Identification of active slip mode in a hexagonal material by correlative scanning electron microscopy

X. Xu*, D. Lunt**, R. Thomas, R. Prasath Babu, A. Harte, M. Atkinson, J.Q. da Fonseca, M. Preuss

School of Materials, University of Manchester, Oxford Road, Manchester, M13 9PL, UK

Abstract

Metals with a hexagonal close packed structure can deform by several different slip modes with different Critical Resolved Shear Stresses, which provides a great deal of complexity when considering mechanical performance of Mg, Ti and Zr alloys. Hence, an accurate but also statistically meaningful analysis of active slip systems and their contribution to plasticity is of great importance for the understanding of deformation mechanism. In the present study, a correlative scanning electron microscopy-based method of slip trace analysis has been utilised to provide statistical, accurate information of slip behaviour in a weakly textured Ti–6Al–4V alloy with a plastic strain of ~2%. This is achieved through grain orientation mapping by Electron Backscatter Diffraction and strain mapping by High Resolution Digital Image Correlation. The initial identification of slip mode was performed by comparing the slip trace captured in the high-resolution effective shear strain map with all theoretical slip planes with an angle acceptance criterion of ±5°. Ambiguity in slip mode identification was further resolved using the Relative Displacement Ratio method, which enables the determination of the Burgers vector directly from the displacement data. The correctness of the identified slip modes has been confirmed by detailed dislocation analysis using Bright Field Scanning Transmission Electron Microscopy on thin foils extracted from specific grains employing Focused Ion Beam. This detailed investigation demonstrates the robustness of the slip trace analysis based on grain orientation and high-resolution strain mapping.

1. Introduction

Titanium alloys have been used in a wide range of industrial applications due to their high specific strength, strong fatigue performance and good corrosion resistance [1]. Ti–6Al–4V is a two-phase ($\alpha + \beta$) titanium alloy and is often referred to as the workhorse Ti-alloy of the aerospace industry due to its combination of good mechanical properties and manufacturability [2]. The two-phase nature of Ti–6Al–4V provides the opportunity to tailor the microstructure and mechanical properties for specific applications by varying the thermomechanical processing route. Typical microstructures can range from equiaxed $\alpha$ grains with predominantly $\beta$ at the triple points to a fully lamellar microstructure with long $\alpha$ laths structures embedded in large $\beta$ grains [3].

Deformation in the $\alpha$-titanium phase (hcp crystal structure), which is by far the dominant phase in Ti–6Al–4V, is complex and incorporates slip and often twinning, with the relative tendency for either deformation mode dependent on a range of factors including alloying content, microstructure, texture, temperature and loading conditions [4]. Typically, the easiest slip modes in $\alpha$-titanium have a $<\bar{1}1\bar{2}0>$ slip direction ($\bar{a}$ type) and occur on the prismatic ($10\overline{1}0$) and basal (0001) planes. At room temperature the Critical Resolved Shear Stress (CRSS) is the lowest for prismatic $\bar{a}$ slip compared to a slightly higher value for basal $\bar{a}$ slip [5,6]. Pyramidal slip on the $\{10\overline{1}1\}$ plane has also been reported along the $<1\overline{1}2\overline{3}>$ direction [7], although evidence for this slip mode is comparatively weak. In addition, slip can also occur along the $<1\overline{1}2\overline{3}>$ direction ($\bar{c} + \bar{a}$ type), which happens either on the pyramidal $\{10\overline{1}1\}$ or $(1\overline{1}2\overline{2})$ planes [6,8]. However, the CRSS at room temperature for $\bar{c} + \bar{a}$ type slip has been reported to be between 1.6 and 3.9 times higher than prismatic $\bar{a}$ slip depending on alloying content [4,5,9]. As one would expect, the CRSS for all slip modes decreases with increasing temperature [10], but the ratio of the CRSS for $\bar{c} + \bar{a}$ to $\bar{a}$ slip increases indicating that $\bar{c} + \bar{a}$ becomes relatively more difficult with rising temperature [11].
As the different slip modes are expected to have a particular role in deformation and failure mechanisms in titanium and other alloys dominated by a hcp crystal structure [12–14], the identification and quantification of these slip modes bears a particular importance. Electron Backscatter Diffraction (EBSD) based slip trace analysis [15] is a well-established technique for determining the active slip modes and has been utilised in studies on a range of alloys [12,13,16–26]. The method compares slip traces angles measured from either backscattered electron micrographs [12,13,16–24] or more recently from strain maps recorded using High Resolution Digital Image Correlation (HRDIC) [25,26] with theoretical slip traces angles calculated using the orientation data. Recently, the Heaviside-DIC analysis has been proposed as an effective method of determining the angle of experimental slip traces with high precision as a benefit from an improved tolerance to the local strain discontinuity in the regions adjacent to slip traces [27]. However, due to the high number of possible slip modes in metals with a hcp crystal structure there is often more than one slip system that has a slip trace angle projection similar to the experimental slip trace angle. Further, this method of slip trace analysis does not provide a solution to differentiate between the slip systems sharing the identical slip plane but with different slip directions, e.g. pyramidal $a'/c$ + $a$ slip. In these cases, CRSS and global Schmid factor [13,22] have previously been used as secondary criteria to predict the active slip system. The Schmid factor is the ratio of the applied stress to the component of shear stress on the corresponding slip system [28] and is normally calculated based on the crystallographic orientation information provided by EBSD and a global stress condition. However, this assumes that the stress state within individual grains is identical with the nominal applied stress [13,22], which is not necessarily correct and subsequently does not provide a satisfactory way of determining the active slip system [29,30]. Recently, the Relative Displacement Ratio (RDR) analysis has been suggested as a method for differentiating between slip systems with different slip directions on the same slip planes [31]. Slip produces a displacement step at each slip band. This is a three-dimensional vector, which is equal to the Burgers vector in the crystal reference frame as one slip system is activated only. In the current research, as the HRDIC measurements are conducted in a two-dimensional plane, only the $x$ and $y$ components of this displacement vector are available. The magnitude of these components both depends on the direction and magnitude of slip, whereas the ratio of the two displacement components depends on the direction of the displacement only. Therefore, the direction of slip, and hence the active slip system, can be determined from this ratio, which is termed as the Relative Displacement Ratio (RDR). The RDR analysis employs a direct comparison between the RDR values obtained from experimental measurement and theoretical calculation from HRDIC displacement map and EBSD orientation dataset, respectively. The schematic diagram, as demonstrated in Fig. 1, illustrates the RDR analysis via the comparison between experimental measurement and theoretical calculation. Fig. 1a displays the measurement of experimental RDR value from HRDIC displacement map. In the current case, the experimental RDR value is the ratio of the relative displacements along the $x$ and $y$ directions from one side of the slip band to the other. Due to the scattering of the experimental values measured from the HRDIC displacement map, this ratio is calculated from multiple measurements that are conducted along the slip band as the slope of a line fitted using a linear regression [31]. This value is then compared to the theoretical RDR values for all possible slip systems within the crystal reference frame defined by the orientation measured by using EBSD. In the current research, the theoretical RDR values are determined as the ratio of the $x$ and $y$ displacements after resolving the vector of theoretical slip direction (i.e. the Burgers vector in case of single slip system activity) into the specimen coordinate system, Fig. 1b [31]. The slip direction associated with the targeted slip trace is then determined as the best match is provided between the values obtained by experimental measurement and theoretical calculation.

