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3D growth of a Short Fatigue Crack within a Polycrystalline Microstructure studied using combined Diffraction and Phase Contrast X-ray Tomography

Michael Herbig¹, Andrew King², Péter Reischig³, Henry Proudhon⁴, Erik M. Lauridsen⁵, James Marrow⁶, Jean-Yves Buffière¹, Wolfgang Ludwig^{1,7}

¹Université de Lyon, Lyon, France, MATEIS, UMR5510 CNRS, 69621 Villeurbanne, France;

² GKSS-Research Center, Geesthacht, Germany;

³ Karlsruhe Institute of Technology, Eggenstein-Leopoldshafen, Germany;

⁴MINES ParisTech, Centre des matériaux, CNRS UMR 7633, BP 87, 91003 Evry Cedex, France;

⁵Risø-DTU, Technical University of Denmark, Roskilde, Denmark

⁶Manchester Materials Science Centre, Manchester, UK;

⁷ European Synchrotron Radiation Facility, Grenoble, France, BP 220, 38043 Grenoble, France

*Corresponding author: wolfgang.ludwig@insa-lyon.fr

Abstract

X-Ray Diffraction Contrast Tomography (DCT) is a recently developed, non-destructive synchrotron imaging technique which characterizes microstructure and grain orientation in polycrystalline materials in three dimensions (3D). By combining it with propagation based Phase Contrast Tomography (PCT) it is possible to get a full picture description for the analysis of local crack growth rate of short fatigue cracks (SFCs) in 3D: the 3D crack morphology at different propagation stages, and the shape and orientation of the grains around the crack. An approach has been developed on the metastable beta titanium alloy “Ti 21S” that allows for visualization and analysis of the growth rate and crystallographic orientation of the fracture surface.

Keywords

Short fatigue cracks, diffraction contrast tomography, synchrotron 3D imaging, in-situ, Ti 21S

Introduction

The short crack stage of fatigue life is of special interest since it can comprise more than 90 % of a component's fatigue life time [1]. Hence, even small improvements in the material's behavior during this period can have a large impact on the overall life time. Since the pioneering work of Pearson [2] in the seventies, numerous authors have studied the growth of SFC in a large variety of metallic materials. This topic has been the subject of review papers and conferences [3,4,5]. One common trend shown by this large amount of work is that SFC growth rates show a very large scatter [6]. Several authors, ([7,8,9]) have suggested that this scatter can be partly explained by the 3D growth of SFC which are in contact with only a few grains (typically between 1 to 5 grains). The orientation and shape of those grains have a direct influence on the local stress field [10]. This in turn, influences the

direction and rate of the local crack growth via the activation of slip systems / plasticity. Therefore, an exhaustive analysis of SFCs would require information on grain shape, grain orientation and crack propagation rate in 3D.

Since the first observations of SFC at the surface of Al fatigue samples by optical microscopy [2], more sophisticated techniques involving beach marking using ink [11] or change in environment [12], serial polishing [13], FIB milling [14] and synchrotron micro tomography [15, 16] have been used to study the relationship between local microstructure and the 3D growth of SFC. All these techniques have merits and disadvantages. Beach marking, for example, has a limited spatial resolution and the method used to mark crack fronts can potentially have an influence on crack growth. Serial polishing provides a higher spatial resolution. The series of micrographs used to reconstruct 3D images of short cracks can be obtained by mechanical milling or by focused ion beam (FIB) machining [17, 18]. In the last case, very high spatial resolution can be obtained allowing to study accurate details of the fracture surface but the maximum volume that can be observed up to now is about $100 \times 30 \times 30$ micrometers³ impeding, for example, the study of a full crack front even in the case of short cracks. Another obvious disadvantage is that those techniques are destructive and can only provide *post mortem* information. Synchrotron radiation X-ray microtomography is a non destructive 3D imaging technique and can be used to circumvent this problem. The technique provides a spatial resolution comparable to optical microscopy (voxel size ~ 1 micrometer) and the use of phase sensitive imaging modes enables detection of cracks with sub-micrometer opening displacements. The evolution of the 3D shape of growing cracks can be obtained in (interrupted) in-situ experiments, however this technique does not provide information on the local grain microstructure along the crack fronts. Grain boundaries can be visualized in some specific material systems via decoration techniques [19] but even in such cases PCT lacks information on the crystallographic orientation which determines the local stress state and plasticity i.e. crack growth.

