# CHARACTERISATION OF HYDROGEN INDUCED FRACTURE IN THE TITANIUM ALLOY IMI685

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#### ABSTRACT

Evidence for sub-surface fatigue crack initiation is often reported for near alpha titanium alloys such as the coarse grained IMI685. With a typical as received hydrogen concentration of 60ppm the initiation site is invariably characterised by quasi-cleavage facetting. Similar facetting is also associated with low temperature dwell sensitive fatigue in the same alloy. This failure mechanism, however, is replaced by  $\alpha/\beta$  interface cracking when the alloy contains higher concentrations of interstitial hydrogen. The present paper explores the local grain orientation and microstructural conditions associated with these various forms of failure through the use of electron back scattered diffraction measurements (EBSD). The observations are related to a previously suggested model for facet formation based upon the pile-up of dislocations at grain boundaries.

#### KEYWORDS

Hydrogen,  $\alpha/\beta$  interface, facets, basal plane, EBSD, dwell sensitivity

#### INTRODUCTION

Sub-surface fatigue crack initiation is commonly reported for titanium alloys (Evans and Bache, 1994). In the near alpha systems containing typical as received hydrogen levels of about 60ppm, such sites are invariably characterised by quasi-cleavage facets with a near basal orientation (Davidson and Eylon, 1980). It is reported, however, that alloys with a relatively high concentration of interstitial hydrogen (>100ppm) fail by a different mechanism in which cracks initiate and grow along  $\alpha/\beta$  plate interfaces (Evans and Bache, 1995a). Clearly, the current drive to combine initiation based lifting methods with damage tolerant failure criteria requires a better understanding of these failure processes and of the localised conditions controlling crack growth.

The present paper characterises quasi-cleavage facets formed under fatigue loading conditions in the near alpha titanium alloy IMI685 (Ti-6Al-5Zr-0.5Mo-0.25Si). This alloy was considered as a suitable vehicle for the investigation due to its relatively large prior beta grain size and the large scale facets that form as a result. The analysis uses back scattered electron diffraction (EBSD) techniques to evaluate the crystallographic orientation of specific grains or alpha colonies in the alloy microstructure. It relies on the recognition of diffraction information

(Kikuchi patterns) obtained from back scattered electrons generated by the localised interaction of a primary electron beam with the crystallographic lattice features in the material. In the case of the titanium alloys, the orientation of the basal plane with respect to fracture surface features and the loading direction is of particular interest.

The fatigue failures were generated in laboratory test pieces. The IMI685 was tested in a coarse aligned microstructural condition with internal hydrogen levels of 60ppm in as received material and 250ppm after modification. Both cyclic and dwell fatigue fracture surfaces from tension and torsion loading modes were evaluated. Detailed optical and scanning electron microscopy studies allowed typical fracture morphologies to be selected for subsequent EBSD analysis. This analysis provided information on the crystallographic orientation of facets and the local orientation of grains immediately surrounding initiation sites. The measurements lend support to a model for facet formation previously proposed by Evans and Bache based on dislocation pile-ups (Evans and Bache, 1994).

# **EXPERIMENTAL METHODS**

Specimens were manufactured from IMI685 supplied in the form of 30mm round bar. The as received alloy contained a hydrogen concentration of 60ppm. Selected specimen blanks were degassed by vacuum annealing at 800°C for 18 hours and subsequently recharged to a relatively high hydrogen content of 250ppm using a hydrogenation technique previously reported (Evans and Bache, 1995a). Finally, each blank was solution heat treated in air above the beta transus (1030°C) for 45 minutes followed by furnace cooling to laboratory temperature and aging at 550°C for 24 hours. This produced a coarse aligned microstructure of transformed alpha plates with a small amount of retained beta at the lath boundaries (illustrated in Figures 3,6 and 7).

Fatigue testing was conducted at ambient temperature on a closed loop servo-hydraulic machine with a maximum load and torque capacity of 50KN and 400Nm. A tubular specimen design was used for both tension and torsion as reported previously (Bache and Evans, 1992). The "cyclic" waveform consisted of a 2 second rise and fall between peak and minimum stress with a one second hold at peak load to facilitate data acquisition. The "dwell" cycles had the same rise and fall times but included a 120 second dwell at peak load. All fatigue tests were carried out at an R ratio of 0.1.