Transmission Electron Microscopy (TEM) based dislocation analysis enables one to determine the slip system with high precision if carried out with great care. However, due to the complexity of sample preparation and small sampling volume, only a few
grains can be analysed using this method. Diffraction contrast imaging using TEM has been used extensively in previous studies [10,32–34] for detailed analysis of dislocations within slip traces. Dislocations are typically imaged under two-beam conditions with selected g vectors, which enables the determination of the Burgers vectors by the traditional g ⋅ b invisibility criterion [35,36]. The screw/edge characteristic of dislocations can also be determined by comparing the orientation of dislocation lines with the Burgers vectors [36]. Previous TEM analysis in deformed titanium alloys suggests that slip traces based on prismatic a and pyramidal c + a slip are typically planar, whereas basal a slip gives rise to wavy slip traces [37,38]. Further, dislocation analysis within slip traces has revealed that a type dislocations on the prismatic and basal slip planes are predominantly of the screw type [10,39], whereas c + a dislocations on the pyramidal slip planes normally have an edge characteristic [10]. Although there is very little experimental evidence of slip in the a direction on the pyramidal (1011) plane [7], cross-slip of the a type screw dislocations from the prismatic to the pyramidal (1011) planes has been observed in CP-Ti, Ti–6Al–4V [9,10,34,40].

The main aim of the present study is to utilise a correlative microscopy approach through the combination of grain orientation mapping by EBSD and HRDIC analyses of shear strain and the Burgers vector to determine the slip mode associated with slip traces. The validity of such an approach is further confirmed through STEM-based dislocation analysis on Focused Ion Beam (FIB) foils extracted from locations with a known strain condition in individual slip traces.

2. Material and experimental procedure

2.1. Material

The material investigated in this study was Ti–6Al–4V provided by Rolls-Royce plc. The as-received material had been forged and annealed resulting in low levels of macro- and microtextures. To produce a simplified microstructure, a sub-β transus solution heat treatment was applied to the as-received material at a temperature of 950 °C for 2 h followed by controlled cooling at 1 °C/min. The resulting microstructure demonstrates an equiaxed z morphology with an average grain size of ~10 μm and an α volume fraction of ~90%. A low temperature annealing/aging treatment (500 °C for 24 h followed by 1 °C/min cooling) was then carried out, which is known to promote either short range ordering or nanoscale η2 precipitation in Ti–6Al–4V [41].

