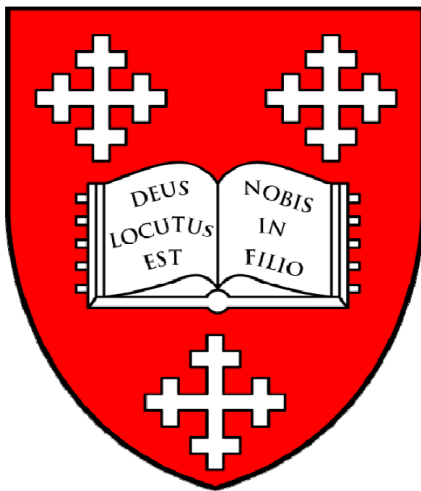


**The interactions between slip band, deformation
twins and grain boundaries in commercial purity
titanium**

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Abstract

Title: The interactions between slip band, deformation twins and grain boundaries in commercial purity titanium

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This thesis apply High Resolution Electron Back Scatter Diffraction (HR-EBSD) technique to a variety of microstructure features and their interactions in pure h.c.p polycrystals. By correlating high quality Kikuchi patterns with a reference pattern, the relative state and distribution of strain, stress, and geometrically necessary dislocation (GND) density can be obtained with high strain sensitivity (10^{-4}) and angular resolution (10^{-4} radian). This technique is companied by a further investigation of subsurface features using Differential Aperture X-ray Micro-diffraction (DAXM) technique. The two technique have shown excellent agreement in capturing the magnitude and distribution of stress and GND.

Stress field and GND distribution induced by slip band and grain boundary interactions, including blocked slip band with no observable slip transfer in SEM and slip transfer, were characterised. It was found that some blocked slip bands lead to high and localised stress concentration in the neighbouring grain while others did not, and no stress concentration were correlated with transferred slip bands. These three categories of interactions were rationalised using a slip transfer criteria (called LRB criteria) by investigating the geometric alignments between the impinging slip system and all possible slip systems in the neighbouring grain. The level of stress concentration were quantified into a

stress intensity factor K , following the Frank, Eshelby, and Nabarro (FEN) model. It was found that the level of stress intensity correlates well with the number of dislocations within the pile up plane.

The slip band and grain boundary interaction case that led to the highest magnitude of stress intensity factor was further investigated using DAXM experiments. The 3D data set informed us additional information hidden below the sample surface. The distribution of stress concentration in 3D is a ribbon conforming to the line of intersection between slip plane and grain boundary. Stress intensity factor calculation along this ribbon have shown large variations which led to a concern that sometimes 2D results might not be conclusive. For example, if damage is observed in sample surface, there is a possibility that large populations of damage already exist below sample surface as a result of the stress fluctuations. The level of stress concentration and distribution measured by both HR-EBSD and DAXM agree with each other and 3D lattice rotation gradient used in DAXM GND calculation was found to affect the range of GND distribution and how fast it decays away from grain boundary.

Twinning is a deformation mechanism in HCP metal that is equally important as dislocation slip. The stress concentrations associated with twin propagation, approaching grain boundary, and thickening were characterised using HR-EBSD, from which the calculated stress tensor were used to generate a local Schmid factor (LSF) map. It was found that during twin propagation, local positive shear provides a favourable LSF condition that promote twin tip extension while suppress it from thicken. When twin tip is approaching the grain boundary, the positive shear stress field no longer favour twin propagation, a

narrow positive LSF field still exist at the tip of twin, promoting it to grow thick. During propagation and thickening process, the LSF seem to only affect the tip of twins and therefore these processes are possibly tip controlled.

Publications and Conferences

Publications:

Y Guo, TB Britton, AJ Wilkinson, ***Slip band and grain boundary interactions in commercial purity titanium***. Acta Materialia (2014) vol. 76 127-134

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Y Guo, D Collins, E Tarleton, F Hoffmann, J Tischler, W Liu, R Xu, AJ Wilkinson, TB Britton, ***3D Measurements of Stress Fields near Grain Boundaries: Exploring Blocked Arrays of Dislocations***, TMS 2015 (Orlando, USA)

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Chapter 1 Introduction

Structural materials are widely used in a diverse range of applications, including energy, transport, and the built environment. Metals are often used as they are strong, tough, and readily formable. Increasing performance of these metals, typically through improved specific strength keeps alloys competitive and opens up new markets for exploitation. In transport, a reduction in weight leads to many important cost and environmental savings, such as a reduction in CO₂ emissions.

In practice, improved properties arise from increased microstructure control and understanding of the role of specific microstructural features, such as grain boundaries, and how defects such as dislocations interact with these features to control strength, ductility and failure. Furthermore, grain boundaries and deformation twinning impart heterogeneous mechanical responses at the local scale which can be critical in damage nucleation processes. If we can understand these processes, it is then possible to engineer materials microstructures for stronger, safer, and more sustainable applications.

The ability to capture full strain tensor at micro-scale is critical to understand the above mentioned problems. This was accomplished by using the state of art electron and X-ray based technique and the results will be presented in the following chapters.

Chapter 2 Literature Review

2.1 Introduction to titanium

2.1.1 History and application:

Titanium is the fourth most abundant structural metal present in earth's crust in the forms of ilmenite (FeTiO_3) and rutile (TiO_2). Early attempts to isolate the metal from these ores involved using TiCl_4 as an intermittent step but the strong tendency of this metal to react with oxygen and nitrogen makes it difficult to produce high purity titanium. The first commercially attractive process to isolate titanium was developed by Kroll in Luxembourg, which involved the reduction of titanium tetrachloride with magnesium in an inert gas atmosphere. The product is called 'titanium sponge' because of its porous and spongy appearance. This Kroll process remains largely unchanged today, and is the most common method of producing titanium [1].

Titanium alloys are traditionally and largely used in airframes and within aero engines for its superior strength to weight ratio. Approximately 10% of the Boeing 777 airframe weight is titanium and it is the first aircraft on which Ti-6Al-4V alloy was outweighed by Ti-10V-2Fe-3Al as landing gear because of the high strength Ti-10-2-3 offers. As a comparison, the Airbus family has titanium usage of only 4-5% and virtually no β type titanium alloys are used [1-3]. Modern aero engines have about 25 wt% of titanium mainly used in fan and compressor sections for disks and blades which have operation temperature tolerance up to 500°C . Although some modern aero engines replace Ti-6-4 for polymer composite as fan blade material, a large number of titanium alloy

components are still used as is shown (Figure 2.1.1) in the following picture of Rolls Royce Trent engine:

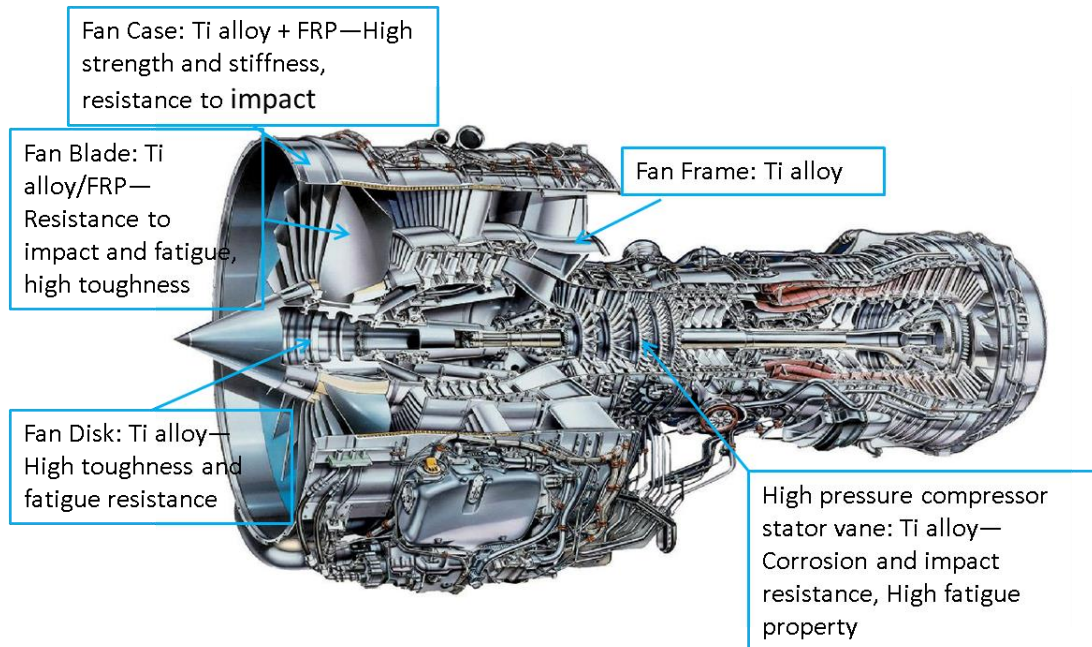


Figure 2.1.1: Titanium usage in a Rolls Royce Trent engine.

Titanium is also used in chemical and power industries as corrosion resistant material because it has high strength, low density, low modulus (high flexibility), and excellent corrosion resistance to seawater [1]. As an example: the drilling riser string in offshore industry is made from Ti-6-4 for its corrosion resistance, sea water fatigue tolerance, and high strength.

One emerging application of titanium is in building applications as exterior walls and roofing materials. An example is the Fukuoka Dome in Japan, which is covered with retractable CP-Titanium roofing (Figure 2.1.2) [1]:



Figure 2.1.2: Fukuoka Dome with retractable titanium roof [1].

In addition to the areas mentioned above, titanium is also used in consumer products such as spectacle frames, sporting goods, watches, cameras. Vanadium (toxic to human body) free β titanium alloys are used as human body implants due to their high fatigue resistance, low elastic modulus, and improved biocompatibility [4].

2.1.2 Physical properties

To illustrate the physical properties of pure titanium in comparison with other common metals, a summary is give in the following Table [1]:

	Ti	Fe	Ni	Al
Melting Temperature (°C)	1670	1538	1455	660
Allotropic Transformation (°C)	$\beta \xrightarrow{882} \alpha$	$\gamma \xrightarrow{912} \alpha$	-	-
Crystal Structure	bcc $\rightarrow$ hex	fcc $\rightarrow$ bcc	fcc	fcc
Room Temperature E (GPa)	115	215	200	72
Yield Stress Level (MPa)	1000	1000	800	500
Density (g/cm ³)	4.5	7.9	8.9	2.7
Comparative Corrosion Resistance	Very High	Low	Medium	High
Comparative Reactivity with Oxygen	Very High	Low	Low	High
Comparative Price of Metal	Very High	Low	High	Medium

Table 2.1.1: Comparison of physical properties among four most commonly used metals [1]. The ‘Yield Stress Level’ in the table indicates the yield strength the corresponding materials could reasonably achieve, depending on the alloy composition and heat treatment condition, for example, Grade 1 CP titanium have tensile strength of 240 MPa while Ti64 could reach tensile strength of 1100 MPa (ASM Aerospace Specification Metals, Inc.).

As is shown in Table 2.1.1, Titanium has the highest strength to density ratio; however, its application is limited because of its high price which is mainly due to its high reactivity with oxygen during production, and as a result inert gas or strict vacuum must be used during production process. On the other hand, the strong affinity to oxygen leads to a quick formation of a stable oxide scale on surface when exposed to air, which enables high oxidation resistance of this material, especially in aqueous acid environments. The oxide layer passivates

titanium as long as it is exposed to oxidizing environments but will quickly break down under reducing conditions or when the temperature is above 600°C [5].

Below 882°C, pure titanium (α Ti) exhibits hexagonal close packed (HCP) crystal structure which is intrinsically anisotropic. Therefore, the elastic properties of single crystal α titanium vary depending on the angle between c axis of the crystal and loading direction. For example, Young's modulus E varies between 145 GPa, for stress axis parallel to the c axis, to 100 GPa, for stress axis perpendicular to the c axis [1]. Less pronounced variations are observed for polycrystalline α titanium with texture. Above 882°C, hexagonal α phase transforms to body centred cubic (bcc) β phase [6]. Certain alloying elements, such as V, Ta, Cr, Mo, Fe, Ni, could retain β phase at temperatures below 882°C. These alloying elements are called β -stabilizer.

Titanium alloys are conventionally classified into α , $\alpha+\beta$, and β alloys based on the variety and amount of alloying elements. Typical to α category is the commercial purity titanium (CP Ti), which differs in oxygen content from 0.18% (grade 1) to 0.40% (grade 2). Yield stress level also increase correspondingly. Grade 7 and grade 12 CP Ti have the same level of oxygen content with grade 2 alloy but offers better corrosion resistance. The most utilized $\alpha+\beta$ alloy is Ti-6Al-4V. This alloy exhibits a good balance of strength, ductility, fracture and fatigue properties. However, it can only be use at temperature up to 300°C [1]. The β alloys show high yield strength and low elastic modulus and therefore steadily gaining usage in areas such as springs and biomedical implantations [1].

2.2 Microstructure related deformation behaviour in CP titanium

Pure HCP titanium has a c/a ratio of 1.586 in room temperature, which is less than the ideal hard sphere model value of 1.633 and therefore is strongly anisotropic. Due to the strong plastic anisotropy and possibility of twinning, hexagonal α Ti has complex deformation modes. Twinning contributes significantly to the low temperature ductility of α Ti. However, twins are suppressed nearly completely in $\alpha+\beta$ alloy mainly because of the small phase dimension, high solute content, and presence of Ti_3Al [1]. In β alloy, twinning also contributes to the ductility at lower temperature but decreases with increasing solute content [1, 7].

The following images demonstrates a quasi-static tensile deformation in CP titanium demonstrating some fundamental microstructural features contributing to the plasticity of this materials before fracture:

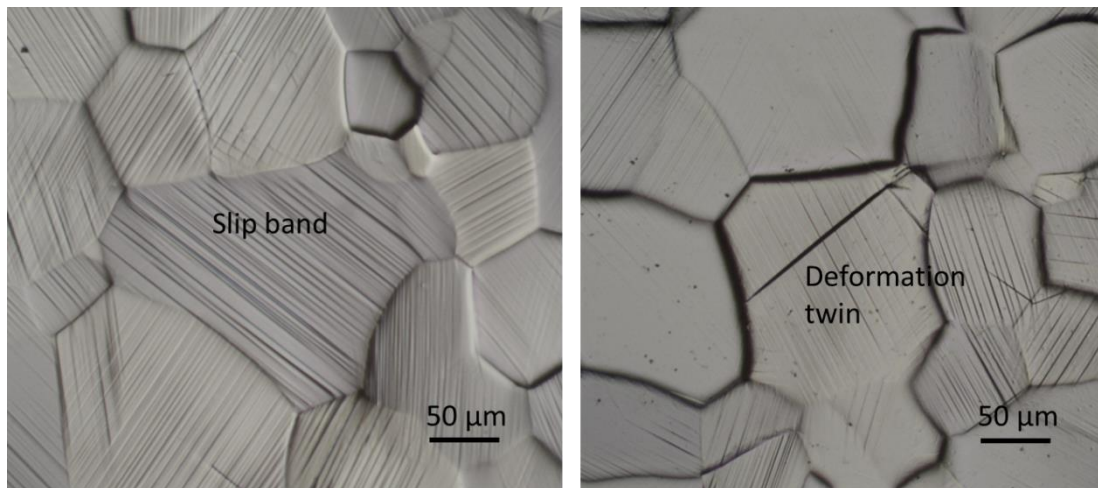
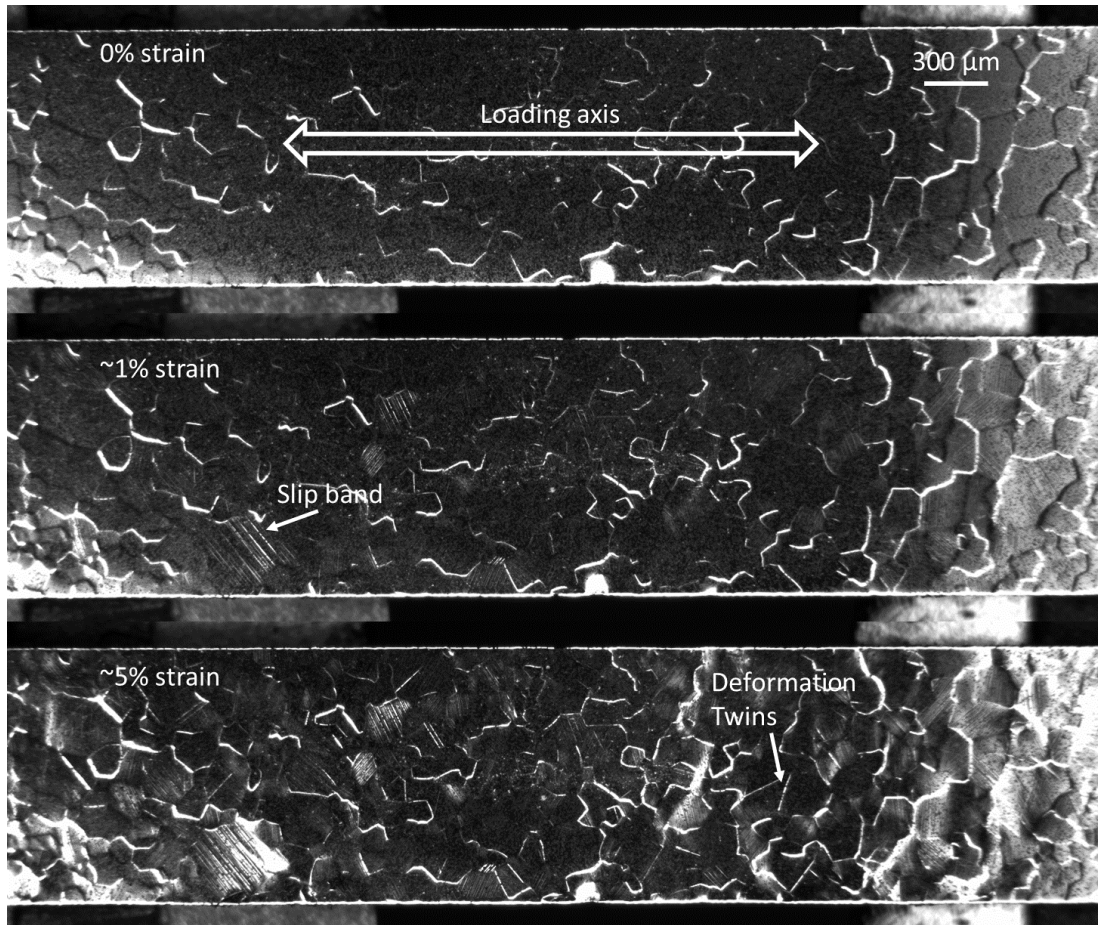


Figure 2.2.1: Optical micrographs showing microstructure changes during tensile deformation of CP titanium. Strain rate is at about 1 $\mu\text{m/s}$. The two optical microscopy images at the bottom are magnified view of the indicated slip band and twin feature on

the top of the image. All images in this figure come from the DIC work related with this thesis.

As is revealed by Figure 2.2.1, early stage of plastic deformation (~1% strain) features the appearance of slip bands. As a result of dislocation motion which terminated at grain boundary in some cases while connected to slip bands in the neighbouring grain in other cases. When the metal is further deformed, twins starts to appear within grains. Indeed, three deformation modes are commonly observed in metals: slip bands, deformation twins, and fracture. Before fracture, slip bands and deformation twins are competing modes of deformation in most BCC and HCP metals with deformation twins contributing more at high strain level, or cryogen temperature, or at explosive loading conditions [8].

2.2.1 Slip systems in α titanium

Dislocation slip usually occurs on close packed planes along close packed directions. For α titanium, the main slip directions are the three close packed directions of the type $\langle 11\bar{2}0 \rangle$. The slip planes that contain this slip direction are the basal plane (0001), three prismatic planes ($10\bar{1}0$), and six pyramidal planes ($10\bar{1}1$), which are indicated in a hexagonal unit cell in Figure 2.2.2. The various slip planes and $\langle a \rangle$ directions offer a total of 12 slip systems as is shown in Table 2.2.1. However, this number can be further reduced to 4 independent $\langle a \rangle$ type slip systems as the shape change of slip system type 3 (see Table 2.2.1) is the combination of that induced by slip systems types 1 and 2 [1, 9], and

there is a redundancy within the three $\langle a \rangle$ slip directions which are all within the basal plane.

