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The growth of cracks within brittle zirconium hydride platelets contained in a ductile zirconium matrix

Dr C. J. BEEVERS* B. Met., Ph.D., and Dr M. R. WARREN* B.Sc., Ph.D.
*Department of Physical Metallurgy and Science of Materials,
The University, Edgbaston, Birmingham 15, England.

Summary

The tensile behaviour of a two phase material consisting of randomly oriented brittle zirconium hydride platelets in a ductile zirconium matrix has been studied at −196°C. Surface metallographic examinations made during interrupted tests revealed the following observations. Two distinct stages in the fracture of the hydride platelets could be distinguished. These are (a), the nucleation and formation of cracks transverse to the major dimensions of the platelets, and, (b) the formation of in-plane cracks which propagate in directions parallel to the major dimensions of the platelets. Stage (a) was related to the intersection of the platelets by matrix twins and slip bands but Stage (b) was not achieved until the spacing of the transverse cracks was of the order of the platelet thickness. The initial formation of the in-plane cracks was apparently controlled by a normal stress condition, but the later part of the crack growth in this stage was closely related to the overall plastic strain of the material. It was noted that at a constant plastic strain the larger the platelets the longer the in-plane cracks contained within them. Increasing grain size and hydrogen content results in larger hydride platelets and the enhanced embrittlement at −196°C under these conditions can be directly related to the increase in length of the in-plane cracks introduced into the ductile matrix.

Introduction

It is now well established that the nucleation and formation of cracks in inclusions and precipitates can be related to the deformation behaviour of the containing matrix. The intersection of slip bands and or twins with carbide precipitates in steels [1-3], and hydride precipitates in zirconium [4, 5] can lead to precipitate fracture. Whilst there is some variation in the reported plastic strains at which initial fracture can occur [1, 3, 5] there is general agreement that crack formation and growth occur continuously with increasing strain until final failure occurs. The influence of precipitate morphology on the occurrence of crack formation has not generally been investigated quantitatively, but there are several observations which indicate that crack formation can occur more readily as the precipitate or inclusion size is increased. Yukawa [6] has investigated the fracture of sulphide inclusions in steel and reported that the larger inclusions are the first to break. Pickering [7] found that the thicker the cementite lamellae in steels the more readily they fractured. In zirconium-hydrogen alloys, the thicker the zirconium hydride platelets the more extensive the crack formation within the precipitates [8]. In an investigation of the initiation of ductile fracture in aluminium-silicon alloys Gurland and Plateau [9] observed that the larger particles fractured before the smaller ones. For platelet precipitates there is also some evidence that the orientation of the platelet to the stress axis can influence the occurrence and extent of precipitate fracture [10-12].

The results to be presented in this paper are concerned with the fracture processes in zirconium containing zirconium hydride platelets. The fracture of the hydride platelets is analysed in terms of a two stage process, (a) crack nucleation in the hydride platelets and (b) crack propagation within the platelets. Crack nucleation, Stage (a), can occur over a wide range of test conditions and precipitate morphologies but does not necessarily lead to severe embrittlement of the ductile matrix. The occurrence of the transition from Stage (a) to (b) however, appears to be a more critical event in the fracture process, leading to the introduction of matrix cracks which can significantly influence the stresses necessary to produce final fracture.

Experimental techniques

Tensile specimens cut from high purity zirconium plate* were hydrided to hydrogen contents of 25, 50 and 100 ppm, following an initial anneal at temperatures from 650°C to 850°C for times from ½h to 4h under a dynamic vacuum of better than 10^{-5} mm/Hg to produce grain sizes in the range 0·0075 mm-0·265 mm.

The hydriding was carried out in a modified Seiverts apparatus into which a known pressure of hydrogen calculated from Pressure-Temperature-Composition (P/T/C) data was introduced [13]. The hydriding temperature of 450°C restricted the possibility of grain growth. The specimens were held at this temperature for 24h to allow complete homogenisation to occur prior to slow cooling at a rate of ~100°C/h. Hydrogen content was checked by means of weight gain and it was found that this method agreed to within ±3% with P/T/C calculations.

The specimens were tested in tension at various temperatures in a modified Polanyi hardbeam tensometer at a strain rate of $\sim 5 \times 10^{-4}$/sec.