For the mechanical test, a dog-bone tensile specimen was obtained using Electric Discharge Machining (EDM). The gauge dimensions were 26 mm in length, 3 mm in width and 1 mm in thickness. Preparation of the specimens involved standard grinding, polishing and chemo-mechanical polishing in a solution of 4:1 Oxide Polishing Suspension (OPS) to hydrogen peroxide to provide a surface finish suitable for HRDIC and EBSD mapping analyses.

2.2. Grain orientation mapping

EBSD orientation mapping was performed on the surface of the gauge section in the region that was later imaged before and after deformation to obtain detailed local strain information by means of HRDIC analysis. The orientation mapping was undertaken using an Aztec EBSD system and a Nordlys II detector in an FEI Quanta 650 FEG-SEM operated at an accelerating voltage of 20 kV. EBSD mapping was carried out using a step size of 0.5 μm from an area of 200 × 160 μm². The coordinate systems for EBSD and HRDIC analyses have been defined as demonstrated in Fig. 2 in combination with schematic diagrams illustrating the thin-foil specimens that were extracted from site-specific regions within each grains of interest for detailed investigations by using TEM based dislocation analysis.

2.3. Mechanical testing and HRDIC

A gold speckle pattern was subsequently generated on the polished surface by gold sputtering and using the gold remodelling technique outlined in Ref. [26] for titanium alloys in order to carry out 2D strain mapping at sub grain resolution to enable the identification of individual slip traces. The specimens were deformed under uniaxial tension at a displacement rate of ~0.1 mm/min (i.e. equivalent to a nominal strain rate of ~6 × 10⁻⁵ s⁻¹) using a Kammrath-Weiss 5 kN tension-compression micro-tester. The test was interrupted at a plastic strain of ~2% estimated from the displacement data, which was further confirmed by averaged strain measurement by HRDIC in a smaller area.

Images for HRDIC were collected before and after deformation in Backscatter Electron (BSE) mode using an FEI Quanta 650 Field Emission Gun (FEG) SEM. The details of HRDIC analysis have been reported previously in Ref. [26]. However, as a brief introduction, an array of 4 × 4 backscattered electron micrographs with a single tile width of 29.6 μm, equating to an area of ~80 × 60 μm², was collected before and after deformation in the unloaded condition. The images taken were subsequently stitched together using ImageJ and processed using the commercially available LaVision DaVis 8 [42] software to generate full-field displacement maps, which were subsequently differentiated to provide the strain field. The local displacement was calculated by applying an FFT cross correlation relative to the image prior to deformation with a final interrogation window size of 8 × 8 pixels², which equates to a spatial resolution to 115 × 115 nm².

Fig. 2. A schematic diagram defining the coordinate systems used for EBSD, HRDIC and STEM analyses in the specimen coordinate system. Schematic diagrams (grey) are also included to illustrate the thin-foil specimens extracted from each individual grains of interest with foil surfaces perpendicular to the slip traces observed on the plane of EBSD/HRDIC analysis.
2.4. EBSD-based slip trace analysis

The active slip modes in the selected grains were predicted using the EBSD-based slip trace analysis technique, where the slip traces obtained by HRDIC were compared with all theoretical slip planes using the orientation data for each individual grain. The 24 slip systems considered were basal $a$(3), prismatic $a$(3), pyramidal $a$(6) and pyramidal 1st order $c+a$(12). The theoretical slip trace angle projections for all the slip systems were calculated by firstly extracting the crystal orientations (Euler angles) from EBSD dataset using a MATLAB® script. The average orientations of each grain were then used as input to give theoretical slip trace angles. The experimental measurements of the slip traces were determined using the open source imageJ software. For each grain, the slip trace angle was calculated by averaging 5 neighbouring slip traces that appear to be parallel to minimise the effects of non-planar features within the traces. The active slip system was then predicted by comparing all the theoretical slip trace angles with the experimentally measured value using an acceptance criterion of $\pm 5^\circ$, which can be considered a conservative approach but has been shown to yield the highest proportion of unambiguous solutions [26,43].

2.5. Relative Displacement Ratio (RDR) analysis

Whilst HRDIC analysis is conventionally used to determine strain maps, in the present case the EBSD/HRDIC analysis was extended to determine the direction of the Burgers vector through a Relative Displacement Ratio (RDR) analysis, which verifies the previously predicted slip mode or further identifies the slip mode when EBSD-based slip trace analysis has failed to provide an unambiguous answer.