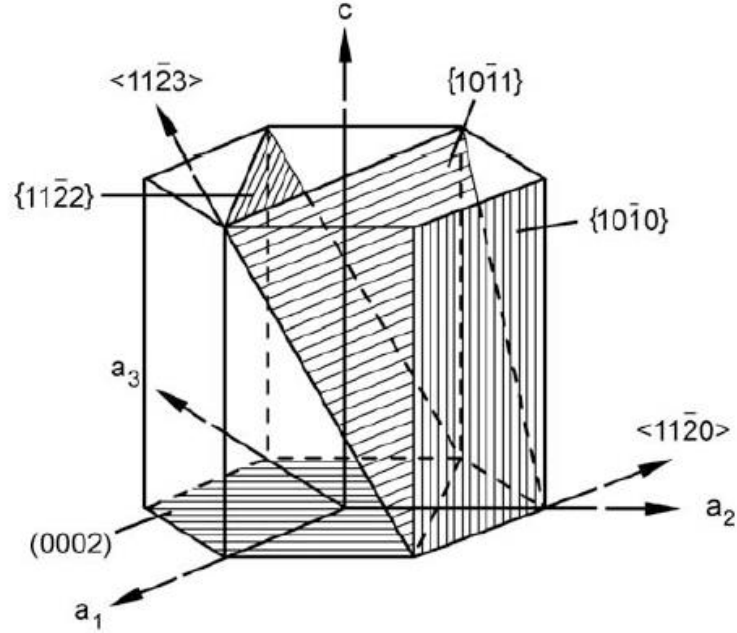


Figure 2.2.2: Slip planes and direction in a hexagonal close packed crystal [1].

Slip system	Burgers vector type	Slip direction	Slip Plane	No. of slip systems	
				Total	Independent
1	a	$\langle 11\bar{2}0 \rangle$	Basal (0002)	3	2
2	a	$\langle 11\bar{2}0 \rangle$	Prism type I $\{10\bar{1}0\}$	3	2
3	a	$\langle 11\bar{2}0 \rangle$	1 st order pyramidal type I $\{10\bar{1}1\}$	6	4
4	c+a	$\langle 11\bar{2}3 \rangle$	2 nd order pyramidal type II $\{11\bar{2}2\}$	6	5
5	c	$\langle 0001 \rangle$	Prism type I $\{10\bar{1}0\}$	3	2
6	c	$\langle 0001 \rangle$	Prism type II $\{11\bar{2}0\}$	3	2

Table 2.2.1: Slip systems in hexagonal close packed metals [9].

According to the Von Mises criterion, at least 5 independent slip systems are required for arbitrary shape change during deformation. Therefore, one of the non-basal slip systems of either type $\langle c \rangle$ with slip direction $[0001]$ or type $\langle c+a \rangle$ with slip direction $\langle 11\bar{2} \ 3 \rangle$ should be activated.

The critical resolved shear stresses (CRSS) to activate different slip systems have been studied using single crystals [10, 11] and micro cantilevers [12]. The CRSS for $\langle c+a \rangle$ slip is a factor of two to three time higher than $\langle a \rangle$ slip on prism planes while the CRSS for $\langle a \rangle$ type slip on basal planes is only 15% higher than that for $\langle a \rangle$ type slip on prism planes. The level of CRSS for the three types of $\langle a \rangle$ slip has been summarized as $(10\bar{1} \ 0) \langle 0001 \rangle \langle 10\bar{1} \ 1 \rangle$ [1].

The percentage of grains in a polycrystalline α titanium that deform with $\langle c+a \rangle$ type of slip is quite low with absence of texture. This is because $\langle a \rangle$ type of slip become easier to be activated when the angle between stress axis and c axis become greater than 10° [11]. In extreme conditions where the applied stress is parallel with c axis of the HCP crystal, under which there would be no resolved shear stress for $\langle a \rangle$ and $\langle c \rangle$ types of slip, deformation twinning could become a powerful competitor to $\langle c+a \rangle$ slip.

Critical resolved shear stress for various slip systems are affected strongly by interstitial atoms, deformation temperature, and c/a ratio [10, 11, 13-15]. It was said that metals with c/a smaller than ideal, prismatic slip is preferred while basal slip is favoured for those with c/a higher than ideal [15].

The hardening mechanism by interstitial atoms is not clear. It was proposed that this kind of hardening is due to Peierls-Nabarro type of interaction between

interstitials and edge dislocation [10, 11]. However, this interaction was proved to be too small to explain the observed strengthening of titanium [14, 16]. Therefore, an alternative chemical interaction mechanism was proposed [14] in which alpha type screw dislocation dissociate sessile parts into one prismatic plane ($10\bar{1}0$) and two first order pyramidal planes ($10\bar{1}1$) and ($10\bar{1}\bar{1}$). The plastic deformation is controlled by sessile-glissile transformation and strengthening occurs when this transformation is affected by lattice distortion induced by interstitial atoms, such as C, N, O, that occupy octahedral sites of the alpha crystal lattice. This mechanism, however, is only valid for relatively high concentration of interstitial atoms [16]. Recent TEM and first principle density function theory work [17] have established a strong interaction between screw dislocation core and oxygen interstitials. A strong pinning effect on screw dislocations was proposed to take place by the combined effect of repulsive solute-dislocation interaction energy, difficulty for mechanical shuffle of oxygen atom in the dislocation core, and local cross slip induced by oxygen interstitials. This work brought new insight into solid solution hardening mechanism by introducing the existence of strong interactions between solute atoms and screw dislocation cores which was previously considered negligible [18].

2.2.2 Basal and prismatic slip in HCP crystals

The preference for activating basal or prismatic slip is not entirely understood. Tyson [19], based on the theoretical treatment of Eshelby [20, 21] and Foreman

[22], calculated the strain energy (E) and lattice frictions (σ_p) in order to study their effect on the choice of primary slip plane.

$$E = \frac{Kb^2}{4\pi} \ln \frac{R}{r_0} \quad (2.1)$$

Where b is the Burger's vector, R is the radius of the strain field of the dislocation, r_0 is the core radius, and K is an energy factor depending on the elastic constants of the crystal. For a HCP crystal whose Burger's vector is in the $\langle 11\bar{2}0 \rangle$ direction:

$$K_{\text{edge}}(\text{basal}) = (c'_{11} + c_{13}) \left[\frac{c_{44}(c'_{11} - c_{13})}{c_{33}(c'_{11} + c_{13} + 2c_{44})} \right]^{\frac{1}{2}} \quad (2.2)$$

$$K_{\text{edge}}(\text{prism}) = \frac{c_{11}^2 - c_{12}^2}{2c_{11}} \quad (2.3)$$

$$K_{\text{screw}} = \left[\frac{1}{2} c_{44}(c_{11} - c_{12}) \right]^{\frac{1}{2}} = \frac{c_{44}}{(A)^{1/2}} \quad (2.4)$$

Where c_{ij} is the elastic constants and $c'_{11} = (c_{11}c_{33})^{\frac{1}{2}}$, and $A = \frac{2c_{44}}{c_{11} - c_{12}}$ is Zener's anisotropic ratio [23]

The energy factor K is linked to the friction stress σ_p of an edge dislocation moving in a hexagonal crystal through the Peierls-Nabarro model [20, 22]:

$$\sigma_p(\text{basal}) = K_{\text{edge}}(\text{basal}) \times \exp \left[-\frac{\pi K_{\text{edge}}(\text{basal})}{c_{44}} \left(\frac{d}{b} \right)_{\text{basal}} \right] \quad (2.5)$$

$$\sigma_p(\text{prism}) = K_{\text{edge}}(\text{prism}) \times \exp \left[-\frac{\pi K_{\text{edge}}(\text{prism})}{c_{66}} \left(\frac{d}{b} \right)_{\text{prism}} \right] \quad (2.6)$$

Where $(\frac{d}{b})$ is the ratio of the spacing between slip planes and the slip distance (magnitude of Burger's vector). For HCP metals: $(\frac{d}{b})_{\text{basal}} = \frac{1}{2}(\frac{c}{a})$, $(\frac{d}{b})_{\text{prism}} = \frac{\sqrt{3}}{2}$. The magnitude of the energy factor K and lattice friction stresses σ_p for a series of HCP metals were calculated [19] using the expression given above and the results are shown in Table 2.2.2.

Metal	c/a	Primary slip plane	$K_{\text{edge}} (10^{12} \text{ dyn/cm}^2)$		$K_{\text{screw}} (10^{12} \text{ dyn/cm}^2)$	$\frac{K_{\text{edge}} (\text{basal})}{K_{\text{edge}} (\text{prism})}$		$\frac{\sigma_p (\text{basal})}{\sigma_p (\text{prism})}$	
			basal	prism					
Cd	1.886	(0001)	0.354	0.508	0.276	0.697		0.153	
Zn	1.856	(0001)	0.556	0.778	0.497	0.715		0.305	
Co	1.628	(0001)	1.247	1.092	0.732	1.139		1.10	
Mg	1.624	(0001)	0.247	0.242	0.167	1.020		1.08	
Re	1.615	(0001)	2.545	2.468	1.668	1.032		0.978	
Tl	1.598	—	0.123	0.0504	0.0443	2.44		5.58	
Zr	1.593	(10-10)	0.540	0.532	0.336	1.016		0.902	
Ti	1.587	(10-10)	0.676	0.551	0.405	1.228		2.35	
Hf	1.581	(10-10)	0.785	0.741	0.538	1.060		1.55	
Y	1.571	(10-10)	0.343	0.337	0.245	1.018		1.288	
Be	1.568	(0001)	1.543	1.449	1.469	1.065		2.001	

Table 2.2.2: Primary slip planes and associated energy factor K and lattice friction stress σ_p for different hexagonal crystal systems. The table is remade from reference [19].

Dislocations tend to glide on close packed slip planes which are usually widely spaced [24]. This means that d/b should be maximized for the chosen slip system, which is in effect a minimization of σ_p . Therefore the ratio of $\frac{\sigma_p(\text{basal})}{\sigma_p(\text{prism})}$ should be an indicator of the relative ease of activation between basal and prismatic slip planes neglecting the anisotropy factor. The magnitude of the energy factor K_{screw} is smaller than K_{edge} and is independent of the slip plane, therefore the energy required to generate edge dislocation is considered

dominant factor affecting the choice of slip planes. It is evident from Table 2.2.2 that neither $\frac{K_{\text{edge}}(\text{basal})}{K_{\text{edge}}(\text{prism})}$ nor $\frac{\sigma_p(\text{basal})}{\sigma_p(\text{prism})}$ correlates well with the observed popular slip plane. If the energy factor ratio and friction stress ratio is to be normalized against c/a ratio, it was found that for the metals above TI, $\frac{\sigma_p(\text{basal})}{\sigma_p(\text{prism})} / \frac{c}{a}$ is mostly smaller than unity except for Mg, and for metals below TI, $\frac{\sigma_p(\text{basal})}{\sigma_p(\text{prism})} / \frac{c}{a}$ is mostly bigger than unity except for Zr. While the ratio $\frac{K_{\text{edge}}(\text{basal})}{K_{\text{edge}}(\text{prism})} / \frac{c}{a}$ is random. This might mean that friction stress plays a relatively more important role in choosing active slip plane than the energy term.

To account for the poor correlation between friction stress and anisotropy factor, the effects occurring at the core of a dislocation was considered important for both FCC and HCP metals [19, 25] in the choice of slip plane. Both E and σ_p depend on the dislocation width, with σ_p been more sensitive to this factor. The wider the dislocation core, the lower the friction stress σ_p . The width of a dislocation is mainly determined by the stacking fault energy separating the partials, while dislocation separates into partials on both basal and prismatic slip plane, the stacking fault energy separating dislocation partials on prismatic plane is higher than that on basal plane, therefore basal slip system is favoured in metals with lower stacking fault energy such as in Cd, Zn, Mg, and Be, while prismatic slip system is popular in metals with higher stacking fault energy such as Ti, Zr, and Hf.

The effect of stacking fault energy on the choice between basal and prismatic slip plane was also explained by the HCP / BCC allotropic transformation [26].

During the phase transformation, a {10-10} plane of the HCP crystal becomes a (112) plane of the BCC crystal and vice versa. The smaller the ratio between BCC to HCP transformation temperature and the melting temperature, $\frac{T_t}{T_f}$, the smaller the fault energy should be on the transformed prismatic plane. While the stacking fault energy on basal plane is not affected by this temperature ratio, it was argued [26] that the prismatic slip plane should be favoured with decreasing $\frac{T_t}{T_f}$. This argument is consistent with the ratio between CRSS, $\frac{CRSS_{\text{basal}}}{CRSS_{\text{Prismatic}}}$, on both basal and prismatic slip planes in Zr, Ti, and Be [11].

2.2.3 Deformation compatibility at grain boundary

Hirth [27] have given an excellent review of the effect grain boundary on mechanical properties of a bicrystal, a summary of which is shown below:

Elastic compatibility

For a perfectly elastically isotropic material, the presence of grain boundary induces no strain localization effect as all grains are essentially deformed the same way. For elastically anisotropic materials, even for near isotropic metals such aluminium and tungsten, stress incompatibility is introduced at the interface.

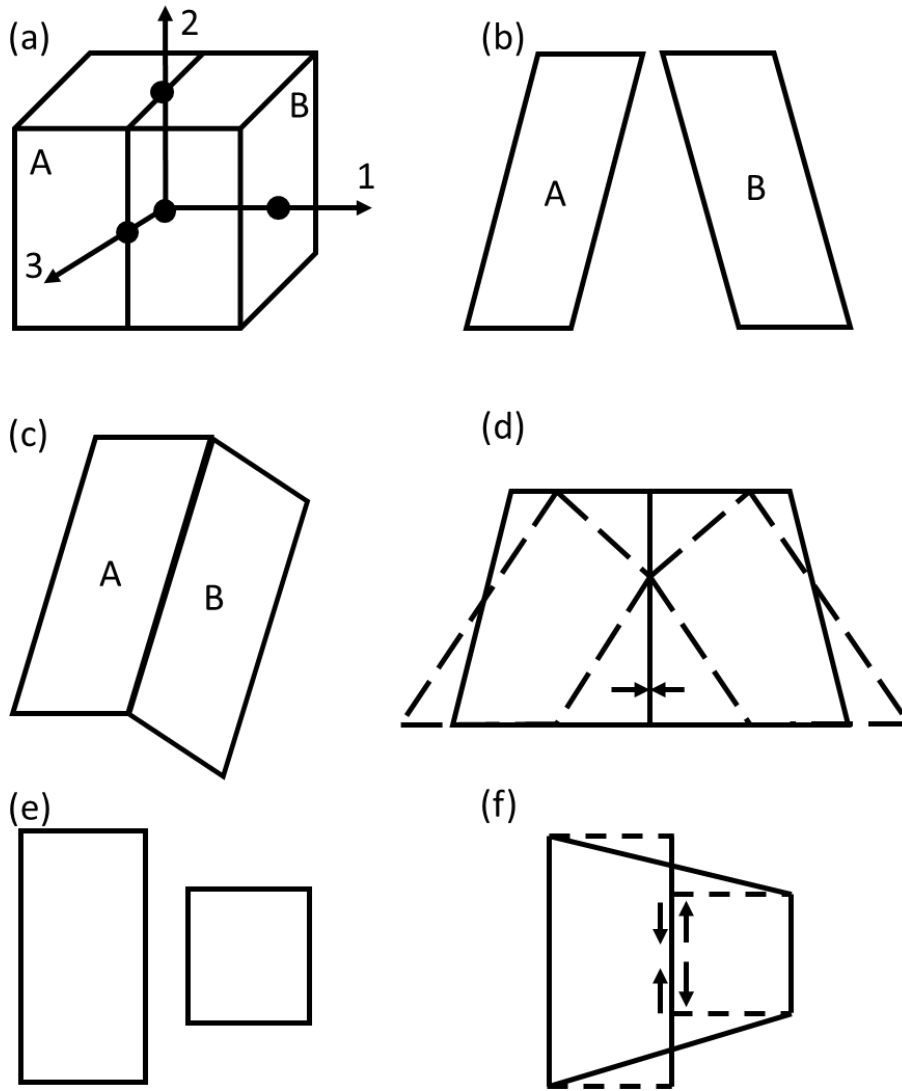


Figure 2.2.3: (a) configuration of a bicrystal and related loading axis (b) shape change when not joined at interface (c) joined at interface but no end constraints (d) shape change with end constraint (e) and (f) alternate response to axial loading.

A simple case of bicrystal deformation is shown in Figure 2.2.3. Under a uniform compressive stress σ_{22} , the bicrystal in figure 2.2.3(a) experiences strain given by:

$$\varepsilon_{ij} = S_{ij22}\sigma_{22} \quad i, j = 1, 2, 3 \quad (2.7)$$

In the isotropic case, the compliance matrix is such that $S_{1111} = S_{2222} = S_{3333} = 1/E$, $S_{1122} = S_{2233} = S_{3311} = -\nu/E$, and $S_{2323} = S_{1212} = S_{3131} = 1/2G$ and all other terms are zero. Under such circumstances only tensile strain in the 2 direction ε_{22} and Poisson contraction in the other two directions $\varepsilon_{11} = \varepsilon_{33}$ occur.

In the anisotropic case, several possible incompatibilities would arise due to other strain components. One possibility is that the imposed stress induces shear strain (Figure 2.2.3(b)). If the component crystals are free to rotate such strain rotates the sample without inducing stress (Figure 2.2.3(c)), otherwise shear stress σ_{12} is induced (Figure 2.2.3(d)). If crystal A and B have a relative misorientation such that their resolved compliance S_{2222} are different, under loading they would experience different tensile strain ε_{22} (Figure 2.2.3(e)). Under this condition, the imposed normal stress σ_{22} would induce shear stress σ_{12} at the interface if the two crystals are welded together. The compatibility requirement pose the following constraints on the elastic displacements and stresses at the interface:

$$u_1^A = u_1^B, u_2^A = u_2^B, u_3^A = u_3^B \quad (2.8)$$

$$\sigma_{11}^A = \sigma_{11}^B, \sigma_{12}^A = \sigma_{12}^B, \sigma_{13}^A = \sigma_{13}^B \quad (2.9)$$

The incompatibility stress at the interface was shown [28] to decrease 'exponentially' away from the interface. While the most common effect of this stress concentration is related to the operation of dislocation sources near the boundary [27], it can potentially lead to the concentration of screw dislocations at harder phase in the interface, promoting crack initiation while on the other hand diverges screw dislocation in the softer phase, blunting dislocation pile up

and reducing the tendency for crack formation [28], a so called ‘focussing effect’ [27].

The effect of the incompatibility stress near interface of a bicrystal was found to affect choice of local slip systems [29]. A pseudo-identical bicrystal of Fe-3%Si which was orientated for easy glide (maximum global Schmid factor of 0.5) on primary slip systems was deformed in uniaxial loading. Apart from the primary slip lines, secondary slip lines across the interface with a global Schmid factor of only 0.182 were also observed. However, when the local incompatibility stress was resolved onto the secondary slip plane together with the global resolved shear stress, the secondary slip systems were found to be the ones with maximum total resolved shear stress. When moving away from the interface, the incompatibility stress decreases, resulting in the choice of new slip systems with maximum resolved shear stress and therefore a curved slip band. The work by Hook and Hirth [29] on bicrystal and some early work on polycrystals [30, 31] tend to point to a conclusion that activation of slip systems predicted by using only the global Schmid factor is not sufficient. A critical resolved shear stress law needs to be employed for initiation of yielding, which takes into account the effect of the elastic anisotropy on stress distribution of a deforming system.

Plastic compatibility

According to the criteria of Taylor [32] and Von Mises [33] for an arbitrary shape change during plastic deformation in a polycrystalline aggregate, six strain

component must be specified. If the deformation doesn't involve volume change, only five strain components are independent, and therefore five independent source of plastic strains are required. The plastic strain sources are not necessarily limited to dislocation slip, deformation twinning and strain induced phase transformations [34] are also effective accommodating mechanisms of plastic deformation.