Experimental results

(1) *Nucleation of transverse cracks within hydride platelets*

A recent critical appraisal of the crack nucleation stage of this fracture process has indicated [5] that of the three major deformation modes in the zirconium matrix, that is $\{11\bar{2}1\}$ and $\{10\bar{1}2\}$ twinning and $\{10\bar{1}0\}$ slip, only $\{11\bar{2}1\}$ twins and prismatic slip bands are capable of initiating fracture in the hydride. This fracture takes the form of short transverse through thickness cracks in the hydride platelets. Examples of this transverse cracking* are shown in Fig. 1 which demonstrates the extent of cracking that can occur when slip bands intersect the planes of the hydride platelets. $\{11\bar{2}1\}$ parallel sided twins begin to form transverse cracks at their intersections with hydride platelets as soon as the yield stress is exceeded, and as the twin density increases due to the increasing strain applied to the specimen during test so the number of cracks caused by this mechanism also increases. However, the $\{11\bar{2}1\}$ twin mechanism for crack nucleation only produces a very low density of transverse cracks per platelet, but as seen in Fig. 1 the slip mechanism gives rise to multiple cracking of the platelets.

In order to ensure ductility in polycrystals five independent deformation modes must be available to guarantee compatibility between the macroscopic strains in contiguous grains [14, 15]. Thus as twinning activity begins to decrease prismatic slip begins to occur on a more widespread scale. This increase in slip activity is generally observed to occur at ~15-25% of the total strain to fracture irrespective of grain size and hydrogen content for specimens tested at –196°C. In material containing 50 ppm hydrogen and having a grain size of 0·18 mm, this stage in the deformation process is attained at ~5-8% plastic strain, and the second mechanism for the nucleation of transverse cracks in the platelets begins to occur. Slip band formation is extensive and transverse cracks are nucleated in the hydride platelets by the stress concentrations generated by the slip dislocations as they pile up at the platelet matrix interfaces.

During these investigations it was also shown that the frequency of transverse cracks nucleated by either the twin or slip mechanisms was independent of the orientation of the hydride platelets to the tensile axis.

From the results presented above it is seen that the nucleation of transverse cracks by the slip mechanism shows an apparent dual involvement of both strain and stress in that the cracks are never formed until about 5-8% strain which is the strain at which extensive slip band generation occurs and that the shear stress concentration which can be developed in the slip bands at the precipitate-matrix interface will be ultimately responsible for the nucleation process.

(2) *Formation and growth of in-plane hydride cracks*

From surface metallographic observations on chemically etched surfaces taken during interrupted tensile tests at –96°C, and also from conventional post failure metallographic examinations together with analyses of results

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* Prepared from 50% cold-rolled arc melted iodide zirconium. Total impurity content ~ 800 ppm, including 30 ppm oxygen and 30 ppm hydrogen.

* A transverse crack in the hydride platelet is considered to be one where the plane of the crack is near normal to the major plane of the platelet and where the major dimension of the crack is of the order of the thickness of the platelet (see Fig. 7.)

taken from the continuous recording of the electrical resistance of the specimen during tensile testing, [5], there is now considerable circumstantial evidence to suggest that the in-plane hydride cracks* (Fig. 2) which lead to final fracture of the zirconium by propagation through the matrix, are developed from the slip induced transverse cracks in the hydride platelets by the operation of a transfer process.

(a) *Experiments supporting a transfer process*

During interrupted tests at –196°C on specimens containing 50 ppm hydrogen and having a grain size of 0·18 mm., the numbers of in-plane hydride cracks on the specimen surfaces were recorded. It was found that no in-plane cracks were present until strains of ~5-8% had been attained and also that the number of cracks increased progressively with strain as the incidence of slip band intersection with the platelets increased. Furthermore, it was observed that the curve of the number of in-plane cracks versus strain bore no resemblance to the curve of the number of transverse cracks caused by the twin mechanism versus strain.

These observations which suggest an association between transverse crack nucleation and in-plane crack formation were supported by measurements of specimen resistance, in that an increase in specimen resistance related to the formation of in-plane cracks did not become apparent until strains of ~5-8% had been attained.

Further corroborative evidence of the transfer from slip induced transverse cracks to in-plane cracks was provided by the results of tests on specimens having a grain size of 0·18 mm and containing 50 ppm hydrogen which were first prestrained at R.T. (15% strain) and subsequently tested to failure at –196°C. The form of the –196°C stress-strain curve so obtained may be compared in Fig. 3 with the curve obtained from a straight-through test at –196°C. Metallographic examination revealed that after 15% plastic strain at R.T. no in-plane hydride cracks were formed but there were many slip-induced transverse cracks. After only 1·5% further extension in the subsequent test at –196°C in-plane cracks (Fig. 4) had been formed within the platelets. This lower value of 1·5% plastic strain to produce in-plane cracks at –196°C following a prestrain at R.T. should be compared with the 5-8% plastic strain required in a straight through test at –196°C. The occurrence of the serrations immediately following yield (Curve B, Fig. 3) gives support to the suggestion that the in-plane cracks formed at small plastic strains.

