracture energy Eg of grain boundaries was allocated randomly between limits of 0.75 and 1.25 of the cleavage energy Ec. Eight simulations were carried out using two crack initiation points, two sets of crystallographic orientations and two stress directions. The results showed that on average about 70% of the fracture was cleavage and 30% grain boundary. However, there was a wide scatter arising from the individual computer runs giving cleavage ranging from 46% to 93%. Also, because of the very limited number of fracture options allowed at each decision point (usually one cleavage plane and one grain boundary) some of the selected fracture facets had rather unfavourable stress factors. This suggested that additional brittle fracture mechanisms would occur in practice and the experimental results have indeed demonstrated that this is the case, see section 4.2.

A second of set eight simulations has therefore been carried out assuming that in each grain of the 2-d polycrystalline model there are two orthogonal cleavage planes with equal fracture energies. One of these corresponds to the basal plane in 3-d zinc and the other to a combination of three variants of {11-20} prismatic cleavage. It is assumed that these have three times the fracture energy of (0001) cleavage, and that the two factors of three cancel. The new results predict that on average about 85% of the fracture is cleavage and 15% grain boundary, the scatter being quite small, cleavage ranging from 80% to 90%. Because, in the model, the two cleavage systems are equivalent, the results predict equal contributions to the overall fracture surface from basal cleavage and prismatic cleavage.

4.2 Electron Backscattered Diffraction

Secondary electron images in the scanning electron microscope showed the fracture surface to be made up of a complex arrangement of predominantly cleavage facets. Electron backscattered diffraction identified the major facets to be cleavage on the (0001) planes. The images were similar to those presented with more detail in section 4.3 using focussed ion beam imaging.

There is a step between subsequent cleavage facets. In spite of difficulties arising from sample topography, electron backscatter diffraction showed that the orientation across this stepped cleavage is constant; the patterns show the same crystallographic orientation, and the same geometry relative to the microscope reference.

4.3 Focussed Ion Beam Imaging and Cross Sectioning

Figure 1 shows a typical fracture surface, with the (0001) cleavage facets in the plane of the image. The insets show the presence of grain boundary failure (top and middle) and the geometry of a second set of cleavage planes. The prismatic nature of the facets suggests {11-20} planes at 90º to the (0001) planes. The insets show grain intergranular boundary failure. Such regions account for a small percentage of the area of the fracture surface.

Figure 2 shows twins imaged at a (0001) cleavage fracture surface, which are visible as a consequence of ion channelling contrast. It is clear that this imaging mode provides a powerful means of combining high-resolution fractographic images with the underlying microstructure. In addition, figure 2 shows a secondary crack propagating along one of the sub-boundaries. These boundaries lie in directions consistent with {10-12} planes, on which deformation twins form in zinc. Hence, the secondary crack is propagating along the twin boundary. As the crack meets further twin interfaces, it follows a zig-zag pattern, as it propagates along the twin boundaries.

A cubic hole of ~8µm across was milled using the ion beam to obtain a cross section of the crack in order to investigate the sub-surface region. The angle between the crack and the basal plane was approximately 20º, and the crack was found to encompass the whole boundary, to the point of delamination from the surface (~12µm deep). The lower right image shows the delamination of parallel cleavage planes.