The correlation between local plastic activity and the growth of SFC has been first investigated via 2D surface observations using electron diffraction techniques such as electron channeling contrast [20] and EBSD [21]. By coupling PCT and EBSD maps obtained *post mortem* it has recently become possible to correlate the growth of a SFC with the local microstructure of an alpha/beta Ti alloy [22]. The experimental task is however challenging as the EBSD maps are obtained by manual serial polishing, leading to a rather large distance between them. Automated serial polishing [23] can be used to obtain serial EBSD maps which provide an accurate 3D description of polycrystalline aggregates (accuracy in vertical slice separation typically 1-2 μm) containing typically several thousand grains. However, to the best of the authors' knowledge this technique has not been used with cracked samples so far. It is likely that the presence of a plastic zone around the crack, the tendency of the crack to be closed by abraded material and the break-out of loose material parts during serial-polishing will remain an issue. To avoid these problems, crystallographic characterization should be carried out prior to the fatigue experiment, with a non destructive technique. This can be achieved thanks to the development of diffraction-based synchrotron imaging techniques such as 'three dimensional X-ray diffraction microscopy' (3DXRD) [24] and 'X-ray diffraction contrast tomography' (DCT) [25,26] which are capable of measuring non-destructively 3D grain shape and orientation in many polycrystalline material systems.

This paper presents a first attempt to combine DCT with «conventional» PCT to investigate the 3D propagation of a SFC *in situ* within a fully characterized polycrystalline aggregate (3D shape of grains

and crystallographic orientations). The experimental method used is described with an emphasis on the methodology developed to extract and visualize the information of interest from the very large datasets obtained. The accuracy of the technique for describing the crack morphology during the fatigue life is discussed. Examples of quantitative parameters that can be extracted from the dataset are given and analyzed in order to show the potential of the technique.

Material and Methods

Material and mechanical testing

The material studied is a metastable beta titanium alloy Tiβ21S (Ti-15Mo-3Nb-3Al-2Si), produced by the Timet company and supplied in the form of a 1.6 mm thick sheet. After heat treatment, (2 hours at 850 ° C in an evacuated quartz tube followed by water quench) the material microstructure consists in grains of the metastable beta phase (bcc) with an average size of 55 μm as determined from DCT. A fatigue sample with a 2 mm long gauge section and a diameter of 600 μm was manufactured by spark erosion cutting. After mechanical polishing of the gauge section using 2400 grit SiC paper and 17 μm SiC paste a wedge-shaped notch (150 μm wide, 2 μm high and 40 μm deep) was inserted in the sample gauge section using focused ion beam machining in order to localize crack initiation as described in Ferrié et al. [27]. The interrupted fatigue tests were performed using a dedicated fatigue machine which can be directly mounted on the synchrotron imaging setup [28]. Constant amplitude tests were performed at room temperature (frequency= 25 Hz) with a tensile cyclic load ranging from 10 to 320 MPa (R = 0.03).

3D grain microstructure characterization by X-ray diffraction contrast tomography

DCT is a synchrotron imaging technique based on Bragg diffraction of grains in a crystalline sample: when a single crystal is rotated in an extended monochromatic X-ray beam it will diffract every time it runs through Bragg diffraction alignment condition with the incoming beam. A detector, larger than the direct beam and closely mounted behind the sample, will capture a part of these diffraction events. In the transmitted beam the intensity diffracted out of the direct beam will be visible as a dark ‘extinction spot’, and the diffracted beam forms a bright ‘diffraction spot’. Assuming kinematical diffraction and negligible orientation and strain gradients within the grain, the intensity distribution inside the extinction and diffraction spots correspond to parallel projections of the 3D grain volume. From the shape and intensity distribution of the diffraction spots, the single crystal’s 3D shape can be reconstructed using Algebraic Reconstruction Techniques (ART) [29]. The orientation of the grain can be inferred from analysis of the diffraction vectors associated to the observed reflections.