(b) *Normal stress criterion*

Several series of specimens containing 25, 50 and 100 ppm hydrogen and having grain sizes in the range 0·0075 mm-0·265 mm were tested at –196°C and the orientations to the tensile axis of hydride platelets in which in-plane cracks had developed were studied. These results [5] plotted in the form of a frequency distribution curve followed a power cosine function ($\cos^m \theta$ with $2<m<4$) and could be fitted to an equation of the type

$$\sigma_n \simeq \sigma \cos^a \theta \cos^b \phi$$

where σ is the applied stress, σ_n the normal stress acting on the precipitate, θ and ϕ are the two angles that a platelet makes to a plane normal to the tensile axis, and where a and b for the ideal case would obey the relationship $a=b=2$. The conclusion drawn from this analysis is that the formation of the in-plane cracks within the platelets depends strongly on the orientation of the platelets to the tensile axis and is probably controlled by a normal stress criterion.

(c) *Growth of in-plane hydride cracks*

An extensive study of in-plane crack growth was carried out. Interrupted tests at 196°C, were performed on six specimens, containing 50 ppm hydrogen and having a grain size of 0·18 mm, which had been polished and etched prior to testing. The strain increments were of the order of 1-2% and after each strain increase the changes in in-plane crack length, *L*, were recorded for ~15 of the cracks visible on each specimen. The cracks recorded were of the type seen in Fig. 4. In order to eliminate the effects of anisotropic strains and the effects of the orientation dependence of formation mentioned above, certain types of cracks were excluded from the subsequent analysis of the results. These were (a) cracks near the edge of the specimen (not measured at all), (b) cracks with an orientation greater than 10° away from the normal to tensile axis (not measured at all), (c) cracks which in the later stages were seen to be developing near or in the neck of the specimen (eliminated from the analysis) and (d) cracks which subsequently interconnected with other cracks (eliminated from the analysis). Of the results remaining after this elimination procedure (93 cracks measured; 38 eliminated by conditions c or d) those cracks for one of the six specimens were recorded as shown in Fig. 5 in terms of crack length vs strain. The figure has been limited to only one of the specimens for reasons of clarity. For this particular specimen 13 cracks were recorded; those designated 4, 6, 11 and 12 subsequently interconnected whilst those designated 7-10 were later seen to be associated with the necked region of the specimen.

From Fig. 5 it is seen that the crack length, *L*, increases linearly with strain. The diagram also shows that this linear relationship with strain only holds for cracks which on the specimen surface appear to be completely contained within a hydride platelet. Once the crack has grown to a certain limiting length imposed by the original platelet dimensions it begins to interact with the matrix at the specimen surface, as denoted by the full dots

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* The in-plane hydride crack is a crack whose major dimensions lie in the plane of the platelet.

in Fig. 5 and subsequently crack growth either ceases or its rate of growth is drastically reduced. From the curves of length against strain the rate of crack growth denoted by $dL/d\epsilon$ was obtained. This parameter, for all viable results from all six specimens, is shown as a function of the length of the hydrides containing the cracks in Fig. 6 which demonstrates a linear increase in growth rate as the hydride platelet length is increased.

Further to this it was also seen qualitatively that the lengths of the cracks when first observed increased as the lengths of the containing platelets increased.

Discussion

The zirconium hydride platelets form such that the major planes of the platelets are parallel to the $\{10\bar{1}0\}$ planes of the zirconium matrix and their major growth directions are the $\langle 11\bar{2}0\rangle$ directions in the zirconium matrix. The crystallographic relationships between the precipitate and the matrix are [16];

$$\{10\bar{1}0\}_{\text{matrix}} \parallel \{111\}_{\text{platelet}}$$

$$\{11\bar{2}0\}_{\text{matrix}} \parallel \{110\}_{\text{platelet}}$$

It should be noted that the major slip mode in zirconium is $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$ [17]. Bulk zirconium hydride is brittle in compression below temperatures of approximately 120°C [18] and the most probable cleavage planes are of the $\{111\}$ type [19]. Thus, from the above considerations the fracture mode of a hydride platelet is expected to be cleavage along planes both parallel and near normal to its major plane.