The principle of the RDR analysis is based on each possible Burgers vector associated with a slip trace resulting in a characteristic displacement gradient across the slip trace, which can be determined using the crystallographic information from EBSD and the displacement maps from HRDIC. Fig. 3 provides a schematic diagram of the RDR process and demonstrates that the present HRDIC analysis provides sufficiently high spatial resolution to clearly describe slip traces, as demonstrated in Figs. 3a–1. To measure the displacement gradient across a slip trace with high precision, the slip trace was firstly identified by manually selecting a point at either end of the slip trace but away from the grain boundary, Figs. 3a–1. The selection of the data points used for RDR measurement was then automated by selecting equally spaced central points along the line and then a point was further placed on either side of this point in the perpendicular direction. For each line of the 3 points selected, the $u$ and $v$ displacements are centred by subtracting the average $u$ and $v$ values for each line, $u_{av}$ and $v_{av}$, hence giving the values of $(u-u_{av})$ and $(v-v_{av})$ for each point. The corresponding displacement maps for the two principal directions, $u$ and $v$ as illustrated in Fig. 2, are shown in Figs. 3a–2 and 3a–3, which indicate a change in the displacements from one side of the slip trace to the other. The experimental RDR value is then calculated for each point from a scatter plot of the $u$ versus $v$ centred values as the gradient of the linear regression. Figs. 3a–4, Fig. 3b demonstrates the calculation of theoretical RDR values as the ratio between the components of each theoretical slip direction (i.e. $3 \ a$, $6 \ c + a$) resolved along the principal directions of the specimen coordinate system, RD and TD as illustrated in Fig. 2. The slip direction associated with the targeted experimental slip trace was determined as the best match achieved between the theoretical and experimental RDR values (e.g. $\mathbf{a}_1$ as in the exemplary case demonstrated in Fig. 3b).

In the present work, the RDR analysis was conducted on nine sets of slip traces within five different grains. Four slip traces were

![Fig. 3. A schematic diagram of the RDR process for (a) experimental measurement and (b) theoretical calculations. (a-1) HRDIC strain map demonstrates the slip traces in the area of interest, where (a-2) horizontal ($u$) and (a-3) vertical ($v$) displacement maps were extracted for the measurement of experimental RDR value ($\text{RDR}_e$) by using linear regression, (a-4). HRDIC was conducted using an interrogation window size of 115 × 115 nm$^2$ to enable a clear visualisation of slip traces. The theoretical RDR values ($\text{RDR}_t$) were obtained from (b-1) EBSD orientation data as the ratio between the $x$ (RD) and $y$ (TD) components of each theoretical slip directions, as demonstrated in (b-2) to (b-4). The $\mathbf{a}_1$ direction is determined as the slip direction for experimental slip traces as the best match is achieved between RDR$_e$ and RDR.$]
selected for the measurement of RDR value within each grain of interest. With the resolution achieved here, the RDR analysis showed an average R-squared confidence value of 0.90 with a minimum value of 0.86 for all slip traces, which indicates a high reliability of the analysis [31]. A detailed comparison between theoretical and experimental slip traces revealed a considerably lower level of mismatch between experimental and the closest theoretical solutions ranges from 0.004 to 0.690, whereas the minimum and the maximum differences of the next nearest solutions are 0.38 and 1.02, respectively. The average difference between experimental and the closest theoretical solutions was further obtained at 0.24 as compared to an average difference of 0.72 for the next nearest solutions.

2.6. Focussed ion beam (FIB) lift out

Electron-transparent foils were prepared from site-specific locations within the region analysed by EBSD and HRDIC using an FEI Quanta 3 D FIB/FG-SEM that is equipped with an Omniprobe manipulator. From each grain of interest, a thin-foil specimen was prepared by using the conventional lift-out technique [44]. The thin-foil specimens were extracted so that the plane traces are perpendicular to the foil surface as illustrated in Fig. 2. Subsequently, the foils were mounted to a chevron post to minimise the bending of specimens [44]. The thinning of thin-foil specimen was conducted initially at an accelerating voltage of 30 kV with a minimum beam current of 0.1 nA followed by a final low-voltage cleaning at 5 kV with a beam current of 70 pA.

2.7. Scanning Transmission Electron Microscopy (STEM)

Verification of the active slip systems predicted using the extended EBSD/HRDIC analysis was conducted using detailed (S) TEM analysis of the dislocation structure for individual grains. The FIB-extracted foils were first examined in an FEI G2 20 Transmission Electron Microscope (TEM) equipped with a LaB6 filament using conventional BF-TEM to provide an overview of each thin-foil specimen. This was followed by more detailed analysis utilising an FEI Talos F200A FEG S/TEM operated at 200 kV in STEM mode in order to reduce contrast variations [45,46] while employing a two-beam condition approach that facilitates the determination of the Burgers vectors by the invisibility criterion [32,46–48]. BF-STEM micrographs were collected from a series of selected zone axes to determine the habit planes of the dislocations that were imaged edge-on.

3. Results

3.1. EBSD orientation mapping and slip trace analysis

Fig. 4 compares the grain orientation map in an inverse pole figure mode (a) with the effective shear strain map (HRDIC) of the same region (b) and a map illustrating the α and β phases (c) including the solutions proposed by the EBSD-based slip trace analysis in combination with the RDR analysis for the grains that were later used for (S)TEM analysis. While the region examined here is quite small, the grain orientation map, Fig. 4a, displays grains of various crystallographic orientations. The HRDIC results are displayed as maps of effective shear strain, Fig. 4b, as this component takes into account all the in-plane strain tensors [26,49]. It can be observed that some grains exhibit intense strain localisation with planar slip traces exhibiting a maximum level of shear strain at ~35%, while others display more diffuse slip with the shear strain within slip traces of less than 10%. Occasionally, slip traces were found to extend across the α grain boundaries but most of the slip traces were constrained within single α grains.