Physical features on the fracture surfaces were characterised by optical and scanning electron microscopy. Crystallographic information was obtained in a Jeol scanning electron microscope (JSM 6100) equipped with an EBSD system incorporating a phosphor screen, low light video camera, and high resolution monitor. The methodology of EBSD pattern generation and analysis are well documented (Dingley and Randle, 1992) and the application of the technique to titanium alloys has been recently reported (Bache et al, 1996). The EBSD orientation data are presented as inverse pole figures.

Selected specimens were metallographically sectioned and mechanically polished at  $90^{\circ}$  to the plane of fracture. This required accurate positioning and alignment of facets in the case of 60ppm material and regions of  $\alpha/\beta$  interface cracking in the higher hydrogen specimens. The orientation of these features was inferred from readings taken on orthogonal sections.

EBSD measurements were also taken directly from the fractured surfaces. These specimens were cleansed in an ultrasonic bath containing a surface degreasing solution and lightly etched using Kroll's agent. The electron beam was focussed directly onto facets or other features on the fracture surfaces. This method proved to be more difficult due to residual plasticity in the region, distortion of the features and alignment problems. Consequently, it could only be applied for a more limited range of features.

#### **RESULTS**

Typical cyclic and dwell fatigue data for IMI685 with 60ppm and 250ppm hydrogen contents are shown in Fig. 1. The tension and torsion results have been correlated through a von Mises equivalent stress criterion. The applied fatigue stress levels are normalised by the monotonic tensile strengths of the alloy with the two hydrogen contents (976 and 998MPa at 60 and 250ppm respectively). On this basis, increased hydrogen levels reduce fatigue life, especially under dwell loading where reductions up to two orders of magnitude are observed compared to the 60ppm material. Specimens selected for subsequent EBSD analysis are indicated by the letters A, B and C.

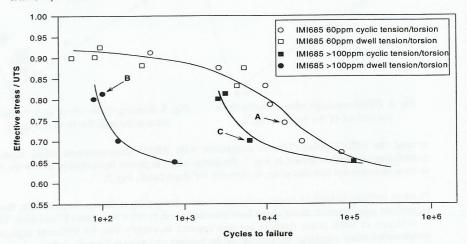


Fig. 1. Low cycle fatigue data for IMI685.

Fracture surfaces displayed characteristic features according to the different hydrogen concentrations. In the as received 60ppm level, quasi-cleavage facets were evident. A single dominant facet orientated approximately perpendicular to the tensile axis was often the site of crack initiation particularly at lower stress levels. In one testpiece (specimen A) chosen for EBSD analysis the facet extended across the wall thickness of the tubular specimen (i.e. 1.5mm), Fig. 2. Its orientation was confirmed as perpendicular to the tensile loading axis by a series of mounted sections through the facet, Fig. 3. The facet surface is remarkably flat and consequently EBSD readings could be taken from the surface at random positions. The measurements are summarised by the corresponding inverse pole diagram of Fig. 4. The essentially basal nature of this facet is evident (demonstrated by the concentration of points

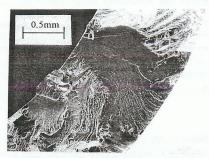


Fig. 2. Facet in 60ppm material.

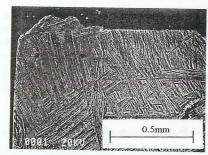


Fig. 3. Section of the 60ppm facet.

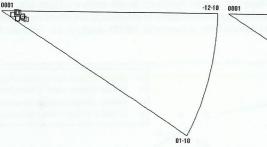


Fig. 4. EBSD readings taken directly from the surface of the facet in Fig. 2.

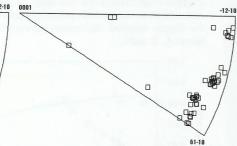


Fig. 5. Readings taken from an orthogonal section through the facet in Fig. 2.

around the [0001] pole). This is consistent with EBSD measurements taken from the metallographic section shown in Fig. 3. Readings taken at points immediately below the facet surface demonstrate that this plane is virtually 90° from basal, Fig. 5.