The type of transverse fracture shown in Fig. 1 is comparable, when formed internally away from the specimen surface, to a through thickness crack in sheet material, and as such it will be relaxed by the matrix at the hydride matrix interface. This relaxation will tend to inhibit unstable growth of the crack in both the precipitate and the matrix. However, since the transverse crack is in part contained in a brittle material there will be the possibility, in internal regions of the platelet, of high elastic stress concentrations at the crack tip. Consequently, if there are significant stress components normal to other cleavage planes lying in or almost parallel to the plane of the platelet, there will be the possibility of cleavage crack formation on these planes. Such a crack once initiated would essentially be free of any matrix relaxation pinning it and it could therefore propagate as an in-plane hydride crack without being impeded until it subsequently interacted with the matrix at the platelet matrix interface.

If this hypothesis is correct then in-plane hydride cracks should be in evidence as soon as the short transverse cracks are formed. This however is not the case. The first transverse cracks are formed at around the yield stress by the interaction of $\{11\bar{2}1\}$ parallel sided twins with the platelets [5], but in-plane cracks are not formed until later in the strain history of the specimen, when the formation of transverse cracks by the slip mechanism also begins to occur. Metallographic evidence has shown that the number of cracks per unit length of platelet caused by the slip mechanism greatly exceeds that resulting from the twin mechanism. It is therefore concluded that the possibility of in-plane crack formation, due to the stress concentration from transverse cracks with a spacing between them which is ≫ platelet thickness, is remote. Ichikawa *et al.* [20] have considered arrays of two or more cracks and have shown that when the crack spacing approaches the crack length this can lead to a reduction in overall stress for fracture. This condition is somewhat analogous to the hydride platelets containing different transverse crack distributions, and is in agreement with the present observations that close spaced transverse cracks can lead to the formation of in-plane cracks.

These ideas are consistent with metallographic observations. In Fig. 7 the change over from transverse to in-plane cracks has been idealised diagrammatically. Referring to the figure it should be noted that the slip bands do not necessarily lie in the plane of the diagram, that is the plane of the platelet. Furthermore the line of intersection of the slip bands with the platelet has been placed at XX′ to simplify the presentation; the line XX′ could be envisaged to occur in any plane intersecting the platelet such that the crystallography of the system is consistent with the interaction of $\{10\bar{1}0\}$ slip bands with platelets lying on $\{10\bar{1}0\}$ planes.

The transverse cracks are pinned above and below the plane of the diagram by matrix relaxation, but the stress concentrations at their tips within the platelet reinforce and flat in-plane cleavage cracks are formed at a, b, c and d. These cracks now coalesce and the crack front, shown by the dotted lines spreads through the platelet until the precipitate matrix interface is reached whence the crack propagation rate is significantly reduced by further matrix relaxation. Assuming that this simplified model is valid, three basic types of in-plane hydride crack should be identified in the specimens. Ideally they are obtained by taking sections from Fig. 8 perpendicular to the plane of the hydride platelet at AA′, BB′ and CC′. The types of cracks observed are shown in Fig. 8 together with the diagrammatical representations as assessed from Fig. 7.

From the study of the influence of strain on in-plane crack length (Fig. 5) it was noted that the in-plane cracks were first observed with a finite length and that this length was achieved within a strain increment of only 1-2%. In order for this to have happened the initial growth of the crack must have occurred at a faster rate than those subsequently recorded and presented in Fig. 5.

It is reasonable to assume therefore that the in-plane crack once formed in the region a, b, c, d (Fig. 7), propagates as a fast cleavage crack, subject to the normal stress criterion described previously, until it interacts with the matrix at some point such as D or D′. Still referring to Fig. 7 it will be seen that, if AA′ is contained in the specimen surface such that ABB′A′ is near normal to the surface, then the crack will appear to have a limited length by the time the crack is slowed down by the matrix relaxation at D and D′. Thus the slow growth recorded in Fig. 6 may be compared to the growth of the crack in the AA′ plane of Fig. 8 after it has interacted with the matrix at levels lower down in the specimen. The slowness of the growth is therefore attributable to the restraining influence of the matrix, since the crack can only grow against this relaxing and pinning effect of the matrix by propagation of the crack in the region of DA and D′A′. This relaxation of the crack by the matrix during the later stages of in-plane crack growth is supported by the fact that the growth of the cracks is strain controlled as evidenced by Fig. 5. Qualitatively it is also seen from Fig. 5 that the larger the platelets the longer the crack length at which strain controlled crack growth occurred. The rate of this growth increased linearly with precipitate length Fig. 6. As the crack length was longer in the larger platelets the driving force for crack growth in these precipitates would be higher and thus could possibly explain the faster growth rate. However, as outlined earlier this growth stage is also associated with the interaction of the in-plane cracks with the ductile matrix and this introduces an element of strain control into the growth of the cracks.