DCT applies this principle to polycrystals (figure 1) where an additional algorithm has to be used to identify which diffraction spots belong to which grain. The 3 main steps of the semi-automated sample reconstruction procedure from diffraction spots involve [26]

- Spot matching: Friedel pairs of diffraction spots (hkl and $\bar{h}\bar{k}\bar{l}$ reflection) from the same grain, observed at sample rotation angles ω and $\omega + 180^\circ$ are identified. Each spot pair determines a diffraction vector in the sample coordinate system.

- Indexing: the pairs of diffraction spots are sorted into sets belonging to the same grain. This is done by applying a combination of real space and crystallographic constraints.
- Reconstruction: the individual grain shapes are reconstructed using 3D ART and assembled into the common sample volume.

Figure 1(a), principle of DCT: 360° rotation of a polycrystalline sample in an extended, monochromatic X-ray beam. The detector is larger than the direct beam. Each grain runs through diffraction alignments several times. Grain position, shape and orientation can be reconstructed from the diffraction position and intensity distribution. (b) PCT: 180° rotation of sample in extended, monochromatic, partially coherent X-ray beam. The detector has the same size as the direct beam. Successive tomographic scans are recorded at regular intervals during the interrupted in-situ test.

The DCT experiment was performed at beam line ID11 at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The sample was illuminated by an extended X-ray beam, tuned to an energy of 40 keV by means of a Si 111 double crystal monochromator. The diffraction images were recorded on high resolution detector system (2048x2048 pixels) with an effective pixel size of 1.4 micrometer, positioned at a distance of 5.5 mm from the sample rotation axis (see [26] for technical details). About 80,000 diffraction spots were captured on 7200 images during a sample rotation of 360°. The total scan time was 20 hours.

During reconstruction, each grain is assigned a grain number. The output of DCT is, first, a volume where all voxels belonging to one grain have the value of its grain number (grain map) and, second, a list relating each grain number to a crystallographic orientation. A 560 µm high sub volume of the fatigue sample containing the notch was reconstructed. Nearly 800 grains out of the 1000 characterized grains were completely included in the reconstructed volume.

Data Acquisition: fatigue crack propagation

The PCT experiment was performed at the ESRF, on beam line ID19. This beam line offers a small source size and a long distance between source and sample (150 m) which results in partially coherent illumination at the sample position. Interference fringes at the boundary between low and high electron density objects enhance visibility of objects smaller than the actual image resolution and enable the detection of the crack in regions where the crack opening is below the sub-micron pixel-size [30].

26 propagation stages between 45 k (45,000) cycles (first signs of growth at the notch root detected) and 75.5 k cycles were characterized at a voxel size of 0.7 µm, an energy of 38 keV and a sample-to-detector distance of 44 mm. Scans were taken under maximum load in order to minimize the contact

areas due to crack closure effects and to enhance the accuracy of crack detection. At an exposure time of 1 s, a single scan of 1000 projections during 180 degree rotation took 20 minutes.

Data Processing and Visualization

A general challenge in 3D imaging (4D if time is considered) is that the datasets obtained contain tremendous amounts of information. In our case we deal with a 7 dimensional dataset, since each voxel in the 3D volume contains information on location in the sample (x,y,z) grain orientation (described for example via the three Euler angles, φ_1 , φ_2 , φ_3) and state of crack progression (number of fatigue cycles: N). Developing appropriate ways of visualizing those different aspects of materials properties and mechanical behavior is therefore one of the crucial tasks which is detailed below.

Most of the data processing was done using Matlab[®], its image processing toolbox and the non-commercial Matlab[®] plug-in Dipimage [31]. The PCT datasets were first aligned with the DCT dataset using the notch and the sample outlines as references. The final stage of the crack was segmented by applying sequentially a 3D median filter to reduce noise in the reconstructed image (8 bits coding), a grey value threshold, followed by a manual removal of artifacts. The resultant segmented crack was used to automate the segmentation of all other propagation stages by confining the search for pixels belonging to the crack to this sub volume. Small, unconnected segmentation artifacts were removed using a size filter. The whole information on crack propagation was merged in a single volume, by labeling each stage with its cycle number and sequentially copying them into the same volume, starting with the last stage and ending with the first, always overwriting all voxels belonging to the crack in the earlier stage.