A different scenario applies to bicrystal as only deformation near the interface is relevant [27]. The plastic compatibility relation for a bicrystal was shown to be:

$$\varepsilon_{23}^A = \varepsilon_{23}^B, \varepsilon_{22}^A = \varepsilon_{22}^B, \varepsilon_{33}^A = \varepsilon_{33}^B \quad (2.10)$$

Since ε_{22} is fixed by the externally applied strain, only four independent strains need to be satisfied for plastic deformation. This requirement is satisfied most commonly by activating four slip systems near the bicrystal interface [29], but other deformation mechanisms such as twinning are also possible.

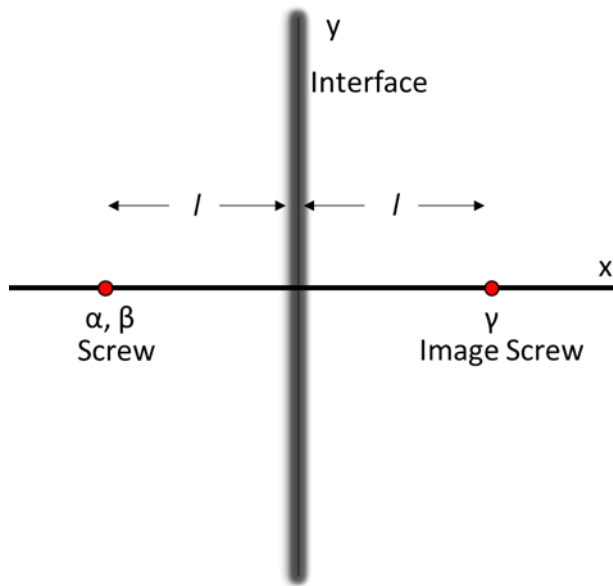
The Taylor theory [32] and its improvement by Bishop and Hill [35] are among the early approaches to tackle anisotropic plasticity. However, their implementations are based on simplifying assumptions of homogeneous strain across each grain in order to maintain continuity across grain boundary. Therefore they don't consider the inter and intra grain interactions in a polycrystalline. The complicated internal and/or external boundary conditions can be approximated using crystal plasticity finite element (CPFE) models, which are based on variational solution of force equilibrium and displacement compatibility, using extensive knowledge gained from experimental and theoretical studies of dislocations and single crystal deformation mechanism, in

a given finite-volume element which is discretized from the overall sample volume [36, 37]. There are a variety of constitutive formulations regarding plastic flow and hardening that can be included in CPFE models. These constitutive laws range from phenomenological model that use critical resolved shear stress as state variable [38, 39], to physically based, size dependent models [40, 41], and interface mechanisms [42]. A comprehensive review of the constitutive models and basics of continuum mechanics underpinning CPFE method can be found in [36] and [37].

Image stresses

A dislocation approaching an interface is subject to a long range force which could either attract the dislocation towards the interface or repulse it from the interface [27], such force is independent of external loading and is named 'Image Force'.

The image stress field arise from elastic compatibility requirement at interface. If a screw dislocation α is placed near a free surface (as shown in the following illustration), its elastic strain field impose a shear stress σ_{xz} at the interface.



The traction free boundary condition at the free surface requires $\sigma_{xz} = 0$, therefore the imposed stress field need to be cancelled by placing an imaginary dislocation of the same strength but with opposite sign, γ , at the mirror image position at the other side of the free surface. The image stress at a free surface tends to attract the dislocation towards the free surface [43]. A slightly different situation apply to dislocations near a grain boundary (or phase boundary) separating two differently oriented grains in an anisotropic material. In this situation, Hirth [27] have shown that the elastic compatibility at the grain boundary need to fulfil the following condition: $\sigma_{xz}^A = \sigma_{xz}^B$ and $u_z^A = u_z^B$. Due to elastic anisotropy across the boundary, satisfying one condition alone will leave the other condition unsatisfied. To meet the two conditions at the same time, two additional elastic fields are needed which is equal to placing two imaginary dislocations β and γ at image positions of α and the grain boundary. The elastic field in grain A is determined by dislocation α and γ and that in B is determined by β . If the effective shear modulus in B is larger than that in A then the

dislocation α will be repelled from the grain boundary and vice-versa [27]. In some cases of slip band and grain boundary interactions, image stress was proposed to be a reason preventing dislocations from entering the grain boundary [29]. The magnitude of the image force is inversely proportional to the distance away from the interface, therefore its effect is pronounced in thin foils. It was shown that the image force cause dislocations to escape from thin TEM foil and also changes the imaging condition close to the thin foil free surface [44].

2.2.4 The effect of grain boundaries on materials properties

The ability to link improved macroscopic materials properties with the increased population of a group of ‘special’ grain boundaries was noticed over 30 years ago [45], motivating the paradigm of research known as grain boundary engineering. The approach to increase the population of ‘special’ boundaries often involves repetitive strain-annealing cycles [46-49] and the thereby increased proportion of ‘special’ boundaries were correlated with increased properties in areas regarding intergranular stress corrosion cracking (IGSCC) [48, 50], creep [47, 51], intergranular corrosion [52], and so on.

The above mentioned ‘special’ grain boundaries refer to those with low Σ numbers according to a method of categorizing grain boundary characters based on the coincidence site lattice (CSL) theory. The theory formed under the discovery that when two purposely oriented crystals of the same phase were brought together, certain portion of the lattice sites overlap, forming a periodic

'super lattice' structure [53]. The inverse of the volume fraction between the overlapping unit-cell of coincident super-lattice with that of the bulk crystal defines a specific Σ number [54], for example, if one in three lattice sites are coinciding the grain boundary is denoted $\Sigma 3$ boundary. A boundary containing higher fraction of CSL is expected to have lower energy state as a result of better atomic fit. For example, it has been shown [55] that for a $\langle 110 \rangle$ tilt boundary of FCC crystals, the energy of the boundary increase with misorientation angle but with significant valleys found at misorientation angles giving rise to $\Sigma 3$ and $\Sigma 5$ configurations.

Statistical analysis [56-58] found a systematic decrease in volume fraction from $\Sigma 3$ type to $\Sigma 29$ boundary type in a non-textured material, while the average proportion of a specific Σ boundary type is often significantly altered by the presence of texture. Recent high energy X-ray diffraction microscopy (HEDM) and forward modelling analysis [59] on high purity nickel probing a volume containing ~ 1000 grains found that the majority of grain boundaries concentrated on those low energy types such as: $\Sigma 3$ $\Sigma 9$ $\Sigma 11$ $\Sigma 27$. The grain boundary Σ type and the corresponding axis/angle pair for cubic and hexagonal crystal systems are clearly tabulated by Action et al. [60] and Bonnet et al. [61] respectively. While these tables [60, 61] provide a theoretical ideal orientation relationship for a grain boundary Σ type, deviations are inevitable due to experimental error or increased number of dislocations in the boundary as a result of plastic deformation. The maximum allowable deviations from the exact CSLs are derived from the systematic similarity between low CSL boundaries and the Read-Shockley model [62] and is given by the Brandon criterion [63]:

$$\Delta\theta \leq 15^\circ \Sigma^{-\frac{1}{2}} \quad (2.11)$$

This criteria was modified by Palumbo and Aust [64] to the form of:

$$\Delta\theta \leq 15^\circ \Sigma^{-\frac{3}{5}} \quad (2.12)$$

This modified form of the Brandon criteria is gaining popularity as it was shown to be most consistent with experimental observations [65]. The maximum deviation angle was also said to capture the maximum dislocation density tolerance of the grain boundary [54].

It is worth the attention that the CSL categorization of grain boundary characters is based solely on the orientations of neighbouring grains [65]. While the CSL provides a convenient 'short hand' method for classification of grain boundaries, suspicions were cast on the link between low Σ number and improved property [63, 66, 67]. The character of grain boundary is fully captured by five parameters, 3 for misorientation of lattices across the boundary (i.e. CSL) and 2 specifying the grain boundary orientation [68]. The five parameter grain boundary character distribution (GBCD) analysis put emphasize on the subsurface grain boundary orientation as it directly affects the density of the CSL site actually occupying the grain boundary plane [63], in another word, the 3D grain boundary orientation dictates the miller indices of the boundary plane. For example, it was shown [69] that for Fe-6%Si alloy if the orientation of the grain boundary plane is such that a 37° [111] tilt grain boundary was formed by low index planes rather than high index planes, greater corrosion resistance was observed. Further, in FCC crystals the coherent twin boundary on a {111} plane and the incoherent twin boundary on a

{112} plane both belong to the $\Sigma 3$ boundary type, however, the energy of the former is much lower than that of the latter [68]. Effect of the grain boundary inclination on $\Sigma 3$ boundaries was investigated in Cu [70] and the grain boundary energy was found to increase as it moves away from full coherent twin configuration. A full five parameter investigation of MgO conducted by Rohrer et al [71] concluded that the grain boundary inclination dependence of grain boundary energy is not restricted to Σ boundaries, it is rather a general phenomenon that occurs to all lattice misorientation. The effect of 5 parameter GBCD on fracture behaviour of polycrystals was demonstrated by Watanabe [72]. It was shown that high temperature fracture highly favours random boundary (no CSL relation) with boundary plane inclined at 45° to the loading axis and the fracturing propensity could be greatly reduced by introducing low Σ boundaries that are either 45° or perpendicular to the loading axis.

The full 5 parameter GBCD can be captured using stereological technique [73], serial sectioning-EBSD [74-76], and synchrotron based technique [77, 78]. The stereological and serial sectioning-EBSD technique were compared by Rohrer et al [76] using IN100 Ni base superalloy. In this research, EBSD data was collected on a series of surfaces systematically removed using focused ion beam (FIB) milling, on each data slice the traces of grain boundaries were recorded and assigned a spatial vector, $\mathbf{r}_i$. The spatial coordinates of triple points can then be identified by the coordinate where 3 grain boundaries meet. A digital image correlation approach was employed to align slices together to form the 3D data set. The relative position between the same triple points in adjoining slices of map define the orientation of the triple line, $\mathbf{l}$, and the cross

product between $\mathbf{l}$ and $\mathbf{r}_i$ define the grain boundary plane normal for each of the three grain boundaries adjoining the triple line. The slice alignment could be further improved by applying a rigid shift to each layer such that the mean triple line direction between two layers is about normal to sample surface [75, 76]. It was reported [76] that the secondary alignment procedure brought the calculated population of $\Sigma 3$ tilt boundary ($60^\circ/[111]$) closer to that captured using the stereological technique. The alignment accuracy could be further improved by increasing the out-of-plane to in-plane ratio (calculate triple line direction between every other slices) as well as the discretisation of the domain of grain boundary types [75, 76]. The serial sectioning-EBSD technique has merits over the stereological technique in that it captures local grain boundary characteristics, however, it is destructive and therefore cumbersome in linking the captured GBCD to global and local mechanical behaviours. Synchrotron based techniques [77, 78] are able to overcome this problem and enable in-situ testing in faster beamline set up [78], but the beam time over demand ratio is a major constraint.

A more dedicated application of FIB 3D serial sectioning technique has emerged to investigate the grain boundary topography [79]. Recent application [80] to nickel base superalloy found that grain boundaries that exhibit high temperature grain boundary sliding generally have a lower level of surface roughness (a few order of magnitude) than those that do not exhibit grain boundary sliding. While it was not made clear whether the surface roughness came into existence before or after deformation, the presence of surface roughness is generally consistent with earlier field ion microscopy investigations

in different material systems [81, 82] and the presence of surface topography was also reported to affect mechanical behaviours such as creep and yielding [83, 84].

The above review leads to the conclusion that neither misorientation nor 2D surface grain boundary traces are sufficient to correlate with materials properties especially when the very local event is to be studied. The effect of subsurface grain boundary plane orientation as well as the local grain boundary topography are all very important factors affecting mechanical and chemical-mechanical responses of materials.

2.2.5 Slip band and grain boundary interactions

Grain boundaries are effective barriers to dislocation motion providing a substantial strengthening mechanism for polycrystalline materials [27]. The pile-up of dislocations against grain boundaries could lead to localised high intensity stress concentrations, especially in planar slip materials. The forward stress generated by slip band/grain boundary interaction is cited to lead to slip transfer [85], deformation twin nucleation [86, 87], cavity nucleation [88], fatigue crack nucleation [89], and some other phenomena [90, 91].

The back stress of a pile-up, which results from cumulative stress fields from each individual dislocation and therefore depends on the size of the pile-up and the number of dislocations in it, tends to counteract the externally applied stress. This leads to a lower resultant stress state driving further dislocation slip [92]. Such a model has been widely used as an explanation for the empirical Hall-

Petch relationship [92-94] which relates yield strength to grain size of a polycrystalline material by the equation: $\sigma_y = \sigma_0 + kD^{-\frac{1}{2}}$, where D is the average grain diameter. The 'friction stress' σ_0 is the stress required to sustain dislocation motion in the interior of a grain [95, 96], while the $kD^{-\frac{1}{2}}$ term is the grain boundary contribution to yield strength [95]. k is often referred to as the Hall-Petch coefficient, and usually captures the average effect of the grain boundaries in the polycrystal. A recent reassessment of much of the literature data concerning grain size effects on yield strength by Dunstan and Bushby [97] has cast doubt on the Hall-Petch relationship, and attempts to rationalize it with other size effects.

A dislocation pile-up, (i.e. a slip band), forms by gliding dislocations, emitted from a dislocation source [98] on the same glide plane, immobilized by obstacles (for example grain boundary) that they cannot pass. When the length scale is reduced to the nano-regime (a few to a few tens of nanometers), the concept of a continuum dislocation pile up no longer exists and instead the formation of single dislocation loops and their deposition at the interfaces is considered [99-101]. The slip band is crystallographic in nature, and this is to be distinguished from microband or shear bands that is observed in metals subject to large deformation [102]. The name 'microband' was adopted to describe a microstructure feature that is a few μm in length and a few hundred of nm thickness, formed from collective dislocation activity in a local zone within a single grain, leading to large local misorientation ($\sim 15^\circ$) within the band [102, 103]. The microband was found to be precursor for shearband formation which

is noncrystallographic and can traverse multiple grains with different orientations [103].

The Hall-Petch coefficient has traditionally been obtained by mechanical testing of samples with varying grain diameter. Hyun et al [104] have reported $k = 0.25 \text{ MPam}^{0.5}$ for grade 2 commercially pure titanium (CP-Ti) deformed at room temperature under tension. While Lederich et al [105] found $k = 0.18 \text{ MPam}^{0.5}$ and $k = 0.40 \text{ MPam}^{0.5}$ for pure Ti deformed under tension at 575K and 295K respectively. For room temperature compression Salem et al [106] reported $k = 0.67 \text{ MPam}^{0.5}$ for CP-Ti. Although $\sigma_y = \sigma_0 + kD_{GB}^{-1/2}$ has now been shown to support macro-mechanical testing data better than the Hall-Petch equation [14], the Hall-Petch coefficients available in the literature do allow the overall effects of crystal structure, alloying additions and other factors on the relative effectiveness of grain boundary strengthening to be assessed. Of course it is to be expected that the nature and geometry of individual grain boundary types should have a pronounced effect on the intensity of local stress concentrations generated [107]. Amongst others, Sangid et al. [108] suggest that the slip band and grain boundary interaction is strongly affected by the character and structure of grain boundaries. This leads to the thinking that boundaries of different characteristic should have different resistance to slip transfer. Therefore, an investigation into the strengthening mechanisms of individual grain boundaries is of theoretical and practical importance.

A large body of theoretical and modelling work has been focused on predicting the resistance to slip transfer using boundary related parameters as has been

reviewed by Morris [109]. Some of the proposed models require grain boundary parameters that cannot be directly measured, while others contain parameters that depend on pre-knowledge of k , rendering those models descriptive but not predictive. The research of Beta et al. [96] suggested a relatively independent model but this suffers from difficulties of accurately determining grain boundary strain energy and ignores stress concentrations that might arise near grain boundary. Theoretical work by Eshelby, Frank, and Nabarro [110] proposed a solution to modelling slip band-grain boundary interactions. This model, illustrated in Figure 2.2.3, consists of an array of edge dislocations blocked by a grain boundary. This causes a stress concentration at the tip of the slip band in the neighbouring grain. This stress concentration, when resolved to the shear plane of the pile-up, attenuates approximately as a 'one over square root distance' fashion directly ahead of the pile-up away from the grain boundary. The resolved shear stress τ can thus be written as

$$\tau = \tau_0 + \frac{K}{\sqrt{r}} \quad (2.13)$$

Where K describes the stress intensity of the stress field and r is the distance from the grain boundary.

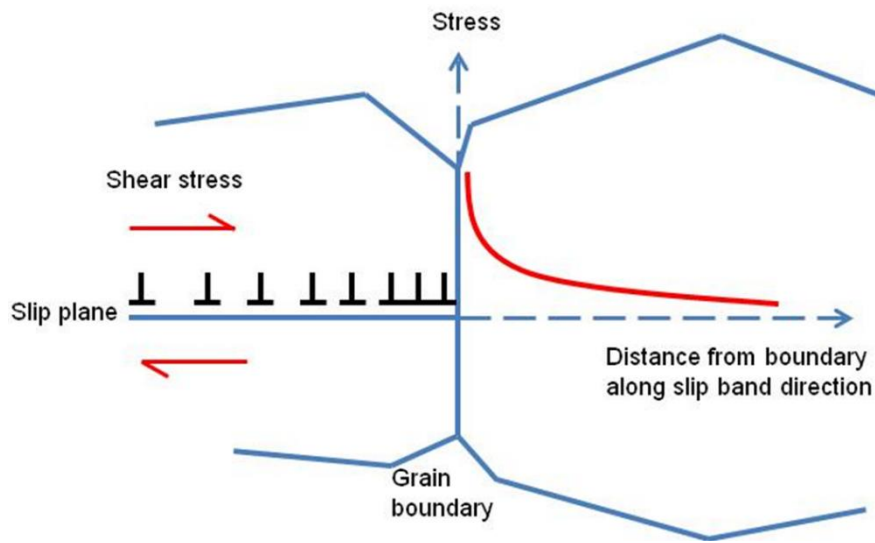


Figure 2.2.3: Eshelby's model of dislocation pile-up at grain boundary. The red curve to the right of grain boundary represents stress distribution ahead of the blocked slip band.

Britton et al. [111] recently mapped the stress distribution near the head of a blocked slip band using the High Resolution EBSD (HR-EBSD) technique [112, 113] and found the stresses to be consistent with the Eshelby-Frank-Nabarro model. The stress intensity factor K can be obtained by curve fitting of the resolved shear stress line profile ahead of the pile-up. This study provided a method to directly measure the stress intensity factor associated with an individual selected slip band-grain boundary interaction. It is therefore important to extend such measurement to different slip band/grain boundary interactions for a systematic evaluation of the strength of grain boundary in terms of the alignment between crystal slip systems.

The stress concentration accumulated by the dislocation pile-up can be redistributed along grain boundary if the Burger's vector of grain boundary

dislocation dissociated from matrix dislocation is within the boundary plane in which it can then glide. This grain boundary gliding mechanism was described in [114, 115]. If the grain boundary dislocations have a component out of its boundary plane then ledges can be created which can in turn lead to the nucleation of boundary cracks [116]. This has been observed by Lee et al. [117] via an in-situ TEM study. The most common stress relief mechanism is by generating new dislocations in the neighbouring grain [118-124].

This could be done by several experimentally observed mechanisms:

(1) Direct transfer, when the incoming slip plane share a common intersection with the outgoing slip plane on grain boundary, and the Burger's vectors of both slip systems are equal, i. e. this transfer mechanism leaves no residual Burger's vector at grain boundary.

(2) Transfer with residual grain boundary dislocation. This mechanism is accomplished by dislocation absorption and subsequent re-emission. The emitted dislocations could either connect to the previous slip band or be displaced along grain boundary [122]. An energy barrier proportional to the magnitude of residual Burger's vector was proposed to play a crucial role in this type of transfer mechanism [108, 124];

(3) New dislocation sources could be activated due to local stress concentration generated around the tip of a blocked slip band. For this type of dislocations to glide across the whole grain, resolved shear stress on their slip plane should be sufficiently large otherwise they cease propagating after a short distance from

grain boundary and other more mechanically favourable dislocation sources could be activated [124].