Fracture surfaces in high hydrogen testpieces were substantially different. In particular, there is significant crack growth along alpha-beta interfaces and at prior beta grain boundaries. There is evidence of basal plane fracture but this appears to simply link the different regions of interface/boundary cracking. By comparing the tension and torsion fracture surfaces, however, it is evident that at least two dominant factors control the fracture response. From the tension specimen (C) it is clear that the orientation of the microstructure and, in particular, the  $\alpha/\beta$  interfaces has a strong influence on fracture path, Fig. 6. The torsion crack path (specimen B), however, is characteristically helical emphasizing the important role of the maximum principal stress, Fig. 7. EBSD measurements taken at random along either crack path contrast markedly with those from the 60ppm material in that they no longer show a preferred tendency for the crack to seek out basal planes, Fig. 8.

Fracture surface features are consistent with the metallographic sections and with the deductions from the EBSD analysis. Thus there is clear evidence of  $\alpha/\beta$  interface cracking, grain boundary separation and propagation across basal planes, Fig. 9. At higher magnifications, the interfaces show evidence of terraces, containing residual material or rafts, Fig. 10. EBSD measurements taken from these rafts demonstrated a body centred cubic

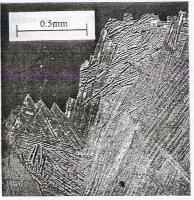


Fig. 6. High hydrogen tension failure.

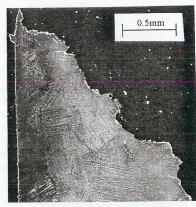


Fig. 7. High hydrogen torsion failure.

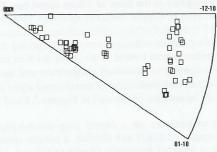


Fig. 8. EBSD readings taken from the section shown in Fig. 6.

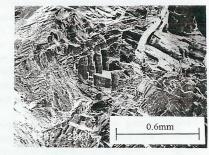


Fig. 9. High hydrogen fracture surface.



Fig. 10. Beta raft upon underlying alpha plate, exposed via  $\alpha/\beta$  interface cracking.

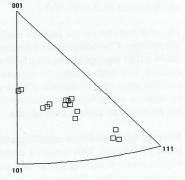


Fig. 11. Body centred cubic readings taken from various beta rafts.

structure, Fig. 11, consistent with the residual beta phase. The relevance of the scatter shown by the points in Fig. 11 is currently under evaluation. On basal planes, ductile voids were observed with an elongated morphology, Fig. 12.

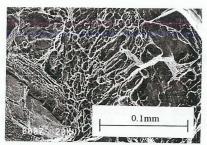


Fig. 12. Ductile voids on high hydrogen fracture surface.

# DISCUSSION

It is clear that the failure response of IMI685 with an aligned microstructure is strongly influenced by the amount of hydrogen present. At 60ppm, the fracture events are dominated by the formation of quasi-cleavage facets. These are known to have a basal plane orientation (Eylon, 1979) and this is confirmed by the present EBSD measurements. Previous studies have shown that facets are still dominant when the hydrogen content is reduced to < 20ppm (Evans and Bache, 1994). On this basis, it can be argued that hydrogen is not a necessary requirement for facet formation. Increasing the hydrogen content to approximately >100 ppm, however, induces an entirely different mode of failure in which the cracks propagate along alpha/beta interfaces and down prior beta grain boundaries. This path is confirmed in the present work through the EBSD measurements and the identification of the beta phase on the plane of fracture. It is now argued that the higher hydrogen content has led to hydride induced crack initiation and growth (Evans and Bache, 1995a). Basal planes at these levels appear to act as links between the interface cracks as evidenced by the presence of ductile tearing and elongated voids.