Models based on the intersection of precipitates by dislocation pile-ups to produce crack nucleation in the precipitates have been proposed [21, 22]. When the pile-up length is large compared with the precipitate radius or thickness the models predict that fracture will occur more easily as the precipitate radius or thickness decreases. At first sight these models would appear to be at variance with the observations [6-9] that the larger the precipitate the more readily they fracture. However, the models particularly concerned with the nucleation and formation of cracks similar in nature to the transverse cracks in Fig. 1. In zirconium hydrogen alloys where the precipitate platelet thickness is progressively increased through increases in hydrogen content a transfer from transverse crack to in-plane crack formation has been observed at 20°C. [8]. This transfer process is consistent with the model outlined previously in that increasing platelet thickness would reduce the relaxation and pinning of the transverse cracks by the matrix. It may well be that fracture of precipitates in other materials is not a single stage process occurring when a nucleation condition in the precipitate is attained, and that, a further stage similar in nature to the in-plane crack formation is necessary to produce recognisable precipitate fracture. If this is the case then care should be taken to evaluate the detailed fracture mode of the precipitate or inclusion if direct correlations with theoretical models are to be made.

The growth of the in-plane hydride cracks into the matrix is possibly strain controlled but analyses of the final propagation of the matrix cracks to produce the final failure of the composite material have shown [23] that the results for this final failure stage are most readily rationalised not in terms of strain to fracture or a tensile stress condition but in terms of maximum matrix crack length and stress, which for zirconium tested at −196°C, obey the relationship,

$$\sigma_f \propto l^{-1/2}$$

irrespective of grain size and hydrogen content, where σ_f is the fracture stress and l is the maximum crack length.

This study [20] of the fracture of zirconium-hydrogen alloys having different grain sizes and hydrogen contents showed that increasing grain size and hydrogen content reduced both the tensile stress and strain to fracture. An increase in grain size and hydrogen content led to an increase in precipitate length and, as shown above, at a constant plastic strain the longer the platelet the longer the crack introduced into the matrix. It is not surprising therefore that increases in grain size and/or hydrogen content produce embrittlement.

Conclusions

In zirconium-hydrogen alloys containing brittle second phase hydride platelets in a ductile zirconium matrix the platelets can exhibit a two stage fracture process.

(a) Transverse crack nucleation can occur as a result of twin and slip band intersection with the platelets. This stage of the process exhibits both strain and stress control in that crack formation due to coarse slip bands does not occur until 5-8% plastic strain and the stress concentrations necessary to nucleate fracture in the platelets are related to the shear stress acting in the slip bands.

(b) The initial formation of in-plane cracks appears to exhibit a normal stress criterion whereas the growth of the cracks at a later stage is controlled by the overall plastic strain superimposed on the material. After formation it was noted that for the same overall plastic strain the larger the hydride platelets the longer the in-plane cracks. Furthermore, during the strain controlled growth stage the rate of crack growth increases linearly with platelet length.

The occurrence of in-plane crack formation in platelets requires prior transverse crack formation such that the crack spacing is of the order of the platelet thickness. The transfer form (a) to (b) is encouraged by increasing platelet thickness and lower test temperatures, both of which restrict the relaxation of the transverse cracks by the ductile matrix.

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Fig. 1. Transverse platelet cracking caused by slip band interaction with the platelet. (0·18 mm. grain size, 50 ppm. hydrogen, test temperature –196°C). × 315.

Fig. 2. In-plane hydride crack formed in a hydride platelet which had previously exhibited transverse cracking. (0·18 mm. grain size, 50 ppm. hydrogen, test temperature –196°C)- × 315.

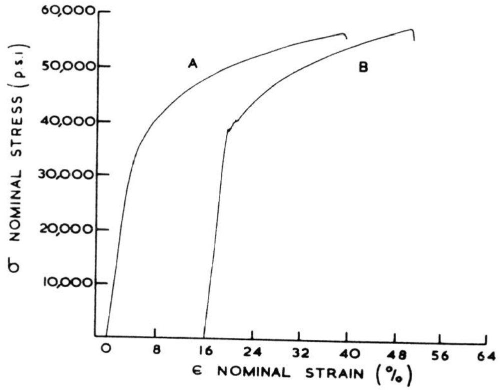

Fig. 3. Nominal stress-strain curves for two specimens tested at –196°C (0·18 mm. grain size, 50 ppm. hydrogen). Curve A was obtained from a straight through test, whilst the specimen used to obtain curve B had previously sustained 15% plastic strain at room temperature.