Early research by Livingston and Chalmers [121] attempted to predict which slip system is more favourable during a slip transfer process, and for this purpose a criteria was proposed:

$$N_{ij} = [(\mathbf{e}_i \cdot \mathbf{e}_j)(\mathbf{g}_i \cdot \mathbf{g}_j) + (\mathbf{e}_i \cdot \mathbf{g}_j)(\mathbf{e}_j \cdot \mathbf{g}_i)] \quad (2.14)$$

Where $\mathbf{e}$ is slip plane normal and $\mathbf{g}$ is slip direction. The subscriptions i and j represent two slip systems on either side of a grain boundary. Slip transfer is expected to the system that maximises N_{ij} , with a high N_{ij} value indicating easier slip transfer. This criterion emphasise the importance of geometric alignment on the choice of transferred slip system. Later work by Shen et al [119] pointed out the importance of incorporating a resolved shear stress on slip transfer and a new model was proposed:

$$N'_{ij} = (\mathbf{L}_i \cdot \mathbf{L}_j)(\mathbf{g}_i \cdot \mathbf{g}_j) \quad (2.15)$$

Where $\mathbf{L}$ is the line of intersection between grain boundary plane and the planes of incoming (subscript i) and outgoing (subscript j) slip systems. Slip transfer is again favoured for slip systems with highest N'_{ij} , i.e. the angles between $\mathbf{L}_i$, $\mathbf{L}_j$ and $\mathbf{g}_j$, $\mathbf{g}_i$ should be minimized. The transferred slip plane therefore is the one that maximize N'_{ij} and the slip direction is the one that have highest resolved shear stress. While this model was successful in predicting all four boundary/pile-up configurations studied by Shen et al., exceptions were found by Lee et al. [123] that, in some cases, systems that generate large grain

boundary dislocations cease to operate even when the resolved shear stress is highest. Taking into account this issue, the slip transfer criteria suggested by Lee, Robertson, and Birnbaum (LRB criteria) consists of three aspects:

- (1) The geometric alignment factor N'_{ij} as shown in equation 2.15 should be maximized;
- (2) The magnitude of residual Burger's vector as the difference between incoming and outgoing dislocations should be minimized;
- (3) The resolved shear stress on the outgoing slip system imposed by the incoming slip system should be maximized.

Although derived from a study of FCC metals, the LRB criteria has had some success in studying slip transfer process in HCP titanium alloys [125, 126].

The first and second points in LRB can be combined if Burger's vector, $\mathbf{b}$, is used to describe the slip direction, $\mathbf{g}$, in equation 2.15. In reality, equation 2.15 is difficult to apply because in current experimental condition it is not easy to determine $\mathbf{L}$.

As an alternative, Werner et al [127] suggested that slip plane normal $\mathbf{n}$ could be used to replace $\mathbf{L}$ as the angle between slip plane normal could never exceeds the angle between intersection lines with grain boundary irrespective of the grain boundary orientation. Therefore an alternative equation taking account of both geometric alignment and residual Burger's vector can be written as:

$$M = \frac{(\mathbf{n}_i \cdot \mathbf{n}_j)(\mathbf{b}_i \cdot \mathbf{b}_j)}{|\mathbf{b}_i||\mathbf{b}_j|} \quad (2.16)$$

Where **n** and **b** are slip plane normal and Burger's vector respectively.

2.2.6 Deformation twinning in titanium alloy

Deformation twinning is a process in which a homogeneous simple shear is imposed to a material. The crystal within the sheared region rotates to a new orientation. The product crystal, called twin, is related to the original crystal, the parent, by either reflection in a composition plane or rotation of 180° about a certain axis or satisfies both conditions in what is known as a compound twin. If the shearing direction of the twin has a component out of the sample surface then a well-defined tilt is produced on the surface of a well-polished grain, which can be readily detected by optical microscopy, electron microscopy, and atomic force microscopy. A detailed review of deformation twinning has been given by Christian and Mahajan [128]. Both deformation twinning and dislocation slip are accommodated by partial dislocation movement, though there are fundamental differences. While dislocation slip is a thermally activated process and therefore depends on temperature and strain rate, deformation twinning is less sensitive to temperature and rate effects. Moreover, deformation twinning is often found to have induced lattice rotation, and is observed to be microstructurally sensitive [129, 130]. A growing twin often terminates at a grain boundary. The effect of twinning on plastic deformation is twofold: it subdivides grains and therefore creating a barrier for dislocation slip

and so increases the work hardening rate; it also contributes to deformation through twinning shear and therefore decreases the work hardening rate [8]. The barrier for twinning is the interfacial energy generated by twin boundary, while the driving force consists two parts: the imposed shear stress and the energy of the region at the two tips of a twin embryo [131].

The crystallographic element of twinning can be represented with the aid of unit sphere:

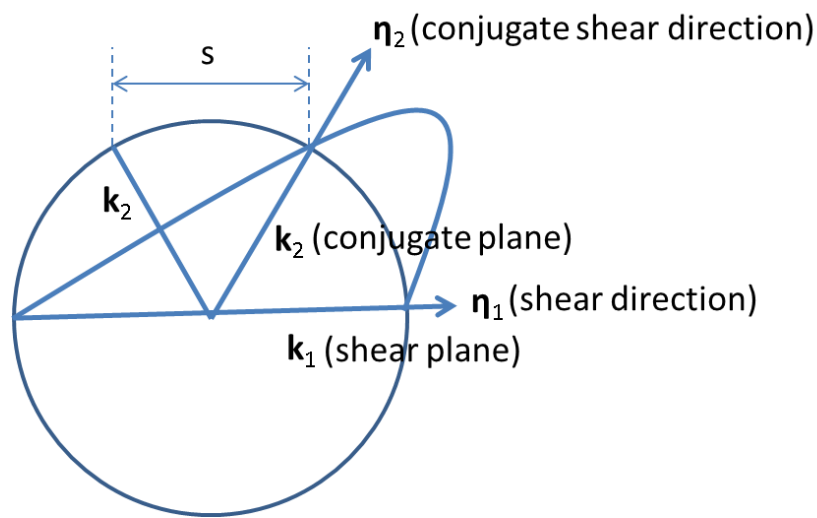


Figure 2.2.4: crystallographic elements of twinning (Remade from reference [132]).

The twinning plane, or invariant plane, intersects the plane of drawing in the shear direction η_1 . During twinning, half of the unit sphere above twinning plane is sheared to an ellipsoid with equal volume. Distortion of planes occurred in all cases except for k_1 and k_2 . k_2 is called conjugate plane which intersects the plane of drawing in conjugate shear direction η_2 . A particular twinning mode of the crystal is defined when k_1 and η_2 , or equivalently, k_2 and η_1 , are given. A twin can be defined as type I and type II according to whether the twinned

volume is a reflection of $\mathbf{k}_1$ or a rotation of 180° about $\boldsymbol{\eta}_1$ respectively. A compound twin contains both of these operations [128]. The shear strain $\mathbf{S}$ (see Figure 2.2.4) is related to the angle between $\mathbf{k}_1$ and $\mathbf{k}_2$ and therefore is uniquely determined by the crystallographic nature of the two undistorted planes [132]. This relationship was considered by Christian and Mahajan [128] as an affine transformation:

$$\mathbf{v} = \mathbf{S}\mathbf{u} \quad (2.17)$$

Where $\mathbf{S}$ is the twinning shear strain, $\mathbf{v}$ and $\mathbf{u}$ represent lattice vectors of the twin and parent respectively.

An algebraical description of the relationship between type I, type II twins and the parent was developed by Kocks et al. [133]:

$$\mathbf{o}_{tw} = \begin{pmatrix} \boldsymbol{\eta}_1 \\ \mathbf{p} \\ \mathbf{k}_1 \end{pmatrix} \quad (2.18)$$

Where $\mathbf{o}_{tw}$ is the orientation of the twin with respect to that of parent, $\boldsymbol{\eta}_1$ and $\mathbf{k}_1$ represent the twinning plane normal and twinning direction, $\mathbf{p}$ is the normal of the plane determined by $\boldsymbol{\eta}_1$ and $\boldsymbol{\eta}_2$ (see Figure 2.2.4).

The transformation matrix for type I and type II twins are:

$$\mathbf{t}^I = \begin{pmatrix} 1 & 0 & 0 \\ 0 & 1 & 0 \\ 0 & 0 & -1 \end{pmatrix} \quad \mathbf{t}^{II} = \begin{pmatrix} 1 & 0 & 0 \\ 0 & -1 & 0 \\ 0 & 0 & -1 \end{pmatrix} \quad (2.19)$$

Therefore, the orientation of twin $\mathbf{g}^t$ with respect to a matrix of orientation $\mathbf{g}^m$ can be written as:

$$\mathbf{g}^t = \mathbf{tO}_{tw}\mathbf{g}^m \quad (2.20)$$

This twin-matrix relationship has been used in automated EBSD (electron backscattered diffraction) analysis to characterise and identify various twinning statistics [134].

Due to the difficulty of activating five independent slip systems in HCP metals during deformation, twinning is an important mechanism that contributes to the observed ductility. In titanium, it was reported that four deformation twinning modes could be activated [135, 136]. Crystallographic elements and parameters of the four twin systems are shown in Table 2.2.3.

Mode	K_1	K_2	η_1	η_2	Misorientation	Shear
T1	$\{10\bar{1}2\}$	$\{10\bar{1}\bar{2}\}$	$\pm\langle\bar{1}011\rangle$	$\pm\langle\bar{1}01\bar{1}\rangle$	$85\langle\bar{12}10\rangle$	0.171
T2	$\{11\bar{2}1\}$	(0002)	$1/3\langle\bar{1}\bar{1}26\rangle$	$1/3\langle 1120\rangle$	$35\langle\bar{11}00\rangle$	0.629
C1	$\{11\bar{2}2\}$	$\{11\bar{2}\bar{4}\}$	$1/3\langle 11\bar{2}\bar{3}\rangle$	$1/3\langle 22\bar{4}3\rangle$	$65\langle\bar{1}100\rangle$	0.221
C2	$\{10\bar{1}1\}$	$\{10\bar{1}\bar{3}\}$	$\langle 10\bar{1}\bar{2}\rangle$	$\langle 30\bar{3}2\rangle$	$54\langle\bar{1}2\bar{1}0\rangle$	0.101

Table 2.2.3: Deformation twinning modes in titanium.

Depending on the type of local strain (tension or compression) twins could accommodate when the c axis of a grain in a polycrystalline metal is parallel with imposed stress, twins can be classified as tension and compression type. As is shown in Table 2.2.3, the $\{10\bar{1}1\}$ and $\{11\bar{2}2\}$ twins are compression twins (denoted C2 and C1), while $\{11\bar{2}1\}$ twin is tensile twin (denoted T2). The $\{10\bar{1}2\}$ twin is of compression type for metals with axial ratio $\frac{c}{a} < \sqrt{3}$ such as Cd and Zn, but it is of tensile type for metals with axial ratio $\frac{c}{a} > \sqrt{3}$ such as Ti. This is

because the shear direction for $\{10\bar{1}2\}$ twin reverses at $\frac{c}{a} = \sqrt{3}$ [135]. A particular twinning mode is favoured when the process involves low level of shear and relative ease of atomic shuffling [128]. The $\{10\bar{1}2\}$ twin is the most common twin type in hexagonal close packed metals and it is the only twin mode in less ductile metals such as Zn and Be. On the other hand, HCP metals that exhibit extensive ductility such as Zr and Ti twin by both tension and compression types. The enhanced ductility observed in Zr and Ti at low temperature is attributed to the profuse $\{11\bar{2}1\}$ tension twins [135].

Although being a very important deformation mode, the exact mechanism of twinning is not clear [132]. It is tempting to assume that the critical resolved shear stress (CRSS) theory that is classically applied to dislocation slip would apply to deformation twinning in such a way that the lower the CRSS the easier the onset of twinning. As a matter of fact Reed-Hill [137] has shown that for the angle between basal plane and stress axis near 90° , $\{10\bar{1}2\}$ twin is less likely to be activated because of the high resolved shear stress, and for this angle near 60° , $\{11\bar{2}1\}$ twin is favoured. However, quantitative calculations made by Akhtar [11] shows that for basal plane—stress axis angle near 90° , $\{10\bar{1}2\}$ twin is first activated although the shear stress is larger than that required for $\{11\bar{2}1\}$ twinning and for basal plane—stress axis angle near 60° plastic deformation does not initiate even when the CRSS reaches the value for $\{10\bar{1}2\}$ twins and $\{11\bar{2}1\}$ twinning starts at much higher CRSS level. The independency of deformation twinning on CRSS was proved by Beyerlerin et al. [130] through their statistical analysis of twinning in magnesium. A conclusion made was that

the observed twin variants do not necessarily have the highest Schmidt factor and twins did not all form in similar orientated grains. However, this analysis was based on global Schmid factor analysis, i.e. effect of external loading. In a polycrystalline material, the stress field build up within each grain could be very different from external loading [138], as a matter of fact recent 3D-XRD investigation [139] seems to suggest that CRSS do exist for twin nucleation based on stress analysis within individual twinned grain.

The twin nucleation mechanism can be either homogeneous or heterogeneous. The homogeneous twin nucleation mode was introduced by Orowan [140], who considered twins to nucleate and grow from a lenticular-shaped volume of uniform twin shear. This twin nucleation mode is not related with microstructural faults such as dislocations and grain boundaries. The heterogeneous twin nucleation mode was based on the zonal dislocation theory proposed by Mendelson [141], which considers the twin nucleus as consisting of a series of glissile partial dislocations, dissociated from matrix dislocation, on successive twin planes, forming a critical size [142]. The common feature of both nucleation mechanisms is that a suitable level of stress concentration is needed.

Wang et al. [136] proposed a slip-stimulated twin nucleation mechanism in commercial purity titanium. In this model, grains with their c axis nearly perpendicular to the applied stress are referred to as soft grains and grains with c axis parallel to stress direction are called hard grains. Deformation twins nucleate in hard grains through slip transfer from a neighbouring soft grain, as is shown in Figure 2.2.5:

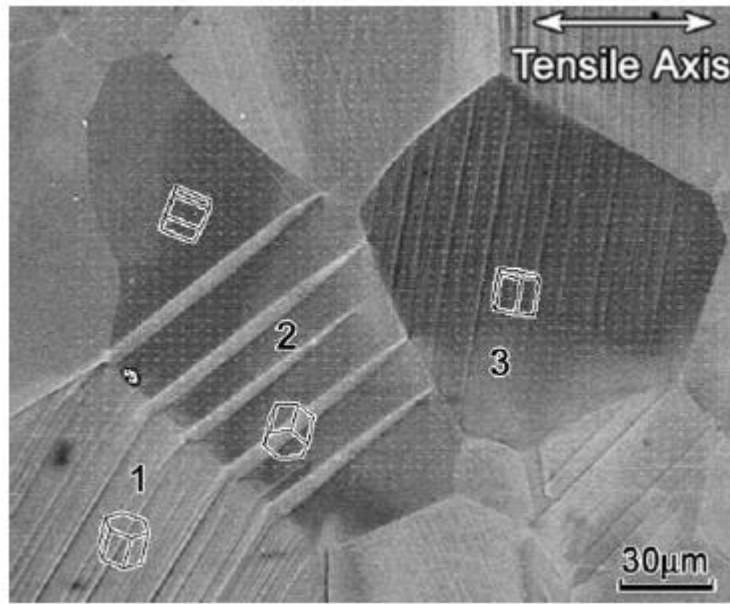


Figure 2.2.5. High resolution SEM image showing the phenomenon of slip assisted twin nucleation[136].

The twins are thicker at the point of slip-twin intersection between grain 1 and 2, and tapers at the intersection between grain 2 and 3. Therefore it was suggested that twins nucleated at the boundary of grain 1 and 2 and grow toward the boundary between grain 2 and 3. To assess the effectiveness of slip related twin nucleation, the Luster-Morris slip transfer parameter m' is calculated:

$$m' = \cos\psi \cdot \cos k \quad (2.21)$$

Where ψ is the angle between plane normal ($\mathbf{n_d}$) of slip in grain 1 and the twinning plane normal in grain 2, and k is the angle between Burger's vector ($\mathbf{b_d}$) of dislocation in grain 1 and twinning Burger's vector ($\mathbf{b_t}$), the geometric relationship between which can be shown in the following figure:

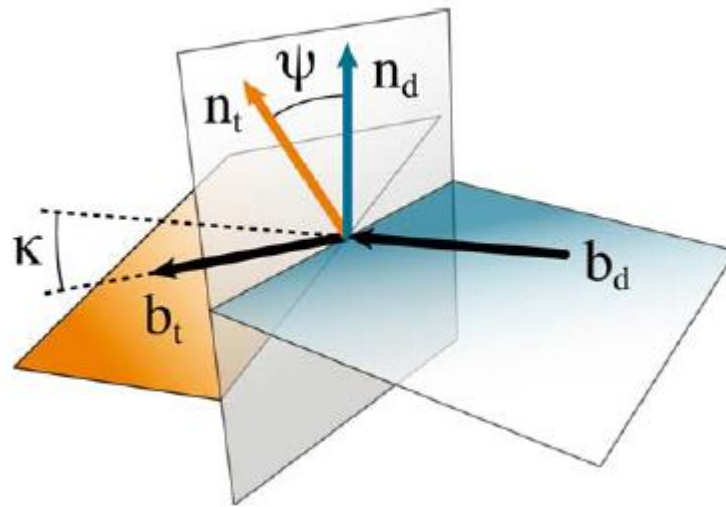


Figure 2.2.6. Geometric representation of the two angles k and ψ : $\mathbf{b}_d$ —Burger's vector of dislocation; $\mathbf{b}_t$ —Burger's vector of twin; $\mathbf{n}_d$ —normal of slip plane; $\mathbf{n}_t$ —normal of twinning plane [136].

It is obvious from figure 2.2.6 that the smaller the two angles, ψ and k , the better alignment the two adjacent deformation systems will be. Therefore larger m' value indicates easier activation of twinning in hard grains by slip transfer from neighbouring grains. Statistical calculations show high values of m' and global Schmidt factor, m_t , on the activated twin variant, for all the grain pairs that indicate a twin-by-slip transfer mechanism, but m' is a more important parameter for the mechanism because some deformation twins with lower m_t but highest m' for all the possible twinning systems were triggered by dislocation slip in neighbouring grains [136]. The dislocation stimulated twins only account for one third of all the twins in the experiment of Wang et al. [136], two more twinning cases were also found: some grains twin without observed dislocation activity in surrounding grains, others have twins in neighbouring grains forming common intersections at grain boundary.

Wang et al. [136] proposed that for the former case, twins were stimulated by dislocations beneath the surface of observation and therefore the m' rule still applies, but no further evidence was supplied to support this hypothesis. For the latter case, it was assumed that it has the same principle as slip induced twinning: twins were nucleated to accommodate the strain heterogeneity induced by twins in neighbouring grains [143]. The Luster-Morris parameter m' was calculated for all the observed twin-twin grain pairs and a generally good alignment between twin systems (high value of m') was found. However, for some twin pairs, each twin has a candidate twin system in the other grain with higher m' value than current twin pair but was not nucleated. Therefore the alignment of twinning system between neighbouring grains does not seem to be the only factor that affects the nucleation of twin-twin pairs.

Meyers et al. [8] developed a constitutive equation to describe the stress for twin nucleation. The theory hypothesises that dislocations pile up at a grain boundary, creating stress concentration necessary for twin initiation, where the level of this stress concentration depends on the size of pile up—the distance between dislocation source and barrier (grain boundary). However, numerical calculations show that the size of pile up is usually much smaller than the grain size, suggesting that other mechanisms are also responsible for the stress concentration necessary to initiate twinning.

It appears that although twinning activity has received significant amount of attention during the past few decades, the mechanism related to twin nucleation remains unclear, whilst suggestions for the nucleation mechanism are not conclusive.