In order to analyze orientation relations between grains and crack, the local normal of the fracture surface needs to be determined. Therefore, the outer layer of the voxels belonging to the crack in the reconstructed volumes is transformed into a surface mesh made of triangles. The mesh was created from the crack in its final stage using the commercial software Avizo[®]. Before segmentation the volume data was oversampled by a factor of 3. That way, less alternating iterations of surface smoothing and mesh simplification had to be carried out in order to eliminate steps arising from the voxelated nature of the original data, and less information about the surface morphology is lost. The final number of triangles was about 1,000,000 for a crack size of 0.14 mm².

5 parameters are assigned to each triangle of the mesh representing the fracture surface by comparing triangle and voxel positions, calculating each triangle's orientation and converting it into the coordinate system of the corresponding grain:

- Vertices: coordinates of the three edge points making up the triangle.
- Propagation stage: the number of fatigue cycles after which this part of the crack appeared. If not all vertices are contained in the same propagation stage it is labeled as belonging to the crack front.

- Grain affiliation: to which grain is the triangle affiliated. If not all triangle edges are contained in a single grain it is labeled as belonging to a grain boundary.
- Physical orientation: surface normal of triangle in the sample coordinate system.
- Crystallographic orientation: surface normal of triangle in the crystal coordinate system of the grain to which the triangle is affiliated.

By assigning a color to each triangle orientation, the local changes in fracture surface orientation can finally be visualized.

In order to measure the local crack growth rate an algorithm was developed that is capable to measure growth rates on cracks with complicated morphologies like branchings. Starting from one crack front and ending at the next one (figure 2a) the algorithm successively finds sets of adjacent triangles and labels them according to which cycle of iteration they were found in (figure 2b). That way, the labels of the last row of triangles before the next crack front (figure 2c) represent the local growth distance. The scaling factor between those label values (number of dilation steps) and the real growth distance da was calibrated at the notch, since it is a flat part where the real distance can be easily measured.

Figure 2: Principle of the algorithm measuring the local crack growth shown on the fracture surface within a single grain. (a) The outlines of the triangles making up the surface mesh are labeled in white. The red lines mark the crack front at given numbers of cycles, the yellow arrow the direction of crack growth. (b) Starting from one crack front and ending at the next the algorithm finds successively arrays of adjacent triangles. (c) The last row of triangles before the next crack front contains the information on local crack growth distance.

Results

1. Voxelated data

Figure 3a shows a 3D rendition of the morphology of the crack at the end of the experiment. The crack which initiates at the FIB notch and grows until about the middle of the sample. On average, one tomographic scan was recorded every 1000 cycles but this number was decreased to 500 to account for the faster growth rate towards the end of the experiment (an animation showing the different stages of crack growth can be viewed as supplementary material linked to the online version of the article). Although the fracture surface appears macroscopically perpendicular to the loading axis, it can be seen from figure 3a that the crack morphology shows pronounced branchings and deviations. This behavior is typical of SFC and most likely due to the presence of grains as the typical scale of deviations (a few tens of micrometers) tend to indicate. This can be investigated in more detail by merging the 3D grain microstructure data (DCT) with those of the growing crack (PCT).

The merged dataset contains detailed 3D information on the evolution of a fatigue crack inside a fully characterized crystalline aggregate. In practice, however, the wealth of information contained by such a dataset is not straight forward to analyze. On figure 3b only part of the grains intersecting the fracture surface have been made visible (see the supplementary on-line video for a detailed view of all grains). As a first approach, figure 3c and d show the crack path on planes clipped within the volume

as they would appear on observations made by sectioning the sample. In some cases strong deviations of the crack are clearly correlated with the presence of grain boundaries (figure 3c) but some deviations can also be observed *within* a grain (e.g. arrow on figure 3d). In order to understand the origins of such intragranular deviations, the evolution of the *whole crack front* within all the grains has to be studied. This is done in the next section.

Figure 3: 3D rendition of the fatigue crack after 75.5 k cycles (the fatigue load is applied vertically) . a) the colors represent the vertical crack position: blue = 0 μm , red = 180 μm . b) 3D rendering of the grains intersecting the fracture surface: some grains have been made transparent to illustrate the crack interactions with the local microstructure. The different colors correspond to different grain orientations. c) and d) «classical» 2D views of the crack path on different planes cut in the bulk of the sample . (See supplementary material on the editor's web page for an animated 3D view of this figure showing the different growth steps of the crack as well as all the grains intersecting the fracture surface). Changes in direction of the crack can be correlated with the presence of grain boundaries (c) but can also be observed within a grain (arrow in d).