Fig. 4. Photomicrograph showing the development of an in-plane crack after only 1·5% plastic strain at –196°C, in a specimen which had previously sustained 15% plastic strain at room temperature. (0·18 mm. grain size, 50 ppm. hydrogen). × 315.

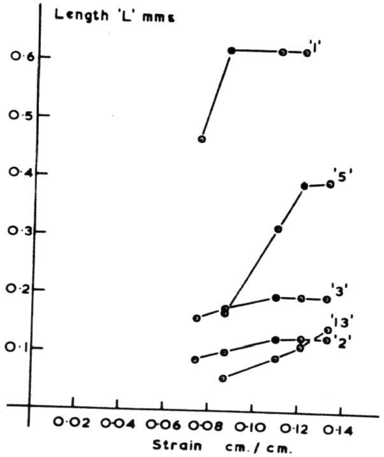

Fig. 5. Showing the length of the in-plane hydride cracks as a function of strain. The numbers adjacent to the various curves refer to specific cracks and the full circles represent the points at which the hydride cracks were first observed to be interacting with the matrix.

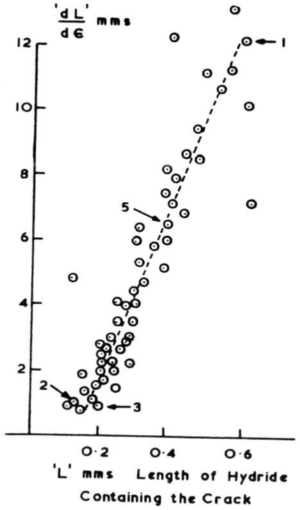

Fig. 6. Showing the rate of in-plane crack extension prior to interaction with the matrix as a function of the length of the hydride platelet containing the crack. All viable data is included, the numbered points referring to Fig. 5.

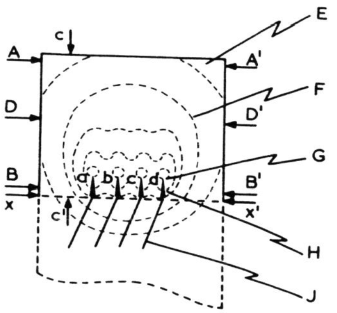

Fig. 7. A diagrammatic representation of a suggested model whereby an in-plane cleavage crack may be formed within a hydride platelet due to the presence of transverse cracks induced by slip interaction with the platelet. ABB′A′ represents a plane within the platelet lying parallel to the major plane of the platelet. This plane intersects the plane KLMN which is perpendicular to ABB′A′ in the line XX′. The slip dislocation pile-ups are visualised as lying at an orientation of θ° to the plane ABB′A′ of the platelet which lies on a prismatic type plane of the matrix.

E: Slip dislocation pile-ups.

F: Transverse crack induced by slip interaction.

a, b, c, d: Regions of the transverse crack tips which are thought to act as the initiation points for the formation of the in-plane cracks.

G: Crack front of the in-plane cleavage crack which propagates radially in all directions contained in the major plane of the platelet from the initiation points a, b, c, d.

The change over from transverse to in-plane cracking in this model is favoured by the applied stress acting in a direction near normal to the plane ABB′A′ (5).

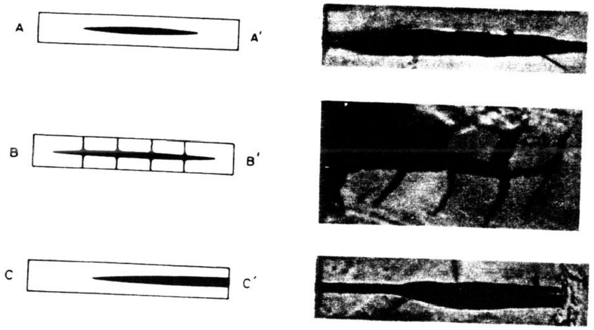

Fig. 8. Diagramatic representations of the types of in-plane hydride cracks obtained by taking sections AA′, BB′ and CC′ perpendicular to the plane of the diagram in Fig. 8. Actual examples of these types of cracks are also shown (AA′ × 570, BB′ × 840, CC′ ×570).