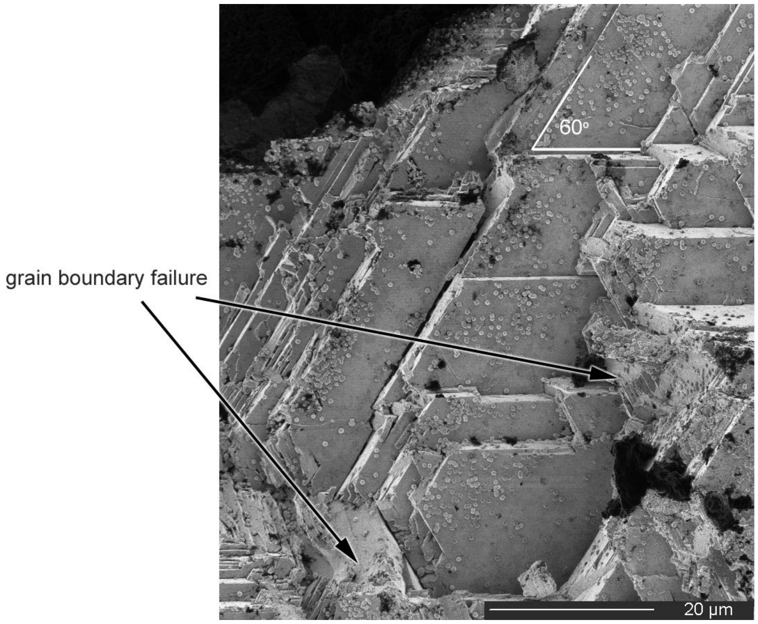

Figure 1 – Ion induced secondary electron image of a typical zinc fracture surface orientated so that the (0001) cleavage planes are approximately in the plane of the image.

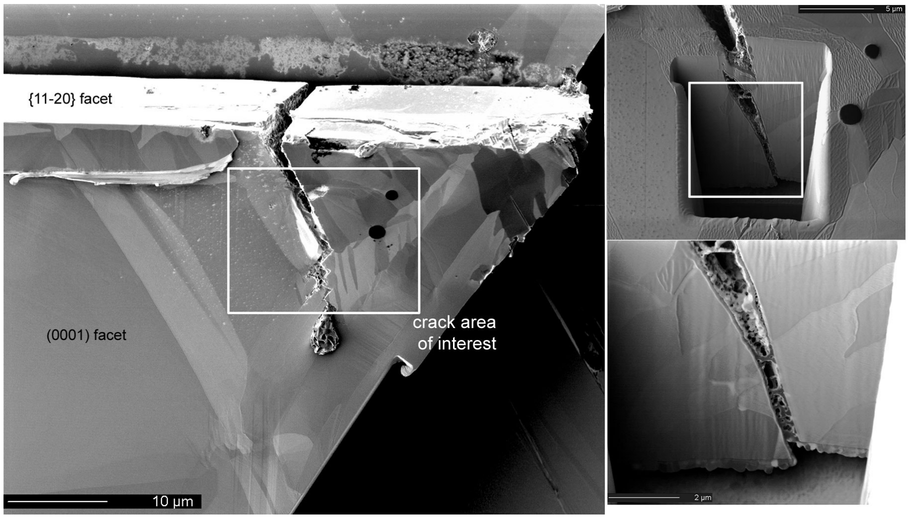

Figure 2 – An ion induced secondary electron image of a secondary crack within a network of twins. The inset images on the right show a cross section of the crack, milled at ~90º to the surface and then imaged at an angle of 45º to the beam.

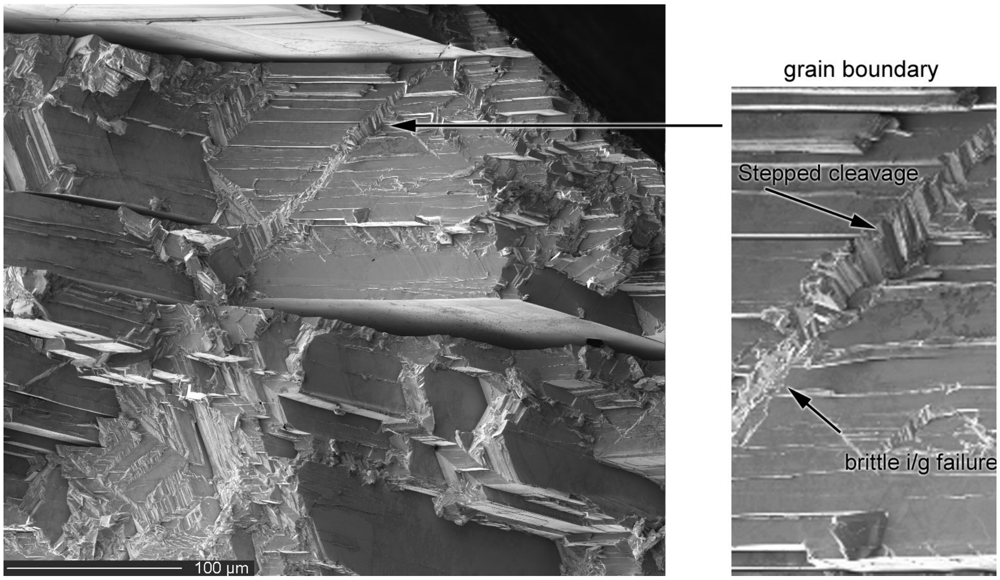

Figure 3 – A fracture surface showing cleavage and brittle grain boundary fracture and stepped cleavage; both accommodate the crack as it propagates across a grain boundary.