2. Meshed data

2.1 Visualization of physical orientations

Figure 4 shows a rendering of the 3D triangular mesh representing the fracture surface after 75.5 k fatigue cycles with a color map representing the orientation of the surface in the sample coordinate system. The good accuracy obtained during alignment of the DCT and the tomography images is shown by the correlation between strong orientation/color changes with the presence of grain boundaries (grains a and b for example). By looking closely at figure 4, however, one can see that the crack can sometimes cross grain boundaries without being deflected (e.g. grains b and c). In some cases changes in the orientation of the fracture surface can also be observed *within* a single grain with a regular pattern of short spatial wavelength (grain d) or on distances comparable to the grain size (grain e). Using the data obtained from DCT, it is possible to further analyze such deflections with respect to the local crystallography; this is done in the next paragraph.

Figure 4: View along the loading axis of the 3D triangular mesh representing the fracture surface. Black lines indicate intersections of the crack with the grain boundaries. The triangle colors represent the orientation of the fracture surface in the sample coordinate system (physical orientation). The color map to the right is the standard stereographic projection of an orientation hemisphere.

2.2 Visualization of crystallographic orientations

Figure 5: View along the loading axis of the 3D triangular mesh which represents the fracture surface. White lines indicate intersections of the crack with the grain boundaries. The triangle colors represent the orientation of the fracture surface in the crystallographic system (crystallographic orientation) as defined on the color map/stereographic triangle on the right. The positions of the slip planes reported for bcc $\{101\}$, $\{112\}$, $\{213\}$ are labeled.

Figure 5 shows a 3D rendering of the crystallographic orientation of the fracture surface. Each triangle has been assigned a colour corresponding to the symmetry related equivalent orientation in the standard triangle of the local crystallographic coordinate system. A striking feature of this figure is that the surface mesh contains many other colors than those corresponding to the bcc «classical» slip planes, i.e. $\{101\}$, $\{112\}$, $\{213\}$ [32]. At the spatial resolution achieved in this study, it appears that the crack is propagating on slip planes only in rare occasions as e.g. in grain e where the fracture surface is closely oriented to a crystallographic $\{101\}$ plane.

3. Chronology of crack growth

Figure 6: 3D rendering of the crack evolution within the sample during the fatigue test. The grey lines indicate intersections of the crack with grain boundaries. *a)* Position of the various crack fronts with a color code varying with the number of fatigue cycles. *b)* Local growth rate da/dN shown with a logarithmic color code.

Figure 6a shows a 3D rendering of the crack development during the fatigue test. It can be seen on this figure that the growth of the crack is nearly continuous throughout the test except for a few places (arrows on figure 6a). The arrows to the left and to the middle mark enclosed areas which get smaller and smaller with each fatigue cycle. As the crack front position is determined automatically on the images, this indicates that the crack was detected later by the algorithm, either because the material was indeed un-cracked in this region, or because the crack was too tightly closed to be efficiently detected. The arrow to the right marks a continuous region which represents an uprising crack branch that covers the parts below. The left side of this branch is a good example for the variance of the crack growth rate which is locally very low compared to the rest of the crack after the same number of cycles.

For a given number of cycles, a range of growth rates can be found along the crack front, especially in the first stages of growth. Parts of the crack front close to the free surfaces tend to grow faster, (especially on the left hand side of the specimen) and some variations can be correlated with the presence of grain boundaries (e.g. grains labeled g, h, i and c). Some grain arrangements seem quite effective in delaying crack growth (e.g. grain j or g). The case of the grains labeled j, k and e on figure 6b is worth being described in detail as it shows how the crack front adapts itself to variation in cracking resistance.

Slow growth rates are observed in grain j with a fracture surface which is not parallel to a specific crystallographic plane (see figure 5). Meanwhile, the crack propagates faster in the neighboring grain k where the crack is orientated close to a $\{112\}$ plane. This induces a rotation of the crack front

towards the right hand side of the figure. When the crack finally enters grain e it therefore comes from the two above mentioned grains (j and k) and the part of the crack front coming from grain k enters grain e along a favorably aligned $\{101\}$ plane and forces the part of the crack front coming from grain j to eventually propagate on that plane. The result of this complex process is an acceleration of crack growth within grain e (figure 6b), when the crack plane changes from non crystallographic to crystallographic. The crack further accelerates when it enters the next grain (grain l) where cracking also occurs on two crystallographic $\{112\}$ and $\{101\}$ planes.