EBSD-based slip trace analysis was conducted in combination with the RDR analysis on Grain 1 - 5 as indicated in Fig. 4c, which revealed that unambiguous slip trace solutions always correlated with either prismatic α slip (e.g. Grains 2 and 5) or basal α slip (e.g. Grains 1, 2 and 3). The RDR analysis further confirmed the above
observations. In addition, slip traces giving multiple solutions were found in Grains 3, 4 and 5. In Grain 3 a limited number of slip traces that had bifurcated could be matched with either basal $\bar{a}$, prismatic $\bar{a}$ or pyramidal $\bar{a}/\bar{c} + \bar{a}$. The RDR analysis confirmed an $\bar{a}$ direction and therefore pyramidal $\bar{c}/\bar{c} + \bar{a}$ could be excluded. In Grain 4, slip traces were matched with prismatic $\bar{a}$ and pyramidal $\bar{a}/\bar{c} + \bar{a}$ based on EBSD analysis but the RDR analysis confirmed a unique $\bar{a}$ direction on the prismatic slip plane. In Grain 5, slip traces containing the segments that appeared to match with the pyramidal $\bar{a}/\bar{c} + \bar{a}$ slip plane were identified in limited number of cases based on the angle acceptance criterion. These segments originated from the primary slip traces identified as prismatic $\bar{a}$ and the segments were shown to retain the same $\bar{a}$ Burgers vector from the RDR analysis.

3.2. Comparisons between slip trace analysis and STEM analysis

3.2.1. Grain 1 - (predicted basal $\bar{a}$ slip)

Fig. 5 displays an effective shear strain map with accompanying schematic diagrams of the slip trace angle projections for all possible slip modes for Grain 1. The configuration of slip traces within this grain is relatively simple, with the slip traces typically planar and aligned at an angle of $\approx 72^\circ$ from the loading direction (Fig. 5a). According to the conventional EBSD-based slip trace analysis, the active slip system is predicted to be basal $\bar{a}$ slip, with a Schmid factor of approximately 0.33 for two of the possible $\bar{a}$ directions and 0.01 for the other (Fig. 5b). The RDR analysis identified an $\bar{a}$ direction Burgers vector with an experimental RDR value of $-0.36$ compared to a theoretical value.
of −0.40. The identified ã direction is associated with a Schmid factor of 0.33.

Fig. 6 shows a BF-TEM micrograph providing an overview of the thin-foil specimen that was extracted from Grain 1 from the normal direction to foil surface. The white circle indicates the region where BF-STEM micrographs shown in (b, c and d) were collected. The BF-STEM micrographs that were collected under various electron beam directions (b.d.) under each imaging condition. The insets in the top right illustrate the orientations of foil surface (grey) and the basal (0001) plane (red) under each imaging condition. The insets in the bottom right illustrate the Kikuchi patterns. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

3.2.2. Grain 2 - (predicted prismatic ã and basal ã slip)

Fig. 7 shows the effective shear strain map with accompanying schematic diagrams of the slip trace angle projections for all slip domains for Grain 2. Slip traces oriented at different angles were observed in Grain 2, suggesting the operation of multiple slip systems. Measurement of slip trace angle reveals that the slip traces are typically aligned at −45° and −80° from the principal stress direction (Fig. 7a). The EBSD-based slip trace analysis predicts that the slip traces at −45° and −80° are basal ã slip and prismatic ã slip, respectively. The ã direction of the slip traces was confirmed from the RDR analysis for the slip traces at −80° with agreement between the measured and theoretical values at −0.84 and −0.77, respectively. The RDR analysis is not conclusive for the 45° slip traces, as the measured value of 1.47 lies between two possible ã type directions with RDR values of −2.52 and −0.77 that have Schmid factors of 0.45 and 0.32 for basal slip, respectively. However, as the calculated RDR value was averaged over 4 slip traces, detailed analysis was also carried for each individual trace to assess whether individual traces had a particular slip direction for basal slip. This analysis suggested a combination of the two basal slip directions, as the RDR value along all the traces fluctuated between the two theoretical RDR values.