Once formed, twins could affect mechanical property of the material by interacting with dislocations and other twins. Interaction between slip and twin leads to dislocation pile up at twin interface provided the type of interaction is repulsive, such as in Ti, Zr, Mg, and Be [135]. Stress concentration due to dislocation pile up may lead to the nucleation of twins of a different variant or may lead to the formation of cracks depending on the ductility of the metal. In ductile metals such as Ti and Zr, twin interface serves as an effective site for nucleation of different twins, while in brittle metal such as Be, crack initiation is typically observed at a twin interface.

Salem et al. [106, 144, 145] observed a three stage work hardening during compression of high purity alpha titanium with average grain size of 30 μm :

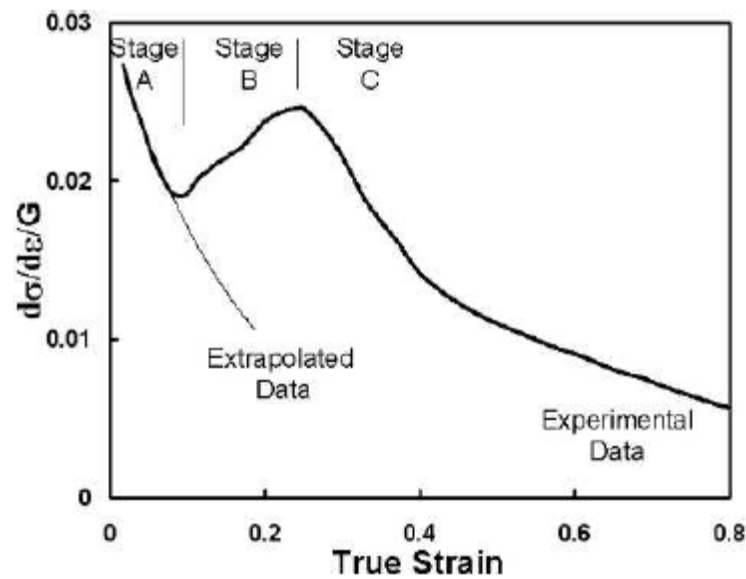


Figure 2.2.7: Three stage strain hardening as a function of strain during simple compression of high purity alpha titanium [145], $\frac{d\sigma/d\varepsilon}{G}$ is the strain hardening rate normalised by shear modulus G .

Twins start to nucleate at the end of stage A (0.11 true strain) and saturate towards the end of stage B (0.2 true strain). One of the contributions deformation twins could offer to stage B work hardening rate is to divide grains into smaller parts and so reduce the effective slip distance of dislocations. This can be described by Hall-Petch relations: $\sigma = \sigma_0 + k_y d^{-1/2}$. However, this relationship is not sufficient to explain the hardening behaviour at this stage. A hypothetical stress strain curve was extrapolated from stage A and extends to higher strain level into stage B assuming the material only deforms by slip. The reduction of grain size necessary to create the observed strain hardening effect was calculated using the Hall-Petch equation. Counting twin boundaries as grain boundaries, it was discovered that this calculated grain size is larger than the experimental observation, which mean there must be some softening effect associated with deformation twins at stage B [106]. Texture analysis revealed that c axis of the twinned region tend to be reoriented from initial hard orientations (c axis within 20° from the compression direction) to soft orientations (c axis about 85° from the compression direction), moreover, the Taylor factor for twinned region was found to be 40% lower than the parent. These indicate that dislocation slip was facilitated in twinned region due to the related texture change and therefore softening is indeed related to deformation twinning. Apart from the mechanisms stated above, a glissile-sessile transformation of dislocation in twinned area, as has been proposed by Basinski et al. [146], could also contribute to the hardening of stage B compressive deformation. To confirm the Basinski hardening proposal, Salem et al. [145] conducted nano-indentation testing on twinned regions and parents

separately. It was found that regardless of the orientation of twins and parent grains, twins are always harder than parent grains, providing indirect evidence for the Basinski hardening mechanism. Therefore, it was concluded that deformation twinning increases the strain hardening rate during compression test through the combined effect of Hall-Petch hardening, Basinski mechanism, and texture softening.

2.3 High Resolution Electron Backscatter Diffraction (HR-EBSD)

In a wide range of applications ranging from structural engineering components to semiconductor structures, the knowledge of the state and distribution of stress/strain field in localised regions in the materials is desirable. For example, a crack could initiate at twin/parent interface where the stress concentration has reached a critical level [135]; in semiconductors, strain accumulation could effectively lead to degradation of the device due to generation of dislocations [147]. Therefore, experimental techniques that can measure and map local elastic strain (stress) and dislocation distribution is desirable.

A variety of strain characterization techniques have been employed such as X-Ray and neutron diffraction, electron diffraction, Raman spectroscopy, cathode luminescence (CL). The spatial resolution and strain sensitivity of those techniques are shown in Figure 2.3.1.

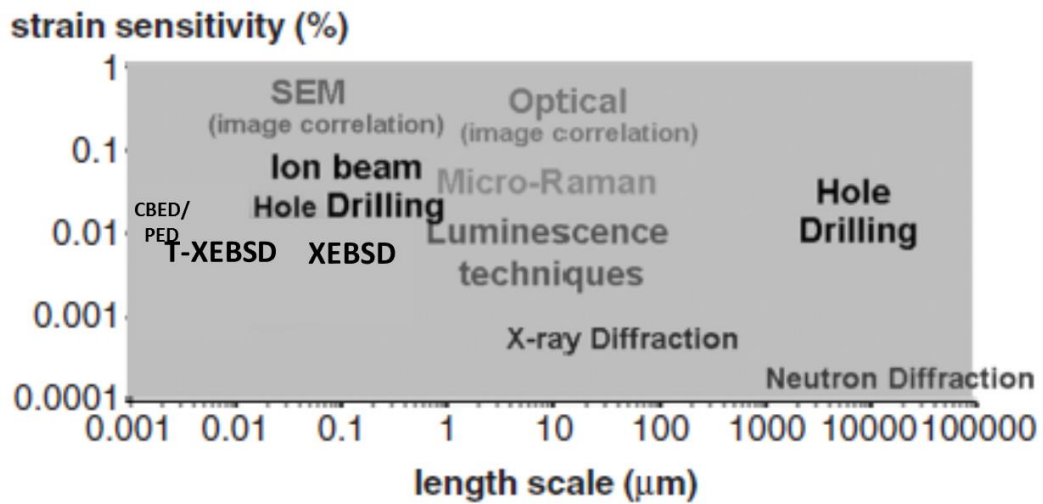


Figure 2.3.1: Strain sensitivity and spatial resolution related with different strain characterization methods [148].

Strain characterization using X-Ray [149] and neutron diffraction [150] show excellent strain sensitivity (strain of the order of 10^{-4} can be determined by X-Ray diffraction) but relatively poor spatial resolution. Recent development of high spatial resolution X-Ray strain measurement method include Differential Aperture X-Ray Laue Micro-diffraction (DAXM) which pushes the spatial resolution to 0.5 micron [77] and the Dark Field X-Ray Microscopy [151] which claims to have spatial resolution down to 0.1 micron. This instrument is currently been installed at beamline ID6 at ESRF. Hole drilling is poor at both strain sensitivity and spatial resolution thus the application is restricted to macroscopic regime. Although the spatial resolution of hole drilling method has been improved into nano regime by incorporating focused ion beam (FIB) based technique [152], the strain sensitivity is not improved. Spectroscopy based techniques such as CL [153] and micro-Raman [154] have spatial resolution of about one micron at best. Both techniques are based on strain

related peak shift and therefore cannot be used to determine full strain tensor [155]. The image correlation method is based on obtaining deformation field by comparing images taken before and after deformation. Strain sensitivity of 10^{-3} and spatial resolution into sub-micron scale was reported using high magnification SEM images [156], but strain information related with the state before first image was taken cannot be obtained. The ease of making fine electron beam plus the strong interaction between electron beam and mater automatically assign high spatial resolution to electron diffraction based strain measurement technique [155]. Transmission electron microscopy (TEM) based technique, typically convergent beam electron diffraction (CBED) [157], offers the highest spatial resolution but suffer from difficult sample preparation and strain relaxation due to small sample thicknesses. A new TEM based deformation gradient measurement technique, Precession Electron Diffraction (PED), has gained tremendous momentum in recent years [158, 159]. The orientation indexing is based on matching an experimental diffraction pattern with a pre-calculated pattern, akin to the EBSD method, and has been used to analyse GND evolution in nano-crystalline titanium with 1 nm spatial resolution [160]. Strain mapping using PED is also possible by diffraction pattern correlation similar to HR-EBSD [161]. The cross correlation based EBSD strain characterization technique (often called high (angular) resolution EBSD (HR-EBSD), is a relatively new but powerful technique that does not suffer so much from sample preparation problem whilst retaining the high spatial resolution electron diffraction techniques can offer [155].

The HR-EBSD technique measures elastic strain and lattice rotation by cross correlating two or more electron backscatter patterns (EBSP) for the small shifts of features between two patterns. It begins in the initial work by Troost et al. [162] and Wilkinson [163] and has been developed into commercial software, CrossCourt (BLG Productions Ltd, Bristol, UK), based on theories developed by Wilkinson et al. [164, 165]. Higher spatial resolution strain measurement using cross-correlation based EBSD method can be achieved in transmission mode (this is referred to as Transmission Kikuchi Diffraction (TKD) or T-EBSD) [166] using standard TEM lamellar, making strain and lattice rotation measurement at 10 nm domains more accessible. This concept has so far not been published but has been attempted [167].

2.3.1 Effect of strain on Kikuchi pattern

The formation of electron backscattered diffraction pattern (EBSP) is the result of the diffraction of the inelastically scattered electrons within the interaction volume between electron beam and the sample, which meets the Bragg condition with certain atomic plane [168]. A schematic of this process is shown below (the image is taken from reference [169]):

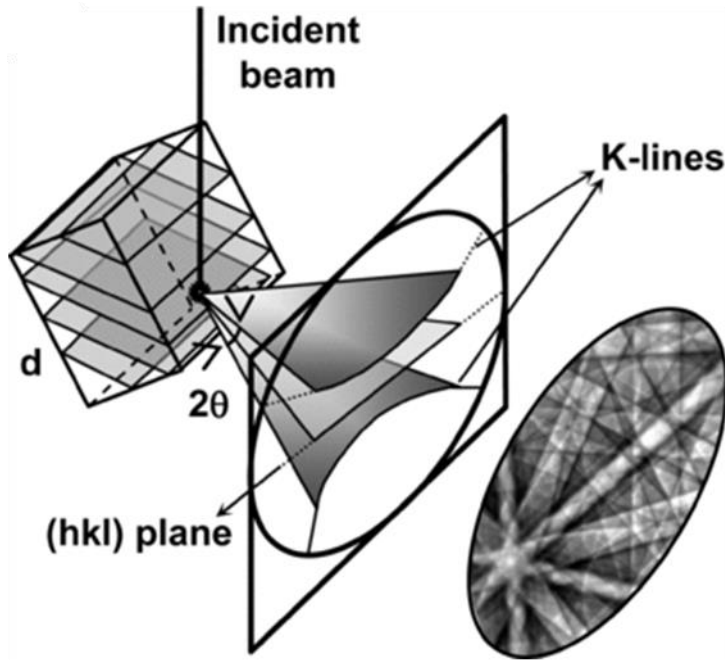


Illustration of the EBSP formation

Elastic strain changes the lattice spacing which further changes the band width in diffraction pattern according to Bragg's law, accompanied by a shift of the zone axis. If the lattice is bent, in addition to zone axis shift there will also be blurring of the edges of the diffraction bands [170], this can be visualized in figure 2.3.2:

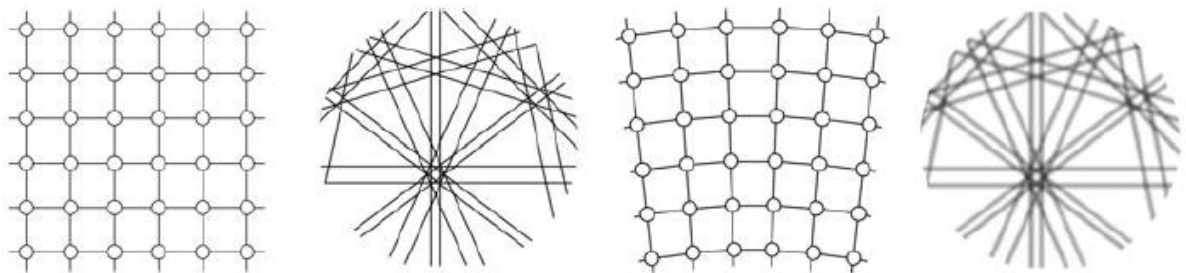


Figure 2.3.2: Crystal lattice and related diffraction pattern in the undistorted state (left) and distorted state (right) [171].

When there is plastic strain present, the distortion to crystal lattice is relieved by forming dislocations. Two types of dislocations can be formed as is shown in figure 2.3.3:

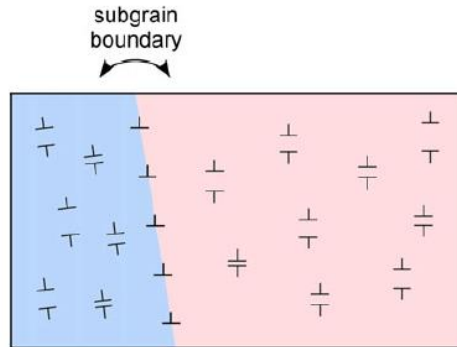


Figure 2.3.3: Schematic of the two types of dislocations and subgrain boundary [171].

As is shown in the above figure, there are regions with extensive amount of dislocations but they form pairs with net Burger's vectors of zero. They are called statistically stored dislocations (SSD) and lead to degradation of diffraction pattern due to local perturbations of diffraction lattice planes [171]. There are also dislocations whose Burger's vectors do not cancel out and the array of which form a low angle grain boundary. These dislocations are called geometrically necessary dislocations (GND). GNDs cause superposition of diffraction patterns from each grain across the subgrain boundary, therefore it also degrades diffraction pattern. Practically, it is impossible to differentiate the contribution of GNDs and SSDs to plastic deformation [171], but the GND content can be found using lattice rotation gradients calculated by the method described in the following section.

2.3.2 Linking pattern shift to strain

The method was developed by Wilkinson et al. [155, 163-165, 172] with further refinement made by Britton and Wilkinson [172, 173]. The following is a generalized description.

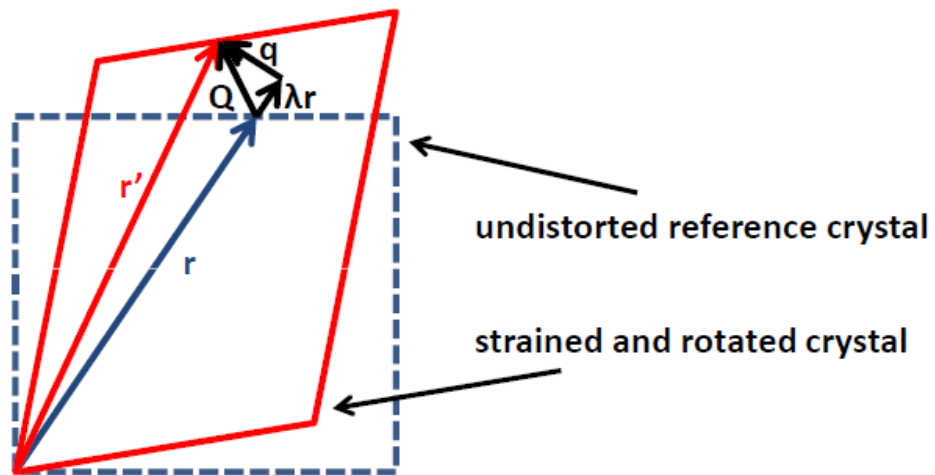


Figure 2.3.4: Schematic diagram showing exaggerated zone axis (r) shift from undeformed state (blue) to deformed state (red), adopted from [155].

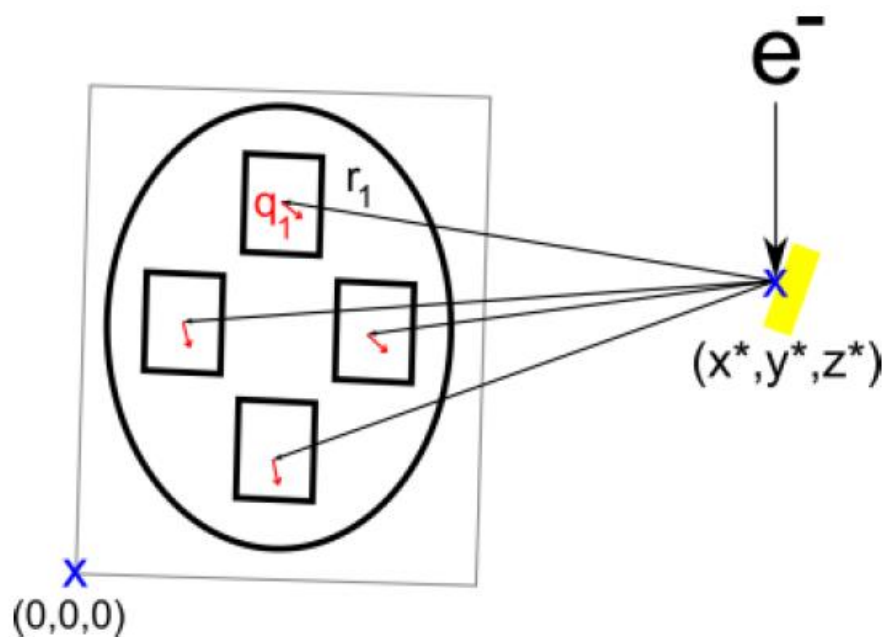


Figure 2.3.5: 3D representations of the parameters r and q for high resolution EBSD analysis. (x^*, y^*, z^*) represents source point (where electron beam lights the sample) [172].

As is shown in Figure 2.3.4, an arbitrary strain and rigid body rotation change a vector $\mathbf{r}$ from reference crystal to $\mathbf{r}'$ in deformed crystal. The change from $\mathbf{r}$ to $\mathbf{r}'$ can be described by a strain gradient tensor $\mathbf{A}$:

$$\mathbf{A} - \mathbf{I} = \begin{pmatrix} \frac{\partial u_1}{\partial x_1} & \frac{\partial u_1}{\partial x_2} & \frac{\partial u_1}{\partial x_3} \\ \frac{\partial u_2}{\partial x_1} & \frac{\partial u_2}{\partial x_2} & \frac{\partial u_2}{\partial x_3} \\ \frac{\partial u_3}{\partial x_1} & \frac{\partial u_3}{\partial x_2} & \frac{\partial u_3}{\partial x_3} \end{pmatrix} \quad (2.22)$$

Where $\mathbf{u} = (u_1 \ u_2 \ u_3)$ is the displacement at position $\mathbf{x} = (x_1 \ x_2 \ x_3)$ and $\mathbf{I}$ is identity matrix.

Therefore

$$\mathbf{r}' = \mathbf{A}\mathbf{r} \quad (2.23)$$

The strain and rotation lead to displacement $\mathbf{Q}$:

$$\mathbf{Q} = \mathbf{r}' - \mathbf{r} = (\mathbf{A} - \mathbf{I})\mathbf{r} \quad (2.24)$$

The EBSD technique can only measure the projection of $\mathbf{Q}$ onto the phosphor screen, which is represented by $\mathbf{q}$:

$$\mathbf{q} = \mathbf{Q} - \lambda\mathbf{r} = \{\mathbf{A} - (\lambda + 1)\mathbf{I}\}\mathbf{r} \quad (2.25)$$

The component of the vector $\mathbf{q}$ can be calculated using the above equation and some mathematical manipulation [148]:

$$q_1 = r_1 e_{xx} + r_2 e_{xy} + r_3 e_{xz} - \lambda r_1 \quad (2.26)$$

$$q_2 = r_1 e_{yx} + r_2 e_{yy} + r_3 e_{yz} - \lambda r_2 \quad (2.27)$$

$$q_3 = r_1 e_{zx} + r_2 e_{zy} + r_3 e_{zz} - \lambda r_3 \quad (2.28)$$

Where e_{ij} is the displacement gradient described in equation (2.22), and λ is an unknown constant.