It is worth mentioning that the change in direction observed in grain e (figure 4) and explained in the previous paragraph corresponds to the intragranular change in direction highlighted by an arrow on figure 3. This illustrates clearly why the details of the interactions of short cracks with grain boundaries cannot be fully elucidated unless a 3D view of the crack front and of the grain shape and orientation is obtained.

Figure 7: Average crack growth rate for each individual grain crossed by the crack.

The data shown on figure 6b can be used to produce a map of the average crack growth rate for each grain crossed by the crack, as shown on figure 7. It clearly shows that the crack globally accelerates as it moves away from the notch. For the latest stages of growth observed, the crack front crosses around ten grains but the crack growth rate is still not uniform and varies from 6 nm/cycle to 82 nm/cycle. It also appears, when looking at figure 7 that, for a given crack length, the average crack growth rate on the left hand side of the specimen is higher than on the right hand side.

Discussion

Material choice

The aim of this study was to investigate the interaction of SFCs with grains in 3D. DCT is a particularly well suited technique as it provides the 3D shape and orientation of polycrystalline aggregates in a non destructive way. At the time the fatigue experiment described in this study was performed, the accuracy of 3D grain boundary maps produced by DCT was still unknown. In order to be able to evaluate this uncertainty the metastable beta Ti alloy “Ti 21S” was selected for the present work. In this material a heat treatment can be performed to decorate grain boundaries with alpha (hcp) precipitates. Those precipitates can be visualized in 3D using PCT [33] and can be used to precisely map the position of grain boundaries. A comparison between the shape of grains obtained by DCT and by PCT carried out on the specimen after the fatigue test, showed that the average error in the grain boundary position determined by DCT was of the order of 2-3 micrometers, for an average grain size of 55 micrometers [26]. This accuracy was deemed satisfactory and the high resolution PCT validation data were not used for the analysis carried out here. The inherent, remaining uncertainty of DCT reconstructions can account for small discrepancies between the location of grain boundaries and changes in direction of the crack that were sometimes observed in this work (see for example arrows on figure 4).

A possible complication of the method driven choice of the material system used in this study, particularly relevant in the case of fatigue, is that the high stress levels reached at the tip of fatigue

cracks might trigger stress-induced martensitic transformation (SIMT) which would change the local stress field and complicate interpretation of the local cracking behavior. EBSD analysis on the surface of samples made from the same material before and after fatigue did not reveal any evidence of SIMT at least at the lengthscale investigated (1 micrometer step size). Given the accuracy of the obtained DCT maps, future investigations can focus on simpler material systems with comparable microtexture characteristics (i.e. monophasic, recrystallisation microstructures).

Before being able to address the topic of correlations between local crack growth rate and the fracture surface orientation the accuracy of the representation of the fracture surface in the crystallographic orientation space should be discussed. From figure 5 one can see that many parts of the fracture surface do not correspond to crystallographic slip planes; instead large areas of the fracture surface are parallel (within a few degrees) to $\{001\}$ and $\{111\}$ planes which are at least 19.4 degrees off from any of the slip planes reported for bcc metals [32].

This could be due to measurement errors induced for example by the DCT grain reconstruction, the alignment of DCT and PCT volumes, the segmentation and the smoothing operations. The error in the orientation as measured by DCT is directly linked to the orientation spread *inside* the grain of interest: the current DCT processing route assigns an *average* grain orientation to all voxels belonging to the same grain and neglects the presence of intragranular orientation spreads. For the material used in this study the intragranular orientation spreads are below 0.2 degrees, justifying the use of these average orientation values. The DCT and PCT volumes were aligned using the sample outline and the notch as registration surfaces. The error introduced by a misalignment of the datasets is of order of the pixel size in all directions and its impact on orientation accuracy is expected to be below 0.5°. In several cases, the deflection of the crack coincides well with a grain boundary, as visible in figure 4. This indicates that i) misalignment between the datasets must be quite low and ii) wrong orientation assignments due to inaccuracies of the grain map only affect small regions close to the grain boundaries.