Fig. 8 displays a BF-TEM micrograph providing an overview of the thin-foil specimen that was extracted from the region as labelled in Fig. 7a and the BF-STEM micrographs that were collected for detailed dislocation analysis. The normal direction to the foil surface was −30° from the [2110] zone axis. The most distinct dislocations are aligned along the basal (0001) plane when observed from the [2110] (Fig. 8c) and the [1010] (Fig. 8d) zone axes. There are also dislocations aligned along the prismatic (0110) slip plane when observed from the [2110] (Fig. 8c) and the [211T] (Fig. 8e) zone axes. The two-beam condition tilting towards [0002] reflection (Fig. 8f) excluded the presence of ã + ã dislocations. Hence, the STEM dislocation analysis does confirm the two slip modes suggested by slip trace analysis.
Fig. 7. Grain 2 - (a) effective shear strain map and (b) the schematic diagrams demonstrating the projections for all possible slip systems compared with the experimental slip traces observed in (a). The white box on the strain map (a) indicates the location where a thin-foil specimen was extracted. The black dashed lines/shadows show the ±5° range from the white dashed experimentally measured slip trace angle in the insets in (a) and schematic diagrams in (b). The experimentally measured RDR value (RDR_e, inset) and the theoretical RDR value of matched solution (RDR_t, schematics) are denoted in red and blue as the basal and prismatic slip planes matched with the slip traces at 45° and 80°, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
Fig. 8. (a) A BF-TEM micrograph showing an overview of the thin-foil specimen that was extracted from Grain 2 from the normal direction to foil surface. The white circle indicates the region where detailed BF-STEM micrographs shown in (c–f) were collected. (b) A schematic diagram illustrating the relative orientations of the [2\(\bar{1}\)10], the [10\(\bar{1}\)0] and the [2\(\bar{1}\)1\(\bar{1}\)] zone axes in a Kikuchi map of hexagonal titanium alloys. (c–f) The BF-STEM micrographs that were collected under various electron beam directions (b.d.). The insets in the top right illustrate the orientations of foil surface (grey), the basal (0001) (red) and the prismatic (0\(\bar{1}\)10) (blue) slip planes under each imaging condition. The insets in the bottom right illustrate the Kikuchi patterns. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
3.2.3. Grain 3 - (predicted basal $\overline{a}$ slip, bifurcated segments have multiple solutions)

Fig. 9 shows an effective shear strain map with accompanying schematic diagrams of the slip trace angle projections for all possible slip modes for Grain 3. The slip traces within Grain 3 are predominantly aligned at an angle of ~118° from the loading direction with bifurcation at ~123° observed with some traces (Fig. 9a). The conventional slip trace analysis indicates that the slip traces at ~118° match basal $\overline{a}$ slip, whereas the bifurcated slip traces at ~123° match basal $\overline{a}$, prismatic $\overline{a}$, pyramidal $\overline{a}$ and/or pyramidal $\overline{c}$ + $\overline{a}$ slip (Fig. 9b). The RDR analysis confirmed the prediction for the slip traces at ~118° as basal $\overline{a}$ slip with the 2nd highest Schmid factor of 0.34 and that the same $\overline{a}$ direction applies for the slip traces at ~123°.

Fig. 10 shows a BF-TEM micrograph providing an overview of the thin-foil specimen that was prepared from the region as labelled in Fig. 9a and the BF-STEM micrographs showing the details of dislocations from various electron beam directions. The normal direction to the foil surface is ~15° from the [1121] zone axis (Fig. 10a), and the speckle-like features in (c-f) are the artefacts produced during the preparation of thin-foil specimen. The dislocations that are aligned along the basal (0001) plane were observed from the [11\overline{2}0] (Fig. 10c) and [1\overline{0}0\overline{0}] (Fig. 10d) zone axes. There are also dislocations aligned along the prismatic (1T00) plane when observed from the [11\overline{2}0] (Fig. 10c) and the [11\overline{2}1] (Fig. 10e) zone axes. The observed dislocations on the prismatic and the basal planes were both determined as the $\overline{a}$ type dislocations under an appropriate electron beam direction [47]. Therefore, the prediction of the basal $\overline{a}$ slip for the predominant slip traces at ~118° has been confirmed by the STEM analysis, and the minor slip traces that has been predicted to have an $\overline{a}$ type slip direction by the RDR analysis have been confirmed to be the prismatic $\overline{a}$ slip.

3.2.4. Grain 4 - (multiple solutions: prismatic $\overline{a}$/pyramidal $\overline{a}/\overline{c}$ + $\overline{a}$ slip, RDR suggests prismatic $\overline{a}$)

Fig. 11 shows the effective shear strain map with accompanying schematic diagrams of the slip trace angle projections for all slip domains for Grain 4 indicating slip traces being aligned at ~82° from the principal stress direction. The EBSD/HRDIC based slip trace analysis predicts that the experimental slip traces at ~82° are either
Fig. 10. (a) A BF-TEM micrograph showing an overview of the thin-foil specimen that was extracted from Grain 3 from the normal direction to foil surface. The white circle indicates the region where BF-STEM micrographs shown in (c–f) were collected. (b) A picture illustrating the relative orientations of the [11\(\overline{2}\)0], the [10\(\overline{1}\)0] and the [11\(\overline{2}\)1] zone axes in a Kikuchi map of hexagonal titanium alloys. (c–f) The BF-STEM micrographs that were collected under various electron beam directions (b.d.). The insets in the top right illustrate the orientations of foil surface (grey), the basal (0001) (red) and the prismatic (1\(\overline{1}\)00) (blue) slip planes under each imaging condition. The insets in the bottom right illustrate the Kikuchi patterns. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
prismatic $\bar{a}$, pyramidal $\bar{a}$ and/or pyramidal $\bar{c} + \bar{a}$ slip. Further analysis using the RDR method shows that the measured value of \(-1.37\) is the matched by a theoretical value of \(-1.71\) for the prismatic $\bar{a}$ slip.