The constant λ can be eliminated by combining pairs of the above equations and yields two equations in eight variables:

$$r_1 r_3 \left[\frac{\partial u_1}{\partial x_1} - \frac{\partial u_3}{\partial x_3} \right] + r_2 r_3 \frac{\partial u_1}{\partial x_2} + r_3^2 \frac{\partial u_1}{\partial x_3} - r_1^2 \frac{\partial u_3}{\partial x_1} - r_2 r_1 \frac{\partial u_3}{\partial x_2} = r_3 q_1 - r_1 q_3 \quad (2.29)$$

$$r_2 r_3 \left[\frac{\partial u_2}{\partial x_2} - \frac{\partial u_3}{\partial x_3} \right] + r_1 r_3 \frac{\partial u_2}{\partial x_1} + r_3^2 \frac{\partial u_2}{\partial x_3} - r_1 r_2 \frac{\partial u_3}{\partial x_1} - r_2^2 \frac{\partial u_3}{\partial x_2} = r_3 q_2 - r_2 q_3 \quad (2.30)$$

Because shifts are only measured in the phosphor screen, so $Q_3=0$, therefore, from equation (2.25):

$$q_3 = -\lambda r_3 \quad (2.31)$$

The right side of equations (2.29) and (2.30) can then be written as:

$$r_3 q_1 - r_1 q_3 = r_3 (q_1 + \lambda r_1) \quad (2.32)$$

$$r_3 q_2 - r_2 q_3 = r_3 (q_2 + \lambda r_2) \quad (2.33)$$

Equation (2.24) can be rearranged as:

$$\mathbf{Q} = \mathbf{q} + \lambda \mathbf{r} \quad (2.34)$$

Substitute equation (2.34) into equation (2.32) and (2.33):

$$r_3 q_1 - r_1 q_3 = r_3 Q_1 \quad (2.35)$$

$$r_3 q_2 - r_2 q_3 = r_3 Q_2 \quad (2.36)$$

Substitute (2.35) and (2.36) into (2.29) and (2.30), and divide the resultant equation by r_3 leads to:

$$r_1 \left[\frac{\partial u_1}{\partial x_1} - \frac{\partial u_3}{\partial x_3} \right] + r_2 \frac{\partial u_1}{\partial x_2} + r_3 \frac{\partial u_1}{\partial x_3} - \frac{r_1^2}{r_3} \frac{\partial u_3}{\partial x_1} - \frac{r_2 r_1}{r_3} \frac{\partial u_3}{\partial x_2} = Q_1 \quad (2.37)$$

$$r_2 \left[\frac{\partial u_2}{\partial x_2} - \frac{\partial u_3}{\partial x_3} \right] + r_1 \frac{\partial u_2}{\partial x_1} + r_3 \frac{\partial u_2}{\partial x_3} - \frac{r_1 r_2}{r_3} \frac{\partial u_3}{\partial x_1} - \frac{r_2^2}{r_3} \frac{\partial u_3}{\partial x_2} = Q_2 \quad (2.38)$$

Equations (2.37) and (2.38) can be written in matrix form as following:

$$\begin{pmatrix} r_1 & r_2 & r_3 & 0 & 0 & 0 & -\frac{r_1^2}{r_3} & -\frac{r_1 r_2}{r_3} \\ 0 & 0 & 0 & r_1 & r_2 & r_3 & -\frac{r_1 r_2}{r_3} & -\frac{r_2^2}{r_3} \end{pmatrix} \begin{pmatrix} \left[\frac{\partial u_1}{\partial x_1} - \frac{\partial u_3}{\partial x_3} \right] \\ \frac{\partial u_1}{\partial x_2} \\ \frac{\partial u_1}{\partial x_3} \\ \frac{\partial u_2}{\partial x_1} \\ \left[\frac{\partial u_2}{\partial x_2} - \frac{\partial u_3}{\partial x_3} \right] \\ \frac{\partial u_2}{\partial x_3} \\ \frac{\partial u_3}{\partial x_1} \\ \frac{\partial u_3}{\partial x_2} \end{pmatrix} = \begin{pmatrix} Q_1 \\ Q_2 \end{pmatrix} \quad (2.39)$$

In this equation, the left part ($\mathbf{r}$) is the vector from source point to centre of subset on phosphor screen (as is shown in figure 2.3.5) and is only related with calibration parameter and the position of subset; therefore it is constant for each subset once the pattern centre is known. The part to the right of the equation, Q_i , is the pattern shift for each subset that can be measured. The middle part contains 8 unknowns, therefore once four or more subsets are

measured, values of $\left[\frac{\partial u_1}{\partial x_1} - \frac{\partial u_3}{\partial x_3}\right]$, $\frac{\partial u_1}{\partial x_2}$, $\frac{\partial u_1}{\partial x_3}$, $\frac{\partial u_2}{\partial x_1}$, $\left[\frac{\partial u_2}{\partial x_2} - \frac{\partial u_3}{\partial x_3}\right]$, $\frac{\partial u_2}{\partial x_3}$, $\frac{\partial u_3}{\partial x_1}$, $\frac{\partial u_3}{\partial x_2}$ can be determined. Normal components of the displacement gradient tensor (equation 2.22) can be calculated by assuming the normal stress perpendicular to sample surface is zero and Hooke's law to write the stress in terms of strains and elastic constants, then an additional equation can be generated, which combined with the calculated values of $\left[\frac{\partial u_1}{\partial x_1} - \frac{\partial u_3}{\partial x_3}\right]$ and $\left[\frac{\partial u_2}{\partial x_2} - \frac{\partial u_3}{\partial x_3}\right]$ allows the normal components to be calculated.

The above procedure allows the calculation of the displacement gradient tensor. This tensor can be split into symmetric and asymmetric parts that represent elastic strains ε_{ij} and rotations w_{ij} respectively:

$$\text{Elastic normal strains: } \varepsilon_{ii} = \frac{\partial u_i}{\partial x_i} \quad (2.40)$$

$$\text{Elastic shear strains: } \varepsilon_{ij} = \frac{1}{2} \left(\frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i} \right) \quad (2.41)$$

$$\text{Lattice rotations: } w_{ij} = \frac{1}{2} \left(\frac{\partial u_i}{\partial x_j} - \frac{\partial u_j}{\partial x_i} \right) \quad (2.42)$$

2.3.3 GND calculation

The effect of the plastic strain history within the sample can be assessed in the form of the content of geometrically necessary dislocations (GND). With the knowledge of the three rotation components calculated using equation (2.42), it is possible to link the GND density to Nye's tensor [174]. The procedure starts

with a physically based model that displacement $\mathbf{u}$ is continuous over sample surface [175] such that:

$$\oint_C d\mathbf{u} = 0 \quad (2.43)$$

Where $d\mathbf{u}$ is linked to displacement gradient tensor $\mathbf{A}$ by $\mathbf{A} = \frac{d\mathbf{u}}{dx}$, which can be split into an elastic part $\mathbf{A}^e$ and a plastic part $\mathbf{A}^p$. This together with Stoke's theorem give rise to the following equation:

$$\oint_C \mathbf{A} dx = \iint_S \text{curl}(\mathbf{A}) ds = \iint_S \text{curl}(\mathbf{A}^e + \mathbf{A}^p) ds = 0 \quad (2.44)$$

Therefore curl of the elastic and plastic displacement gradient must have opposite sign:

$$\text{curl}(\mathbf{A}^e) = -\text{curl}(\mathbf{A}^p) \quad (2.45)$$

The net Burger's vector over a surface patch S is defined as:

$$\mathbf{B} = -\iint_S \text{curl}(\mathbf{A}^p) ds \quad (2.46)$$

And the Nye's tensor [174], $\boldsymbol{\alpha}$, is defined as:

$$\mathbf{B} = \iint_S \boldsymbol{\alpha} ds \quad (2.47)$$

Equating the right hand side of equation 2.46 and 2.47 gives:

$$\boldsymbol{\alpha} = -\text{curl}(\mathbf{A}^p) = \text{curl}(\mathbf{A}^e) \quad (2.48)$$

Wilkinson and Randman [176] have shown that Nye's tensor $\boldsymbol{\alpha}$ takes the following form as a summation of rotation and elastic strain gradients:

$$\begin{aligned}
\boldsymbol{\alpha} = & \begin{pmatrix} \frac{\partial \omega_{12}}{\partial x_3} - \frac{\partial \omega_{13}}{\partial x_2} & \frac{\partial \omega_{13}}{\partial x_1} & \frac{\partial \omega_{21}}{\partial x_1} \\ \frac{\partial \omega_{32}}{\partial x_2} & \frac{\partial \omega_{23}}{\partial x_1} - \frac{\partial \omega_{21}}{\partial x_3} & \frac{\partial \omega_{21}}{\partial x_2} \\ \frac{\partial \omega_{32}}{\partial x_3} & \frac{\partial \omega_{13}}{\partial x_3} & \frac{\partial \omega_{31}}{\partial x_2} - \frac{\partial \omega_{32}}{\partial x_1} \end{pmatrix} \\
& + \begin{pmatrix} \frac{\partial \varepsilon_{12}}{\partial x_3} - \frac{\partial \varepsilon_{13}}{\partial x_2} & \frac{\partial \varepsilon_{13}}{\partial x_1} - \frac{\partial \varepsilon_{11}}{\partial x_3} & \frac{\partial \varepsilon_{11}}{\partial x_2} - \frac{\partial \varepsilon_{12}}{\partial x_1} \\ \frac{\partial \varepsilon_{22}}{\partial x_3} - \frac{\partial \varepsilon_{23}}{\partial x_2} & \frac{\partial \varepsilon_{23}}{\partial x_1} - \frac{\partial \varepsilon_{21}}{\partial x_3} & \frac{\partial \varepsilon_{21}}{\partial x_2} - \frac{\partial \varepsilon_{22}}{\partial x_1} \\ \frac{\partial \varepsilon_{32}}{\partial x_3} - \frac{\partial \varepsilon_{33}}{\partial x_2} & \frac{\partial \varepsilon_{33}}{\partial x_1} - \frac{\partial \varepsilon_{31}}{\partial x_3} & \frac{\partial \varepsilon_{31}}{\partial x_2} - \frac{\partial \varepsilon_{32}}{\partial x_1} \end{pmatrix} \quad (2.49)
\end{aligned}$$

As HR-EBSD measures only on the flat surface (x_1, x_2) of the sample so that components in $\boldsymbol{\alpha}$ containing gradient in the x_3 direction is not accessible (coloured red in equation 2.49). If the lattice rotation gradient is significantly larger than the elastic strain gradient [176], the elastic strain gradient can be set to zero, leaving 5 out of 9 components of the Nye tensor plus $a_{11} - a_{22} = \frac{\partial \omega_{31}}{\partial x_2} - \frac{\partial \omega_{23}}{\partial x_1}$ that can be calculated.

The components of Nye's tensor are connected to different types of dislocations on all possible slip systems through:

$$\boldsymbol{\alpha}_{ij} = \sum_s \rho_s \mathbf{b}_i^s \mathbf{l}_j^s \quad (2.50)$$

Where ρ_s is the density of the s^{th} type of dislocation and $\mathbf{b}^s$ and $\mathbf{l}^s$ are its Burger's vector and unit line direction respectively. Equation 2.50 was rewritten by Wilkinson et al [176] in the following form:

$$\mathbf{A}\boldsymbol{\rho} = \boldsymbol{\Lambda} \quad (2.51)$$

Where dislocation densities are represented by a column vector $\boldsymbol{\rho}$ (m by 1, m depends on the number of possible dislocation types). 6 of the measured lattice rotation gradient make up the column vector $\boldsymbol{\Lambda}$, and the s^{th} column of matrix $\mathbf{A}$ contains the tensor product of the Burger's vector and line direction of the s^{th} type of dislocation that gives rise to the observed lattice curvature. Therefore, equation 2.51 can be written in the following detailed form [111]:

$$\begin{pmatrix} b_1^1 l_1^1 - \frac{1}{2} \mathbf{b}^1 \mathbf{l}^1 & . & b_1^1 l_1^1 - \frac{1}{2} \mathbf{b}^1 \mathbf{l}^1 \\ b_1^1 l_2^1 & . & b_1^1 l_2^1 \\ b_1^1 l_3^1 & . & b_1^s l_3^s \\ b_2^1 l_1^1 & . & b_2^s l_1^s \\ b_2^1 l_2^1 - \frac{1}{2} \mathbf{b}^1 \mathbf{l}^1 & . & b_2^s l_2^s - \frac{1}{2} \mathbf{b}^s \mathbf{l}^s \\ b_2^1 l_3^1 & . & b_2^s l_3^s \end{pmatrix} \begin{pmatrix} \rho_1 \\ . \\ \rho_s \end{pmatrix} = \begin{pmatrix} \frac{\partial \omega_{23}}{\partial x_1} \\ \frac{\partial \omega_{31}}{\partial x_1} \\ \frac{\partial \omega_{12}}{\partial x_1} \\ \frac{\partial \omega_{23}}{\partial x_2} \\ \frac{\partial \omega_{31}}{\partial x_2} \\ \frac{\partial \omega_{12}}{\partial x_2} \end{pmatrix} \quad (2.52)$$

2.3.4 Measuring small pattern shift in HR-EBSD

In order to accurately determine pattern shift using cross correlation based method, a series of high quality EBSD patterns must be obtained, the detailed technical parameters can be found in [148, 155, 164, 165, 177]. The pattern of the highest quality in the series was chosen as the reference and compared

with other patterns in the series for pattern shifts. To do this, each pattern is divided into several widely dispersed regions of interests (ROI). It was found that 20 ROIs decrease the standard deviation of displacement gradient tensor to 10^{-4} [164]; a larger ROI size increase the accuracy of measurement but smaller ROI improves computational efficiency and is preferred when strains and rotations cause non-uniform shifts across the pattern [165]. A typical ROI of 256x256 pixels for a 1000x1000 pixel Kikuchi pattern were used in the research by Wilkinson et al. [164, 165].

For each ROI, the mean intensity is calculated and then subtracted from every pixel. The resulting intensity is then multiplied by a weighting function:

$$f(x, y) = \cos\left(\frac{\pi x}{256}\right)\cos\left(\frac{\pi y}{256}\right) \quad (2.53)$$

Where (x, y) is the position of the pixel relative to the ROI centre.

This weighting function brings the intensity at edge of the ROI to zero, as is shown in figure 2.3.6 (c), as well as preventing aliasing and leakage problems when fast Fourier transforms are calculated for each region.

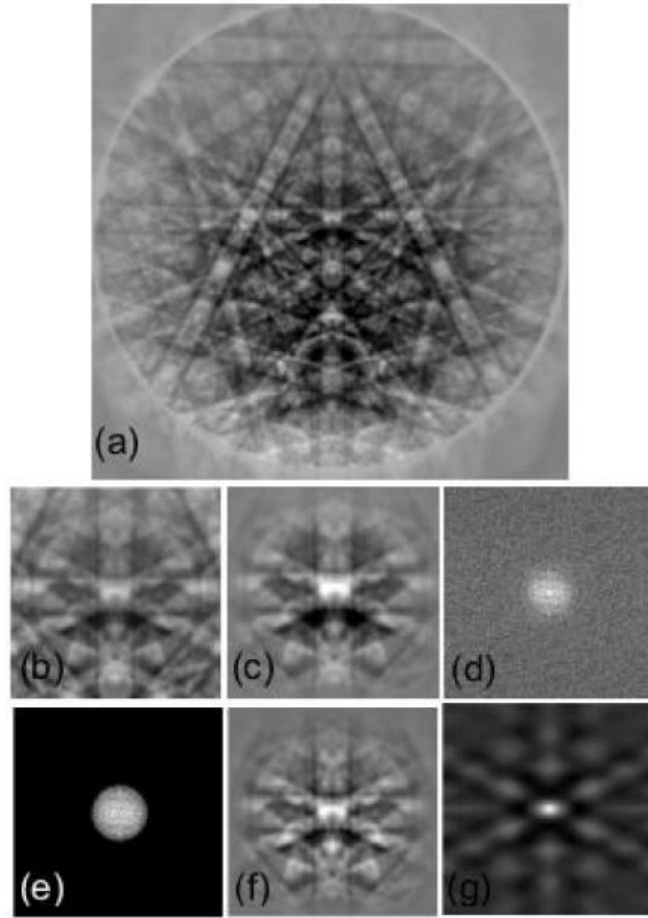


Figure 2.3.6: (a) An EBSD pattern obtained from GaN, (b) a region of interest subtracted for analysis, (c) diffraction pattern after applying the weighting function, (d) fast Fourier transform of (c), (e) pattern in fourier domain after filtering, (f) back transfer to real domain from (e), (g) cross correlation with a reference pattern processed by the same procedure.

The reason for transforming diffraction pattern to Fourier domain is that it is more computational efficient to calculate cross correlation function at this ROI size. In the Fourier domain, the pattern is filtered by a high pass frequency filter to remove low frequency components and long range back ground gradient and a low pass frequency filter to remove high frequency fluctuations associated

with noise of the CCD readout [165]. The ROI after filtering is shown in figure 2.3.6 (e) and (f).

The normalized cross correlation function (χ) can be calculated by comparing patterns in Fourier domain:

$$X^2 = \frac{\sum FG}{\sqrt{\sum F^2 \sum G^2}} \quad (2.54)$$

Where F is the FFT of the reference and G is the FFT of the test ROI. The cross correlation function is represented as an intensity distribution, as is shown in figure 2.3.6 (g), where the intensity at each point represents the degree of correlation. Therefore the point with maximum intensity is the best correlation between two patterns. The height of the cross correlation peak is affected by surface contamination, surface topography, and extent of plastic deformation near surface. Therefore the quality of data produced can be assessed by taking geometric mean of normalised peak heights for all ROIs on a pattern, and a mean value below ~0.25 indicates problems in the test pattern [155].

Chapter 3 Slip band and grain boundary interactions in commercial purity titanium

3.1 Materials and experimental procedures

The material used in this research was commercial purity titanium supplied by Timet UK Ltd (Birmingham). The chemical composition is listed in Table 3.1 [111]. Tensile test pieces with a gauge dimension of 30mm x 3mm x 1mm were cut using electron discharge machining. The long axis of the tensile piece was along the rolling direction of the as received bar. The tensile pieces were heat treated in vacuum at 830°C for 24 hours to grow the grain size to an average of ~300 μm . Samples were then ground using silicon carbide paper down to 4000 grit followed by chemical-mechanical polishing using 50nm colloidal silica suspension.

Caution needed to be taken here regarding polishing CP-Ti to a good enough surface finish for high quality EBSD patterns. Polish using colloidal silica alone will give a mirror surface finish but this won't yield an analysable Kikuchi pattern. A 'clean' grain structure was not revealed under optical microscope with polarized illumination, the surface was observed to be covered with speckle objects that could not be removed by further colloidal silica polishing. This problem can be solved by etching the polished sample using a solution with 1% HF and 10% HNO_3 in water to remove the surface contamination layer. This polish-etch-polish process was repeated until a crisp grain boundary structure was visible in optical microscope with polarized illumination and high quality diffraction pattern be observed in SEM. Another point to be noted regarding

sample grinding is that due to the abnormally large sample surface area, machine grinding will usually leave the sample surface with unequal scratch distribution, which will affect the efficiency of the subsequent grinding processes. Unidirectional hand polish with intermittent application of 90% colloidal silica + 10% hydrogen peroxide was found to yield better results.

Element	Fe	O ₂	N ₂	C	Ti
Composition	0.35 wt%	700ppm	35ppm	0.01 wt%	Balance

Table 3.1. Chemical composition of the as received grade 1 commercially pure titanium [111].

The polished sample surface was patterned with carbon particles (~3 µm size, from wasted toner cartridge) which was fused to the surface by heating to 100°C. This is to make sure that the sample surface was covered by random patterns suitable for digital image correlation (DIC) and that they are firmly stuck onto the surface. This procedure, however, leaves sub-micron scale surface traces that is visible in SEM (but not in optical microscopy) and could not be easily washed off.