The spatial resolution of the PCT images is of the order of twice the voxel size, i.e. close to 1.5-2 micrometers in our case. The period of undulations of the fracture surface should at least comprise 5 pixels to be visualized on the reconstructed images. It has been shown by various authors [34,35,14] that fracture surface facets in different metallic alloys can be observed at a much finer scale. Therefore the PCT technique used in this work can only show a mesoscopic, average fracture surface. There is however no further degradation of the spatial resolution caused by the meshing of the fracture surface which reproduces with fidelity those crack undulations which are large enough to be visible in the PCT reconstruction (figure 8a).

Figure 8a) Superposition of the crack in grain f (figure 5) after 75.5 k cycles as reconstructed from PCT (grey) with the cross-section through the mesh (color) at the same position with applied crystallographic color coding as shown in figure 5. The PCT reconstruction shows only smooth undulations indicating that sharp kinks might have been lost at the resolution used. The good correlation between crack and mesh shows that no substantial error was introduced by the meshing procedure. The red color of the cross-section through the mesh represents a surface orientation close to $\{001\}$. b) Schematic drawing of the double slip mechanism suggested by Neumann [36] showing how alternating crack propagation on $\{101\}$ planes (green) can result in a macroscopic $\{001\}$ plane (red). c) Common reference system for a) and b).

Compared to the work of Marx et al. [18] who FIB milled a notch along a highly activated slip plane into the sample, the notch used here is perpendicular to the loading axis and has been machined without taking the local microstructure into account. Marx et al. observed stage I cracks growing from the notch along slip planes while the crack measured here mostly doesn't show such clear signs of stage I growth. But the fact that the crack investigated here shows pronounced deflections from the notch plane when emanating from the notch (figure 3,4) indicates an influence of the grain orientation on the crack path. The poorer spatial resolution of the technique used for this study (compared to the FIB method) could explain the absence of clear correlations between fracture surface orientation and slip planes, as detailed below.

Interestingly, the average plane of the crack shown on figure 8a is close to a $\{001\}$ plane, an orientation which has been observed in several places of the fracture surface (red areas on figure 5). Although such a plane has not been reported as a slip plane for the bcc structure, a possible mechanism for its occurrence on the fracture surface might be the mechanism suggested by Neumann [36] where cracking occurs on alternating planes, as shown schematically in figure 8b. As explained before, the resolution employed in the current study may have been insufficient to reveal the presence of finer facets corresponding to a possible crack growth on $\{101\}$ planes.

Following the same idea, three different types of propagation can be distinguished on figure 5 : if the frequency of plane changes is low, a larger surface area appears with a color corresponding to a slip plane (e.g. grain e on figure 5). At an intermediate frequency of plane changes, individual slip events can be visible and appear as stripes with alternating colors (grain d on figure 5). At a high frequency of plane changes, as in the case of grain f, the individual slip events are not resolved and the effective surface orientation is a combination of the normals of the involved slip planes. On figure 5, red and blue parts of the fracture surface could therefore represent crack growth at a high frequency of plane changes while striped areas could result from intermediate frequency crack growth.

SFC growth at very low frequencies of plane changes corresponds to what is often termed single slip in the literature [37]. Double slip corresponds to crack growth at higher frequencies. If our interpretation concerning measured and real fracture surface orientation is correct, then there is a fluent transition between these two growth modes and there is no direct correlation between frequency of plane changes and crack length, at least for the crack lengths investigated in this experiment. This would indicate that there is no fundamental difference between single and double slip. This would support the classification of Düber et al. [38] who assign both growth modes to stage I of SFC growth. But other than Düber et al. we can't draw a clear line between the two modes and therefore suggest to describe the short fatigue growth mode during stage I in terms of frequency of plane changes (e.g. low/middle/high frequency stage I crack growth).