Fig. 12 displays a BF-TEM micrograph providing an overview of the foil that was extracted from the region as labelled in Fig. 11a and the BF-STEM micrographs showing details of the dislocations within slip traces. The normal direction of the foil surface is \(-13^\circ\) from the [1123] zone axis (Fig. 12a). The dislocations that are aligned on the prismatic (1100) slip plane were observed from the [0001] (Fig. 12c) and the [TT23] (Fig. 12d) zone axes. These observed dislocations became invisible when reflections on the (T101) (Fig. 12e) and the (1T00) (Fig. 12f) planes were selected, indicating a Burgers vector of [TT20]. Therefore, the use of the EBSD/HRDIC based slip trace analysis in combination with the RDR analysis predicted the correct slip system, as confirmed by the STEM analysis.

3.2.5. Grain 5 - (predicted prismatic $\bar{a}$ slip, RDR suggests $\bar{a}$ direction)

Fig. 13 shows an effective shear strain map with accompanying schematic diagrams of the slip trace angle projections for all possible slip modes for Grain 5. Slip traces with the identical projections at \(-58^\circ\) and \(-108^\circ\) from the principal stress direction were observed (Fig. 13a). Both types of slip traces could be matched with the prismatic $\bar{a}$ slip (Fig. 13b). The RDR analysis further confirmed that both types of slip trace have an $\bar{a}$ direction with a measured value of \(-1.23\) (58°) and 0.81 (108°) matched to a theoretical value of \(-0.98\) and 0.52, respectively. In addition, two slip traces at 58° have been shown to contain minor segments that bifurcate at \(-71^\circ\) (Fig. 13a). In this case the trace segments were too short for a reliable analysis but an initial analysis suggested pyramidal $\bar{a}$/$\bar{c} + \bar{a}$ slip, while the RDR analysis indicated the same $\bar{a}$ direction as the predominant slip traces with a Schmid factor of 0.45 for the corresponding pyramidal slip system. The identical $\bar{a}$ type slip directions between the trace segments at \(-71^\circ\) and the primary...
Fig. 12. (a) A BF-TEM micrograph showing an overview of the thin-foil specimen that was extracted from Grain 4 from the normal direction to foil surface. The white circle indicates the region where BF-STEM micrographs shown in (c–f) were collected. (b) A picture illustrating the relative orientations of the [0001], the [1123] and the [0111] zone axes in a Kikuchi map of hexagonal titanium alloys. (c–f) The BF-STEM micrographs that were collected under various electron beam directions (b.d.). The insets in the top right illustrate the orientations of foil surface (grey) and the prismatic (1100) (blue) slip plane under each imaging condition. The insets in the bottom right illustrate the Kikuchi patterns. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
Fig. 13. Grain 5 - (a) effective shear strain map and (b) the schematic diagrams demonstrating the projections for all possible slip systems compared with the experimental slip traces observed in (a). The white box on the strain map (a) indicates the location where a thin-foil specimen was extracted. The black dashed lines/shadows show the ±5° range from the white dashed experimentally measured slip trace angle in the insets in (a) and schematic diagrams in (b). The experimentally measured RDR value (RDR_e, inset) and the theoretical RDR value of matched solution (RDR_t, schematics) are denoted in blue and yellow as the prismatic and pyramidal slip planes matched with the slip traces at 58°/108° and 71°, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)
traces at ~58° suggests that the bifurcation occurs upon the cross-slip of dislocations between the prismatic and pyramidal slip planes.

Fig. 14 displays a BF-TEM micrograph providing an overview of the thin-foil specimen that was extracted from the region labelled in Fig. 13a and BF-STEM micrographs showing the details of dislocations. The normal direction to the foil surface is ~8° from the [1211] zone axis (Fig. 14a). When observed from the [1213] (Fig. 14c) and the [2423] (Fig. 14e) zone axes, the dislocations that are aligned on the prismatic ([010]) plane were observed. From an electron beam direction close to the [1213] and the [2423] zone axes, dislocations that are aligned on the prismatic ([100]) plane were observed. The dislocations on other slip planes, including the pyramidal [1010] slip plane, were not clearly observed to be aligned when the specimen was tilted to other accessible orientations (e.g. the [0T11] zone axis, Fig. 14g). Hence, all observed dislocations were 4 type dislocations (Fig. 14h), which supports the result of the RDR analysis. Therefore, the STEM analysis has confirmed the prediction of the extended slip trace analysis for the amount of dislocation from 58° and 108°, whereas only the prediction of slip direction made by the RDR analysis was also supported by the STEM analysis for the minor slip traces at 71°.