The mechanical property data also display important variations depending on the amount of hydrogen present. Figure 1 displays one aspect of this, in which it is evident that at 60ppm the fatigue lives of dwell and non-dwell loading conditions are similar i.e. for the aligned microstructure there is not a significant dwell sensitivity. Previous work (Evans and Bache, 1994) has confirmed a similar pattern of behaviour for low hydrogen levels. In contrast, the 250ppm hydrogen concentration induces a large dwell sensitivity in which the 120 second hold at maximum load causes a two orders of magnitude reduction in life compared with the cyclic waveform. Conversely, if the data are plotted on a time basis the response at the two hydrogen levels is reversed. The time to failure for cyclic loading of 60ppm material is now considerably shorter than the equivalent dwell cycle. At the same time the rate and level of strain accumulation is significantly larger under dwell. At >100 ppm, however, both dwell and cyclic experiments fail in approximately the same time, Fig. 13. Furthermore, the strains at failure are approximately the same for both loading modes - about 0.0055 at 750 MPa. Subsequently, when the strain - time records under dwell loading for 60 and 250ppm levels are compared, it

is found that they initially superimpose but that the latter prematurely fails. These various sets of data clearly support the observed change in fracture mode with hydrogen content.

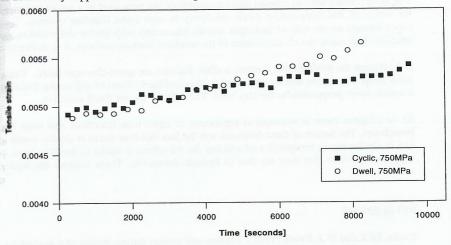


Fig. 13. Strain accumulation in IMI685 with 250ppm hydrogen content.

It is well established that facet formation requires both shear on the basal plane and a tensile stress perpendicular to the resultant slip band (Wojcik et al, 1988). The interesting variation in this case is that the facets are essentially perpendicular to the applied stress. Thus they are well positioned for the tensile stress but would find shear difficult. It has been suggested (Evans and Bache, 1994) that this difficulty is resolved when an adjacent grain is favourably orientated for slip. The resultant dislocation pile-up will induce both shear and tensile stresses on the perpendicular basal plane thereby precipitating facet formation. Hydrogen at this level acts simply through modification of the deformation response which may include a reduction in stacking fault energy and hence more extensive pile-ups. At the higher hydrogen content it has already been intimated that the fracture surface features are consistent with hydride formation. It is now argued that this view is reinforced by the mechanical response and in particular the strain-time behaviour. Hydride formation is associated with an increase in volume locally (Moody and Gerberich, 1982). This change, therefore, will be facilitated by strain accumulation. It is thus relevant that both cyclic and dwell failures occur at approximately the same strain suggesting that there is a critical value for each stress level. It is also noticeable that the failures occur at virtually the same time suggesting that diffusion of hydrogen to specific locations at the alpha/beta interfaces is the rate controlling event. The fact that similar features are evident along the entire crack path indicates that these events are occurring throughout the volume of the testpiece. In fact the superposition of the strain-time records during the early dwell life for each hydrogen level and the subsequent premature failure at 250ppm indicates a general breakdown throughout the sample with local link-ups through ductile tearing in regions where the interface is not favourably orientated with respect to the maximum principal stress e.g. the elongated voids on basal planes. In conclusion, it is argued that there is a logical and progressive change in deformation and fracture behaviour with increasing hydrogen content which involves interactions between deformation and hydrogen atoms which eventually leads to hydride formation.

# CONCLUSIONS

The alloy IMI685 with an aligned alpha morphology has been used as a model material to study the nature of low temperature dwell sensitivity in near alpha titanium systems. The present paper focuses on the role of hydrogen and its interaction with plastic deformation. Particular attention is given to the characteristics of the resultant fracture surfaces. It is shown that:

- i) At 60ppm the predominant fracture surface features are quasi-cleavage facets. These have a basal plane orientation and are attributed to the combined effects of slip on the basal plane and a tensile stress perpendicular the slip band. The failures are not dwell sensitive.
- ii) At 250ppm there is widespread separation of alpha-beta interfaces and prior beta grain boundaries. The nature of these fractures and the fact that they occur at similar levels of strain and in similar times, irrespective of whether the waveform is cyclic or dwell in form, strongly supports the view that they are due to hydride formation. These failures are highly dwell sensitive.

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