The sample was deformed in tension to 1% plastic strain measured by in-situ digital image correlation. The applied stress reached a maximum value of 235 MPa. EBSD maps featuring various slip band/grain boundary interactions were acquired including a number of boundaries with blocked slip bands and a number of boundaries with transferred slip bands using JOEL JSM 6500

scanning electron microscopy. The maps had an average size of 10 μm x 25 μm and were obtained at 20 KV with 14 nA beam current over a square array of 0.2 μm step size. At each interrogation point the back-scattered electron diffraction pattern, consisting of 1000 x 1000 pixels, was captured and saved as TIFF image format with 12-bit depth using OIM Data Collection V5.

For cross correlation, the patterns were transformed into the Fourier domain and filtered to remove high frequency noise and low frequency background gradients. For each grain a reference point was picked towards the interior of the grain, where it was assumed that the stress state was relatively low and uniform. For each pattern, 50 regions of interest with 256 x 256 pixel size were selected with one at the middle of the pattern, 19 others surrounding it in a ring and the remaining 30 randomly distributed. These region of interests on test patterns were compared with the corresponding parts on the reference pattern using cross-correlation [178] with which pattern shifts as small as 0.02 pixels can be detected [179]. These pattern shifts were used to fully determine the displacement gradient tensor (DGT) using the assumption that the stress perpendicular to the sample surface is zero [180]. Locally the lattice rotations could be quite large and so the pattern remapping algorithm developed by Britton and Wilkinson [113] was used. This involves using a first pass of cross-correlation analysis to estimate a finite lattice rotation which is used to remap the test pattern intensities in a virtual reorientation of the test region. A second pass of cross correlation analysis is then used to determine the lattice strains and a correction to the lattice rotation. The DGT from the first and second passes are combined to give a total deformation gradient $\mathbf{F}$ from which the

Green strain tensor ($\mathbf{E}$) was obtained: $\mathbf{E} = \frac{1}{2} (\mathbf{F}^T \mathbf{F} - \mathbf{I})$. These elastic strain fields were then used to calculate stress fields using elastic constants (in GPa) for titanium: $C_{11} = 162.4$ $C_{33} = 180.7$ $C_{44} = 117$ $C_{66} = 35.2$ $C_{13} = 69.0$ [181]. Polar decomposition of $\mathbf{F}$ into the product of rotation and stretch tensors using singular value decomposition methods enables the lattice rotation to be determined. These finite rotation tensor were used to estimate 6 infinitesimal rotation gradients using procedures documented in [23] and linked to 6 out of 9 components of Nye's dislocation tensor [174].

The GND density presented in this paper is a lower-bound estimation. For GND calculation of α -Ti the following dislocation types were used: 3 $\langle a \rangle$ screw and 6 $\langle c+a \rangle$ screw; 3 $\langle a \rangle$ edge on basal plane; 3 $\langle a \rangle$ edge on first order prismatic plane; 6 $\langle a \rangle$ edge on first order pyramidal plane; 12 $\langle c+a \rangle$ edge on first order pyramidal plane [182]. The GND density for each type of dislocation is found using a standard linear programming algorithm by minimizing the possible total GND line energy [175].

It is important to note that the absolute strain state of the reference pattern is unknown. Therefore the elastic strains presented here are relative values. However, this uncertainty does not affect the GND density as it is only related with lattice curvatures [176].

The EBSD code initially reports stresses in the external sample reference frame. For slip band/grain boundary interactions that lead to stress concentration these stresses were rotated such that the shear stress on the slip plane of the deforming grain in the Burger's vector direction was obtained across the two

grains. From the rotated resolved shear stress field, line profiles were extracted along the direction of the pile-up. The stress intensity factor K was then determined by fitting the stress line profile using non-linear method to the equation proposed in [111]:

$$\sigma_{31}^r = A + K/\sqrt{X + B} \quad (3.1)$$

Where σ_{31}^r is the resolved shear stress imposed by the dislocation pile-up in the neighbouring grain, X is the distance along the line profile away from grain boundary. The constant A is introduced to allow for uncertainty of stress state at the reference point while the constant B is introduced to allow for the uncertainty of grain boundary position.

For a number of the blocked slip bands atomic force microscopy (AFM) was used to determine the height of the slip bands. A Pacific Nanotechnology Nano-R with SPM Cockpit model AFM was used in contact mode to map the surface topography over regions $45\mu\text{m}$ by $45\mu\text{m}$. Images were analysed on-line and topography changes across the slip band averaged over several points along the slip band.

3.2 Slip trace analysis and stress tensor rotation

The EBSD data collection and orientation analysis software used in this analysis (OIM EDAX) fix a Cartesian coordinate system onto a hexagonal crystal in the following manner:

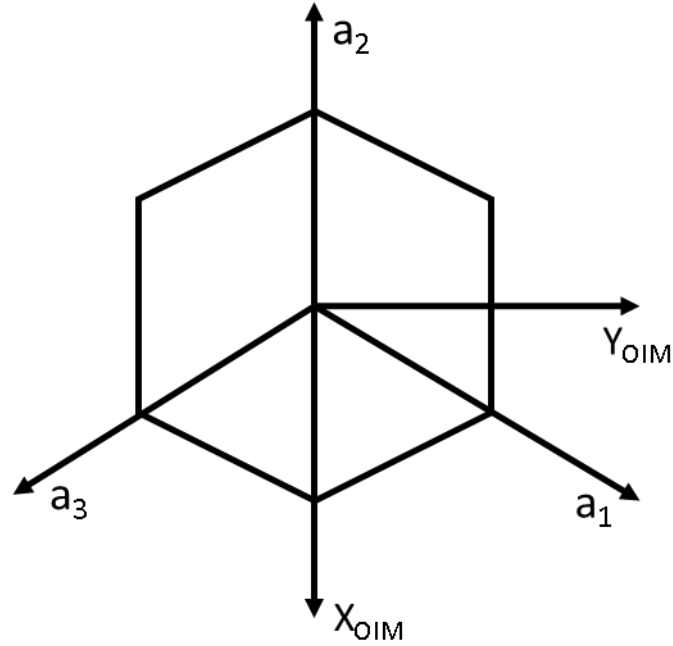


Figure 3.1: Top-down view of the definition of a Cartesian coordinate system on a hexagonal crystal used in OIM software.

Figure 3.1 demonstrates the position of OIM Cartesian reference system (X_{OIM} , Y_{OIM}) relative to the crystal reference frame (a_1 a_2 a_3) onto which miller indices was defined. Once the Miller-Bravais indices on crystal reference frame (h k i l) are known, they can be transformed onto the indicated OIM convention using the following transformation matrix:

$$T = a \begin{pmatrix} \cos 60^\circ & \cos 60^\circ & 0 \\ -\sin 60^\circ & \sin 60^\circ & 0 \\ 0 & 0 & c/a \end{pmatrix} \quad (3.2)$$

Any vectors $\mathbf{v}_c$ referenced in the $(a_1 \ a_2 \ a_3)$ coordinate system can be transformed into (X_{OIM}, Y_{OIM}) coordinate system $\mathbf{v}_t$ through the operation:

$$\mathbf{v}_t = [\mathbf{T}]\mathbf{v}_c \quad (3.3)$$

However, in non-cubic crystal systems the crystal plane and its normal do not share the same indices [183]. In order to find the plane normal from its Miller indices the transformation matrix has to be transformed into reciprocal space. If the first, second and third column of the transformation matrix $\mathbf{T}$ is designated a , b , and c respectively, then 3 vectors in reciprocal space can be defined:

$$\mathbf{a}^* = \frac{\mathbf{b} \times \mathbf{c}}{V} \quad \mathbf{b}^* = \frac{\mathbf{c} \times \mathbf{a}}{V} \quad \mathbf{c}^* = \frac{\mathbf{a} \times \mathbf{b}}{V} \quad (3.4)$$

Where V is the unit cell volume and a second transformation matrix can be defined:

$$\mathbf{T}_R = \begin{pmatrix} \mathbf{a}_1^* & \mathbf{b}_1^* & \mathbf{c}_1^* \\ \mathbf{a}_2^* & \mathbf{b}_2^* & \mathbf{c}_2^* \\ \mathbf{a}_3^* & \mathbf{b}_3^* & \mathbf{c}_3^* \end{pmatrix} \quad (3.5)$$

Any slip plane with the indices $(h \ k \ l)$ in $(a_1 \ a_2 \ a_3)$ reference system can now be transformed to (X_{OIM}, Y_{OIM}) reference system through the operation:

$$\begin{pmatrix} x \\ y \\ z \end{pmatrix}_{OIM} = [\mathbf{T}_R] \begin{pmatrix} h \\ k \\ l \end{pmatrix} \quad (3.6)$$

The slip plane normal coordinates in OIM reference frame found using the above procedures are then rotated to sample reference frame using rotation matrix constructed from measured Euler angles. These are then cross product with the sample surface normal $(0 \ 0 \ 1)$ to form the trace of the slip plane and

the one that coincide with the observed slip band is identified as the active slip plane. As HCP crystals naturally have lower degree of the symmetry, the plane traces are in most cases very distinguished from each other. In cases where multiple slip traces converge with the observed slip trace the active slip trace was taken as the one having lower CRSS and higher Schmid factor.

Once the active slip plane is identified a rotation matrix can be constructed:

$$R_{\text{slip plane}} = \begin{pmatrix} b_1 & l_1 & n_1 \\ b_2 & l_2 & n_2 \\ b_3 & l_3 & n_3 \end{pmatrix} \quad (3.7)$$

Where b_i and l_i are the unit Burger's vector and line direction of an edge dislocation on the identified slip plane and n_i are their cross product. This choice of rotation matrix defines a Cartesian reference frame on the active slip plane such that x axis corresponds to the Burger's vector of an edge dislocation, y axis the line direction, and z direction the plane normal (see Figure 3.2). This construction of the rotation matrix have the convenience that the σ_{13} component of the stress tensor after rotation converge with the shear direction of the active slip system.

Stress tensor as mapped on sample surface can then be rotated onto the active slip plane by the operation:

$$\sigma_{ij}^r = R_{\text{slip plane}} * \sigma_{ij} * R_{\text{slip plane}}^T \quad (3.8)$$

3.3 Results

Analysing the HR-EBSD data sampled from large populations of slip band-grain boundary intersections lead to an identification of three broad categories of slip band-grain boundary interactions. These were:

- (1) Blocked slip band with a stress concentration ahead of it;
- (2) Slip transfer where the slip band in one grain leads directly to a slip band in the neighbouring grain;
- (3) Blocked slip band with no long range slip band in the neighbouring grain but no obvious stress concentration.

The results for each category is shown in the following sections.

3.3.1 Blocked slip band that lead to stress concentration

This type of interaction features a blocked slip band in one grain with no macroscopic slip transfer into the neighboring grain, observed from surface topography. Localized stress concentration was observed around the point of intersection between slip band and grain boundary using HR-EBSD.

One example of such interactions is shown in Figure 3.2. The slip band was formed to accommodate macroscopic tensile loading (for all images in this paper the tensile direction is horizontal with respect to the image) as is revealed on the EBSD image quality map (Figure 3.2a). No slip transfer or slip band formation was observed in the neighboring grain. The orientation relationship

between the two grains is close to a 70° rotation about x_1 axis as is revealed by the superimposed unit cell structures. The slip plane of the slip band was found by trace analysis and the crystal orientation from EBSD and is indicated in red color. This slip system corresponds to a low CRSS $\langle a \rangle$ prismatic slip system that has a high (0.50) Schmid factor. HR EBSD output of the full elastic stress tensor is illustrated in Figure 3.2b. Stress concentration induced by the blocked slip band is revealed most clearly in the σ_{11} and σ_{12} components. The stress tensor shown in Figure 3.2b is measured in sample frame as defined by x_1 and x_2 (x_3 component is perpendicular to the image surface and pointing into the page). Figure 3.2c shows a more useful axis system, x_1^r x_2^r x_3^r , which is described by the Burger's vector (x_1^r) and line direction (x_2^r) of the active slip system on the top grain. The resolved shear stress on the projection of the active slip plane is the σ_{13}^r component of the tensor, and is shown in Figure 3.2d.

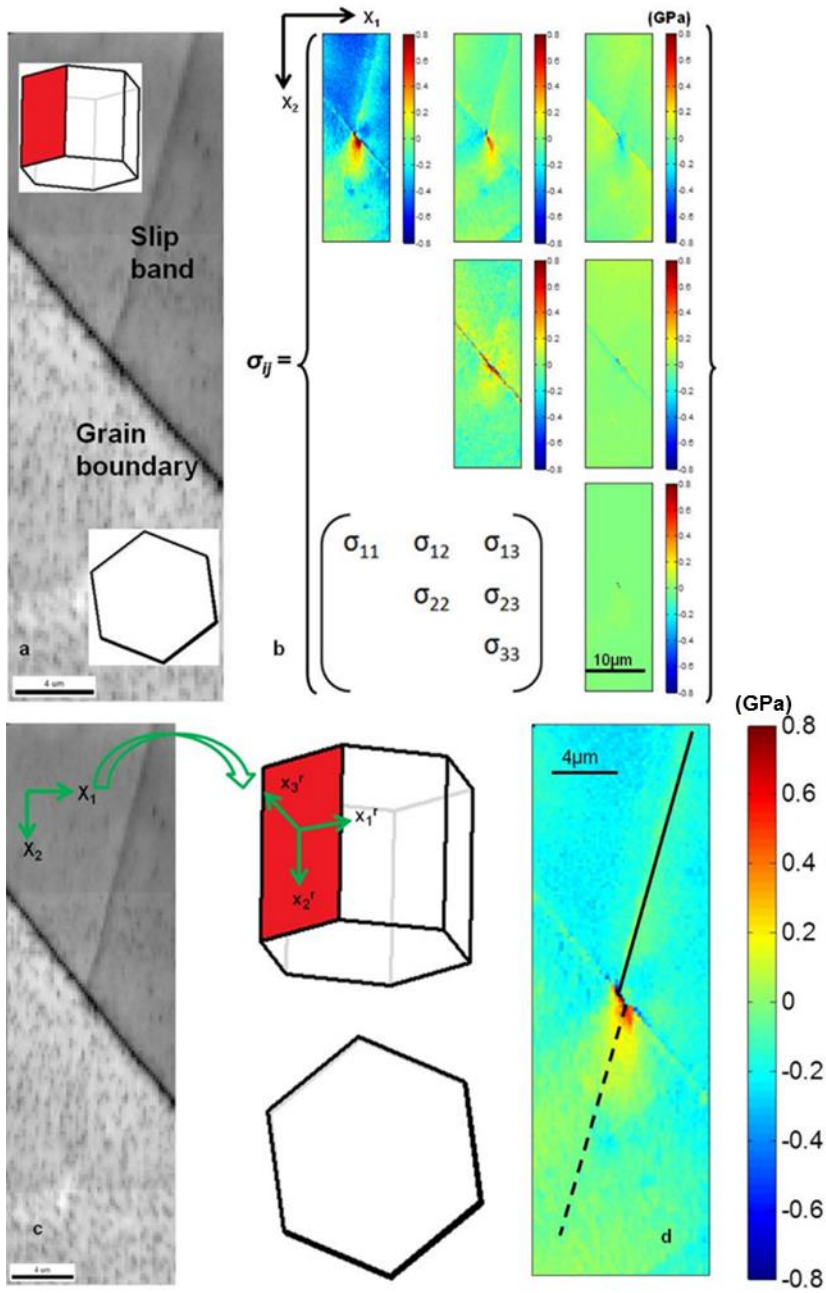


Figure 3.2: (a) EBSD image quality map of a blocked slip band with grain orientation overlapped; (b) HR EBSD stress output; (c) Coordinate system defined on sample reference frame (x_1 x_2) and slip plane reference frame (x_1^r x_2^r x_3^r); (d) The σ_{13}^r component of the stress tensor after rotation, dashed line represent direction along which stress profile is extracted (all colour scales in GPa).

A line scan along the direction of the slip band on the upper grain but extended into the lower grain (indicated by the dashed line) was extracted and the resulting stress profile is shown in Figure 3.3. These data points fit well with the model proposed by Eshelby et al [110].

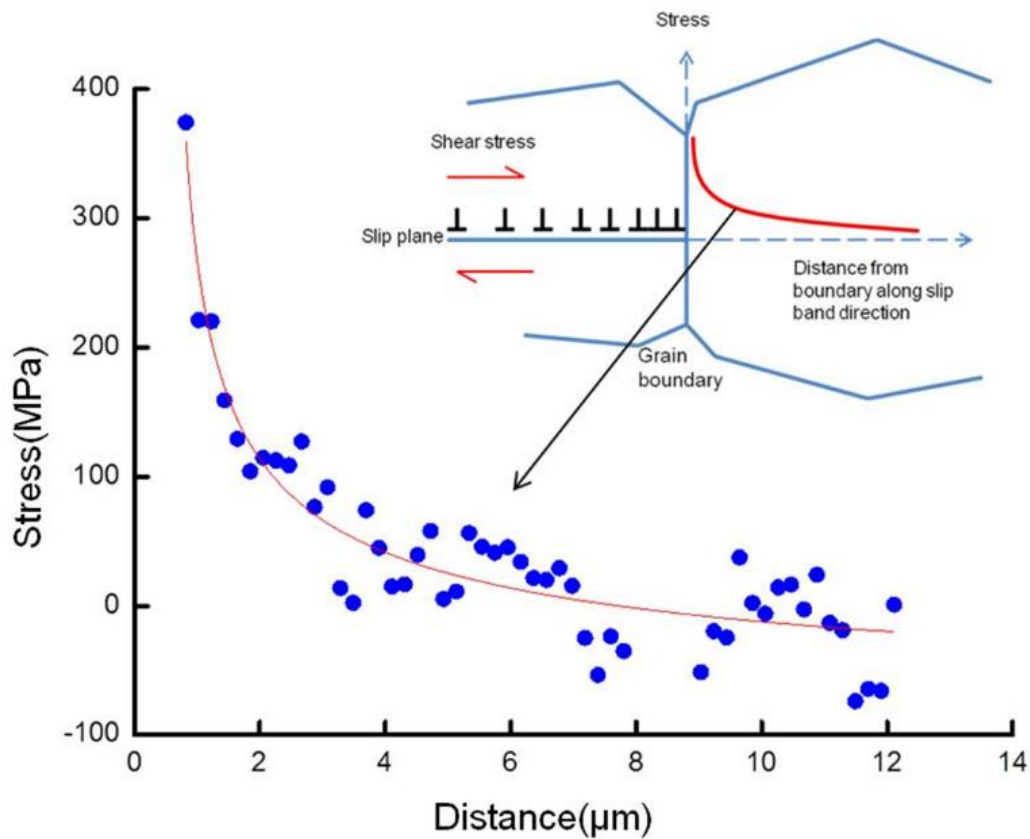


Figure 3.3: Stress concentration ahead of the blocked slip band as in Figure 3.2d with comparison to the model described by Eshelby et al [110].

Fitting the experimental stress profile with equation 3.1 allows a K value of 0.78 (± 0.08) MPa m^{1/2} to be obtained for this boundary, where the error indicates standard error from best fit parameters. The same procedure was performed for four other boundaries in this category.