4. Discussion

Slip trace analysis based on grain orientation mapping by EBSD combined with high resolution strain and Burgers vector direction analysis using HRDIC has been compared with STEM-based dislocation analysis to assess the accuracy of slip trace analysis when studying a material with a high number of possible slip systems, in the present case Ti–6Al–4V. Table 1 compares the slip system predictions using the traditional EBSD-based slip trace analysis employing a ±5° angular acceptance criterion with and without the addition of the RDR analysis to the slip systems determined from BF-STEM based analysis. Excellent agreement has been found for the majority of slip traces, particularly when enhancing the conventional EBSD-based slip trace analysis by identifying the Burgers vector direction through the RDR analysis. While there might be cases in which the extended slip trace analysis only provides unambiguous information about direction of slip instead of the full slip system (Grain 3), slip trace analysis is advantageous in its capability of performing statistically sound analysis of slip modes as compared to STEM-based dislocation analysis that is limited by time-consuming preparation and small sampling volume. Further, the HRDIC analysis in combination with EBSD provides not only the number of slip traces but also the shear strain associated with each individual slip trace, which enables validation of plasticity models as reported in recent work [50,51].

All of the grains analysed in this study were favourably oriented for either prismatic or basal slip, which are typically the easiest slip modes to activate in titanium alloys due to their comparatively low CRSS compared to pyramidal slip [4,9,52]. In addition, typical textures and loading directions in Ti–6Al–4V often give rise to relatively high Schmid factor values (>0.3) for prismatic slip and basal slip. This means that the likelihood of observing pyramidal slip in the present work is very small. In the grains studied here, only short segments of pyramidal slip traces were found in Grain 5 and they were...
confirmed to be the $\bar{a}$ slip by the RDR analysis (Fig. 13 and Table 1). Although the experimental observation of pyramidal $a$ slip is limited in the literature, the cross-slip of dislocations from the prismatic to the pyramidal slip planes has been reported [4,34,40,53] and is consistent with the observation of the pyramidal $a$ slip traces appeared as segments belonging to primary slip traces on the prismatic slip plane (Fig. 13).

Previous work has highlighted the challenge of unambiguously identifying slip modes when undertaking conventional slip trace analysis in metals with a hcp crystal structure. While in weakly textured Ti–6Al–4V only 10% of the investigated slip traces provided multiple solution [26], a Ti–6Al–4V containing large macrorozones preferentially orientated for prismatic slip during tensile loading displayed 20–30% slip traces failing to give an unambiguous slip mode determination [43]. In a Zr-alloy (Zircaloy–4) with a split basal texture loaded along the rolling direction the fraction of slip mode ambiguity even raised to 40% [34]. It is clear from such comparison that the crystallographic texture plays an important role in slip traces being nearly parallel for different slip modes. An important aspect of the present slip trace analysis is the extension to include the determination of the Burgers vector direction (RDR analysis), which provides further confidence in the selected slip mode or can remove ambiguity. This was observed in Grain 4 where the majority of slip traces were matched with multiple solutions containing different $a$ and $\bar{c}+\bar{a}$ slip directions, whilst the RDR analysis provided a unique $\bar{a}$ slip direction enabling a unique slip system to be suggested. This has demonstrated that slip trace analysis is significantly enhanced by including the RDR analysis, providing a reliable toolbox to quantitatively determine slip modes in a polycrystalline metal with a hcp crystal structure.

An interesting aspect of the RDR analysis is that it enables one to distinguish between the three possible slip directions for basal slip despite having the same slip trace. This has highlighted that the activated slip system cannot be inferred from a Schmid factor analysis, which only considers the global stress direction. Example here is Grain 3, where the RDR analysis identifies basal slip systems associated with the 2nd highest Schmid factor. This finding is of course not surprising as the elastic and plastic anisotropy of individual grains create complex stress conditions at the crystal level, particularly in weakly textured material, which are not taken into account during a Schmid factor analysis. It is also worth mentioning that exact knowledge of slip direction becomes particularly important when trying to understand grain neighbourhood effects and possible slip transfer across grain boundaries in a polycrystalline aggregate.

5. Conclusions

Correlative microscopy combining EBSD grain orientation mapping with HRDIC analysis and BF-STEM diffraction contrast imaging has confirmed the validity of a combined method of EBSD/HRDIC based slip trace including RDR analysis. In particular, BF-STEM analysis distinguished prismatic $\bar{a}$ and basal $\bar{a}$ slip when those were unambiguously determined from the same slip traces based on the EBSD/HRDIC based analysis. In cases where the orientation/strain-based analysis failed to provide an unambiguous answer, additional RDR analysis identified the slip mode that was later confirmed by the BF-STEM dislocation analysis.

The combined method of the EBSD/HRDIC based slip trace including RDR analysis has demonstrated the capability of determining active slip system within each individual slip trace with sufficient accuracy. This also potentially facilitates the verification of the numerical crystal plasticity modelling as an essential step to understand detailed plastic deformation mechanism within complex hexagonal alloys.

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