The few areas which appear to be parallel to slip planes in figure 5 are clearly the minority of the facets observed. In some cases such areas are correlated to a higher crack growth rate. The crossing of grains j, k, l and e detailed above is quite illustrative of this effect. When the crack front enters grain e after being delayed in grain j, it clearly accelerates when cracking occurs on a $\{101\}$ plane which becomes the dominant plane for the rest of the grain. That the measured crack growth rate is higher in areas where the measured surface orientation is close to a slip plane could be explained as a matter of

the ratio between real and measured fracture surface area. If the crack changes its planes above the frequency that can be resolved the surface area as measured on the mesh will be below the real fracture surface area. Crack growth rates measured in these areas will be below the real growth rates. For this reason the measured crack growth rates in the areas in figure 5 that are mainly green and purple (i.e. $\{101\}$ and $\{112\}$ slip planes) are in general the highest. On the other hand the overall crack growth rate on the left side of the crack seems to be higher than on the right (figure 6,7) although the left side doesn't show a higher density of areas oriented closely to slip planes (figure 5). This might be either explained by the lack, on the left wing, of pronounced barrier effects due to grain misorientations such as the transition between grain j and e or, by a higher load on the left side due to a slight tilt of the sample in the fatigue machine. After passing the grain boundary the crack re-accelerates but is still retarded compared to the left wing so that its overall growth rate remains smaller.

Quite generally speaking the ability of a grain to slow down a crack (see for example grain j) should be related to the ability of the grain to activate plasticity. This is linked with the local value of the Schmid factor *i.e.* to the local grain orientation and to the local stress tensor. Although the former is known, the latter is more complex to evaluate as the presence of the crack tip and neighbouring grains in this elastically anisotropic material induce a tri-axial stress distribution different from the macroscopically applied uniaxial loading [39]. An illustration of this point can be found in figure 9 which shows the crystallographic normals recorded for grain e (see figure 5). In this grain, it was shown that after a transient state corresponding to the crack entering the grain, cracking occurs mainly on a $\{101\}$ plane with a local acceleration of the crack growth rate. Although figure 9 shows indeed a concentration of normals around two $\{101\}$ planes, it can be seen that these planes do not correspond to the highest Schmid factors if a uniaxial stress state is assumed in this grain. Although the $(0\bar{1}1)$ plane has a maximum value of 0.5, it does not correspond to the (101) plane which is observed.

Figure 9: Pole figure showing the distribution of the crystallographic normals of the fracture surface observed within grain e (see figure 5). Values of the Schmid factors for the different $\{101\}$ planes are given with a color scale indicated on the left, assuming an uniaxial stress state in the grain.

Finite Element Crystal Plasticity (FECP) calculations taking into account the full 3D shape of the crack together with the grain arrangements can in principle be performed to evaluate the local stress state at the crack tip. First calculations have been carried out based on polycrystal volumes containing artificially introduced notches [40] and the incorporation of the complex crack geometry into the numerical model is currently under investigation.

Conclusions

The combination of the non-destructive synchrotron imaging techniques X-Ray Diffraction Contrast Tomography (DCT) and propagation based Phase Contrast Tomography (PCT) is a powerful tool for SFC analysis since it precisely provides all crucial information: grain shape, grain position, grain orientation and crack propagation in 3D. The sample microtexture, the orientation of the fracture surface with respect to crystallographic slip systems, the crack retarding effect of grain misorientations and the local crack growth rate can be studied from combined synchrotron imaging and diffraction experiments as described in this paper. In the current article we focused on the interpretation of the measured fracture surface orientation. Transformation of the crack into a surface mesh and assigning a data structure containing information on grain orientation and crack propagation history to each of the surface elements enables quantitative analysis of various aspects of the crack propagation process. The physical colourcoding reveals correlations between crack deflection and grain boundaries and allows for the identification of crack-retarding microstructure configurations. The crystallographic color coding reveals correlations between crystallographic slip planes and fracture surface orientation as well as local crack growth rates. The spatial resolution achieved in the current study enables identification of microscopic crack facets with a minimum size of about 5 μm . Crack plane changes below this lengthscale can not be resolved and will be assigned an average orientation which may obscure the crystallographic nature of the fracture surface. The combination of the presented methodology with X-ray microscopy observations of the fracture surface on the one hand and crystal plasticity simulations on the other hand provides a powerful framework for future investigations of the physical mechanisms governing the propagation of SFCs. The proposed methodology applies to monophasic materials fulfilling some conditions on sample dimensions, grain size, texture and mosaicity.

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