5 CONCLUDING COMMENTS

It should be noted that, as the computer models used in the simulations reported here are 2-d. Hence, the results do not include accommodation effects which are required in 3-d because cleavage planes in neighbouring grains do not meet in a line in their common grain boundary. If only one cleavage plane is available it has been shown geometrically that about two-thirds of the failure then has to be grain boundary or some other non-cleavage mechanism. The present experimental results show ample evidence for such mechanisms, particularly multiple prismatic cleavage. The modelling work is currently being refined in several ways. In particular a facility for introducing different fracture energy values for different cleavage planes is being introduced so that for example (0001) and {11-20} cleavage can be treated independently. However the main development is in generating three-dimensional models. Use of such models will enable energy values for different mechanisms to be deduced by comparing the simulation and experimental results.

The experimental results show the fracture surface to be complex. A relatively small amount of grain boundary failure is observed. This is due to an additional set of cleavage planes, perpendicular to the basal (0001) plane, along the {11-20} planes. This gives rise to a further three cleavage planes. So the total number of cleavage planes available for brittle failure is four. The accommodation mechanism at the grain boundary is a combination of brittle intergranular fracture and multiple stepped cleavage, similar to that observed by Qiao and Argon [3] for bcc iron 3% silicon alloys.

It is clear that more work has to be done in terms of modelling the fracture in polycrystalline zinc. The models discussed in this paper are mainly based around 2-d simulations. The third dimension will allow the additional factors identified in the experimental work, namely: secondary cleavage along {11-20} planes, the stepped cleavage accommodation at grain boundaries, and the presence of deformation twins. It is thought that the twins are formed as a result of a limited amount of plasticity at the time of the initial fracture event. The effects of twins on crack propagation and the formation of secondary cracks, which propagate along the twin boundaries are factors that will be considered further. Experimentally, the intention is to explore the effect of temperature on the fracture energy of zinc, and how the formation of deformation twins influences by changes of the fracture regime, from brittle to ductile.

ACKNOWLEDGEMENT

The authors would like to acknowledge the EPSRC for funding this conjoint work.

REFERENCES

[1] Crocker, A.G., Smith, G.E. And Flewitt, P.E.J., Computational Modelling Of Fracture In Polycrystalline Materials, Intl Materials Review (2004, In Press).

[2] Smith, G.E., Crocker, A.G., Moskovic, R. And Flewitt, P.E.J., Models To Describe Brittle And Ductile Fracture In Ferritic Steels, Philosophical Magazine A-Physics Of Condensed Matter Structure Defects And Mechanical Properties 82 (2002) 3443-3453.

[3] Qiao, Y. And Argon, A.S., Cleavage Cracking Resistance Of High Angle Grain Boundaries In Fe–3%Si Alloy, Mechanics Of Materials 35 (2003) 313-331.

[4] Schmid, E. And Boas, W. Plasticity Of Crystals, F.A. Hughes And Co., Ltd., London, 1950.

[5] Greenwood, G.W. And Quarrell, A.G., The Cleavage Fracture Of Pure Polycrystalline Zinc In Tension, Journal Of The Institute Of Metals 82 (1954) 551-560.

[6] Moore, A.J.W., Twinning And Accommodation Kinking In Zinc, Acta Metallurgica 3 (1955) 163-169.

[7] Pratt, P.L. And Pugh, S.F., Twin Accommodation In Zinc, Journal Of The Institute Of Metals 80 (1952) 653-658.

[8] Goldstein, J.I. And Yakowitz, H. (Eds.) Practical Scanning Electron Microscopy, Plenum Press, London, 1975.