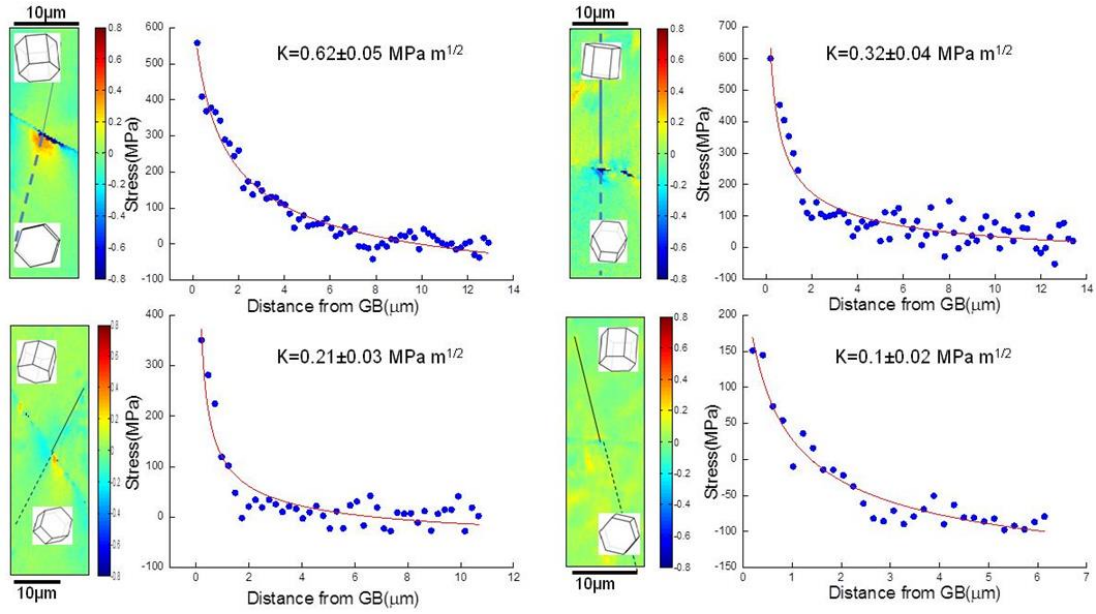


Figure 3.4: Stress profile in front of blocked slip bands and fitted stress profiles. Solid line represents the position of incoming slip band and dashed line is the direction (along slip band direction) along which stress profile was extracted

The results are summarized in Figure 3.4 which shows the resolved shear stress maps overlaid with corresponding grain orientations indicating different grain boundary geometries, along with the stress variation on a line directly ahead of the slip line. The K value, as obtained from fitting each stress profile with equation 3.1, is indicated for each grain boundary. The LRB criteria was employed in such a way that the combination of M factor (equation 2.16), between the incoming slip system and various of possible slip systems in the neighbouring grain, and the resolved shear stress (RSS), described by Schmid factor (S), were used to characterize each case of interaction. For all five slip band—grain boundary interactions in Figures 3.2 & 3.4, trace analysis revealed that each of the slip bands were due to $\langle a \rangle$ prismatic slip. The best alignment (i.e. highest M) between these prismatic slip systems with possible $\langle a \rangle$ basal,

$\langle a \rangle$ prismatic, $\langle a \rangle$ pyramidal, and $\langle c+a \rangle$ pyramidal slip systems and related Schmidt factors (S) are compiled in Table 3.2.

	Boundary 1	Boundary 2	Boundary 3	Boundary 4	Boundary 5
	K=0.78	K=0.62	K=0.32	K=0.21	K=0.1
Prismatic $\rightarrow$ Prismatic	M ₁ =0.33	M ₁ =0.40	M ₁ =0.47	M ₁ =0.53	M ₁ =0.54
	S ₁ =0.23	S ₁ =0.49	S ₁ =0.40	S ₁ =0.30	S ₁ =0.28
	M ₂ =0.11	M ₂ =0.40	M ₂ =0.06	M ₂ =0.01	M ₂ =0.01
	S ₂ =0.47	S ₂ =0.49	S ₂ =0.46	S ₂ =0.49	S ₂ =0.50
Prismatic $\rightarrow$ Basal	M=0.35	M=0.48	M=0.23	M=0.28	M=0.33
	S=0.23	S=0.01	S=0.03	S=0.14	S=0.01
Prismatic $\rightarrow$ $\langle a \rangle$ Pyramidal	M=0.48	M=0.6	M=0.49	M=0.56	M=0.61
	S=0.02	S=0.33	S=0.25	S=0.17	S=0.20
Prismatic $\rightarrow$ $\langle c+a \rangle$ Pyramidal	M=0.60	M=0.54	M=0.83	M=0.82	M=0.78
	S=0.33	S=0.40	S=0.27	S=0.27	S=0.11

Table 3.2: Geometric alignment (M) between incoming slip plane and possible slip planes in neighbouring grain and Schmidt factor on corresponding slip system (S). Only the best alignments (i.e. highest M) are listed except for M₂ values in second row of prismatic $\rightarrow$ prismatic alignment.

For this category of slip band-grain boundary interactions, the incoming prismatic slip systems are better aligned (i.e. higher M) with $\langle c+a \rangle$ pyramidal slip system in adjacent grains, however the magnitude of the $\langle c+a \rangle$ Burger's vector is too large compared to $\langle a \rangle$ slip and transfer would require accommodation of additional $\langle c \rangle$. This coupled with the higher CRSS and generally low Schmid factor means slip is not transferred to $\langle c+a \rangle$ slip system.

On the other hand, easy slip systems (prismatic) have poor alignment (i.e. low M) with the incoming slip band and low Schmid factors. Those easy slip systems that do have high Schmid factor, almost at the maximum value of 0.5, were however found to have the worst alignments with incoming prismatic slip systems.

The stress ahead of the blocked pile-up is likely to depend on the number of dislocations within the pile-up. The analysis of Eshelby et al. [22] indicates that the stress intensity increases as $\sqrt{N}$ when the number of dislocations in the pile-up (N) is large. AFM maps, combined with the orientation of the active slip system, were used to measure the slip band heights h , from which N was obtained from knowledge of the Burger's vector length and slip direction established from EBSD. Figure 3.5 shows a plot of the measured stress intensity ahead of the blocked slip band as a function of $\sqrt{N}$ (equation 3.9). Boundary 3 (Table 3.2) is not shown in Figure 3.5 as the position of which on sample surface was lost. Instead, the result obtained by Britton et al [111] was incorporated and is indicated by the triangular mark in Figure 3.5.

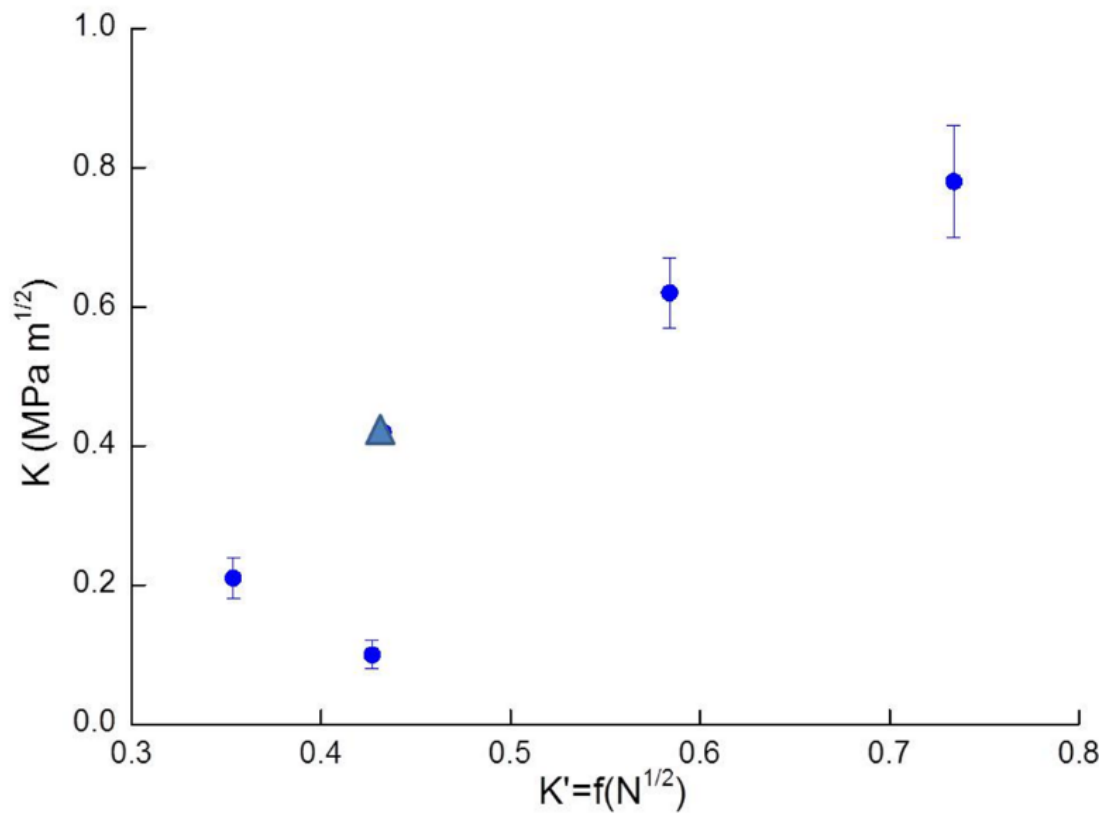


Figure 3.5: K (stress intensity factor, $\text{MPa m}^{1/2}$) as a function of the square root of the number of dislocations, N , in the slip band.

It can be seen from Figure 3.5 that the number of dislocations in the pile up correlates well with the measured stress intensity. In the case of no slip transfer and under the local slip system alignment condition specified in Table 3.2, more dislocation density in the pile up plane result in higher magnitude of stress concentration in the neighbouring grain. The only available literature so far [111] reporting the same measurement (triangle in Figure 3.5) fits in well with the current observation.

3.3.2 Slip transfer

For this type of interaction, the slip band is transferred across the grain boundary (it is not clear whether it is direct transfer or not under the resolution of current experimental condition). As ex-situ experiment does not tell which slip band is the incoming band and which is the outgoing band it is necessary to resolve the stress tensor onto the slip plane for each individual slip bands. For one of the slip band/grain boundary interactions of this category, the σ_{13}^r variation is shown in Figure 3.6.

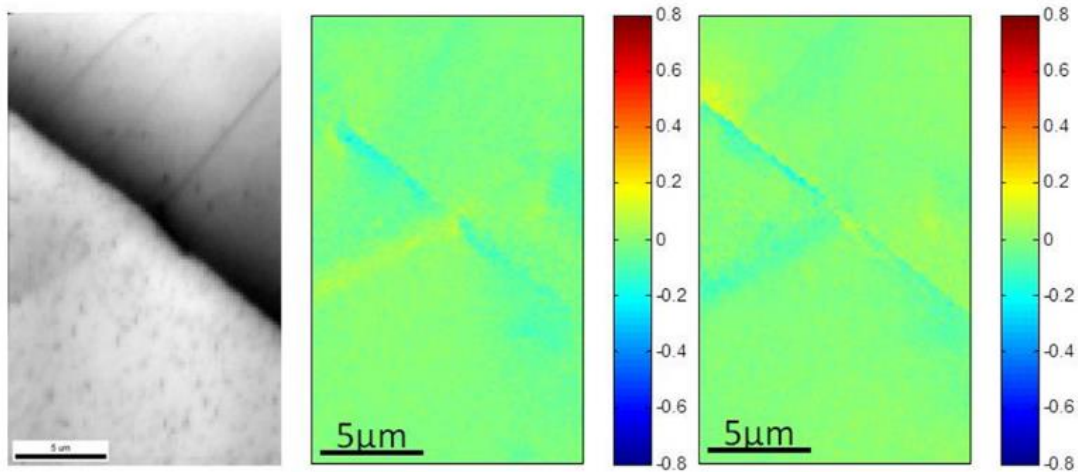


Figure 3.6: Scanning electron microscopy image showing a slip transfer feature (left), and σ_{13} shear stress resolved onto the active slip system of the top grain (middle) and bottom grain (right).

It can be seen from the stress map that there are low level of diffuse stress variations near each slip band but no strong localized stress concentration like those in Figure 3.2 was induced by either of the slip bands. No 'one over square root distance' stress distribution can be observed ahead of either slip band. Seven instances where slip bands continued on either side of the grain

boundary were mapped using HR-EBSD and in all cases there were no significant stress concentrations at the grain boundary. For geometric alignment investigation, slip band on prismatic plane was assumed to be the incoming slip band and the M factor and Schmidt factor S were calculated for the possible outgoing slip band, these are compared in Table 3.3 for the possible slip systems in the 'transferred' grain.

	Case 1	Case 2	Case 3	Case 4	Case 5	Case 6	Case 7
Prismatic→	M ₁ =0.94	M ₁ =0.46	M ₁ =0.95	M ₁ =0.41	M ₁ =0.84	M ₁ =0.73	M ₁ =0.91
Prismatic	S ₁ =0.26	S ₁ =0.33	S ₁ =0.33	S ₁ =0.39	S ₁ =0.47	S ₁ =0.43	S ₁ =0.47
Prismatic→	M=0.04	M=0.71	M=0.19	M=0.64	M=0.45	M=0.68	M=0.15
Basal	S=0.03	S=0.41	S=0.03	S=0.20	S=0.18	S=0.11	S=0.05
Prismatic→	M=0.67	M=0.80	M=0.78	M=0.72	M=0.9	M=0.43	M=0.50
<α>Pyramidal	S=0.18	S=0.31	S=0.25	S=0.42	S=0.18	S=0.01	S=0.35
Prismatic→	M=0.56	M=0.32	M=0.52	M=0.44	M=0.66	M=0.40	M=0.76
<c+a>Pyramidal	S=0.10	S=0.13	S=0.11	S=0.11	S=0.38	S=0.17	S=0.31

Table 3.3: Best alignment (maximum M) between prismatic slip system in one grain and various slip systems in the other grain. Coloured cells indicate the observed slip plane of the 'transferred' slip system.

By comparing the alignments in each column for the seven slip transfer cases, it can be seen that the observed 'transferred' slip systems are the ones that have the best geometric alignment (i.e. highest M) with the 'incoming' slip system. Transferred slip bands were formed on either prismatic slip plane or <a>pyramidal slip plane. The transferred slip systems also tend to have a

relatively high Schmid factor based on the far field applied loading. This can be noticed by comparing the S values for each column of Table 3.3.

3.3.3 Blocked slip band that does not lead to stress concentration

The third category of slip band/grain boundary interaction appear similar to the first category (Section 3.3.3) in that SEM imaging shows a slip band terminating at a grain boundary. The difference is that HR-EBSD reveals that these blocked slip bands do not lead to stress concentration. Two examples of the six cases mapped are shown in Figure 3.7.

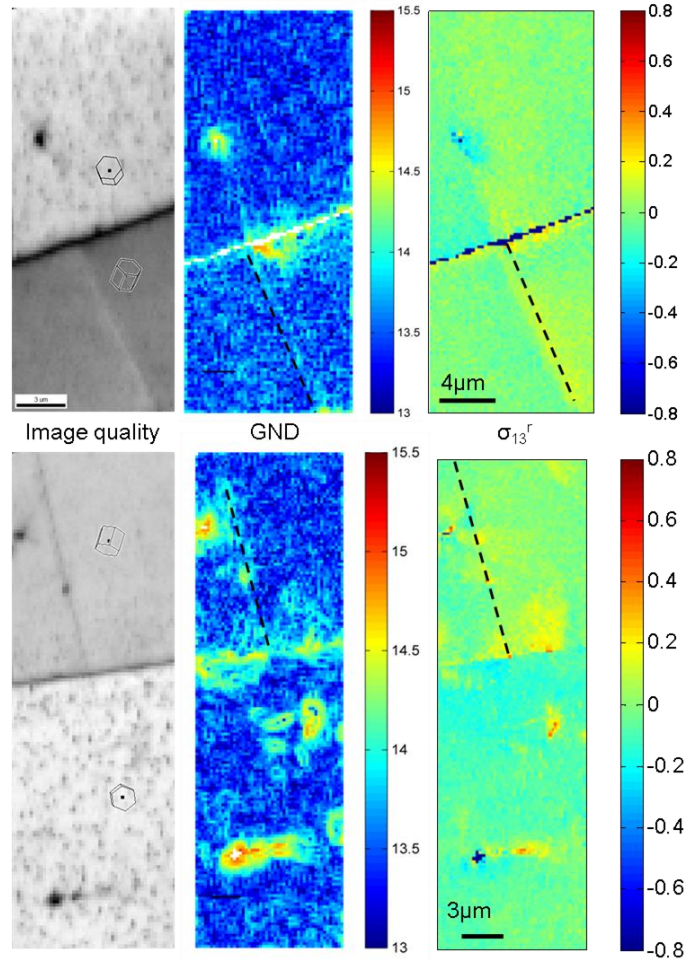


Figure 3.7: Image quality map visualizing a blocked slip band, corresponding geometrically necessary dislocation (GND) density map (colour bar is on Log_{10} scale), and σ_{13}^r shear stress (GPa). Black dash line indicates slip trace.

The distinguishing feature for this category of interaction is that no localized stress concentration can be seen around the intersection of the slip band and the grain boundary as is revealed by the σ_{13}^r resolved shear stress fields in Figure 3.7. The lower bound estimation of the GND density distributions for these cases are also analysed. In one case (Figure 3.7 top) there is localized GND concentration near the head of the blocked slip band which suggests an interaction in which some dislocation content is reflected back from the grain

boundary. While in another case (Figure 3.7 bottom) there is no localized GND concentration but some evidence of increased GND density distributed along grain boundary. The geometric alignments for the six instances of this type of interaction that were analysed are listed in Table 3.4.

	Case 1	Case 2	Case 3	Case 4	Case 5	Case 6
	$M_1=0.72$	$M_1=0.39$	$M_1=0.67$	$M_1=0.87$	$M_1=0.70$	$M_1=0.48$
Prismatic→	$S_1=0.35$	$S_1=0.34$	$S_1=0.44$	$S_1=0.27$	$S_1=0.48$	$S_1=0.38$
Prismatic	$M_2=0.05$	$M_2=0.05$	$M_2=0.00$	$M_2=0.11$	$M_1=0.70$	$M_1=0.04$
	$S_2=0.48$	$S_2=0.48$	$S_2=0.42$	$S_2=0.49$	$S_1=0.48$	$S_1=0.41$
Prismatic→	$M=0.58$	$M=0.70$	$M=0.39$	$M=0.39$	$M=0.49$	$M=0.53$
Basal	$S=0.04$	$S=0.09$	$S=0.07$	$S=0.04$	$S=0.13$	$S=0.28$
Prismatic→	$M=0.92$	$M=0.78$	$M=0.74$	$M=0.88$	$M=0.84$	$M=0.65$
<a>Pyramidal	$S=0.26$	$S=0.30$	$S=0.35$	$S=0.21$	$S=0.42$	$S=0.22$
Prismatic→	$M=0.50$	$M=0.41$	$M=0.64$	$M=0.60$	$M=0.68$	$M=0.58$
<c+a>Pyramidal	$S=0.11$	$S=0.01$	$S=0.20$	$S=0.03$	$S=0.14$	$S=0.01$

Table 3.4: Best alignments (except for M_2 in prismatic→prismatic alignment) between the blocked slip plane and various slip systems in the neighbouring grain. Boundary 1 and Boundary 2 correspond to Figure 3.7 top and bottom respectively.

From the comparison between the alignments with various slip systems, the blocked pile-up planes are better aligned with <a> pyramidal plane that have relatively lower Schmid factor. Prismatic slip systems, on the other hand, have generally both lower M value and Schmid factor, compared to the <a> pyramidal system. However the M and S values of prismatic slip systems are not so distinct from those grains in Table 3.3 for cases where slip transfer did occur. Similar to blocked slip bands in the first category of interactions (Table 2),

prismatic slip system with higher Schmid factor have the worst alignment (M_2) with incoming slip bands.

3.3.4 Residual Burger's vector, stress intensity factor, and M

The data was investigated for correlation between category of interaction and residual Burger's vector between the incoming (prismatic) slip system and various slip systems in the neighbouring grain, stress intensity factor K , and geometric alignment factor M . For each of the three types of slip band - grain boundary interactions Figure 3.8 plots the alignment factor M assuming transfer to either prismatic or $\langle a \rangle$ pyramidal slip systems against the corresponding Schmidt factor S . The filled symbols on Figure 3.8 represent the best aligned $\langle a \rangle$ prism slip system, while the hollow symbols are for the best aligned $\langle a \rangle$ pyramidal slip systems. The background red colouring of the plot indicates the general tendency for the cases where slip transfer was observed to combine both good alignment (i.e. high $M > 0.7$) and large shear stress (i.e. high $S > 0.3$) on the selected slip system. While blocked slip bands, leading to a measureable stress intensity, (blue background shading) tended to be observed when the slip system alignment was relatively poor (M factor below ~ 0.5) but over the whole range of possible Schmidt factors. Blocked slip bands with very low induced stress intensity factors tend to be in a regime with intermediate M but most often with significant Schmidt factor. There is significant overlap between the three regions in this M - S space possibly due to the varying amount of slip on the different slip bands observed. It seems that although both M and

S influence the situation the alignment factor M takes priority in controlling slip transfer.

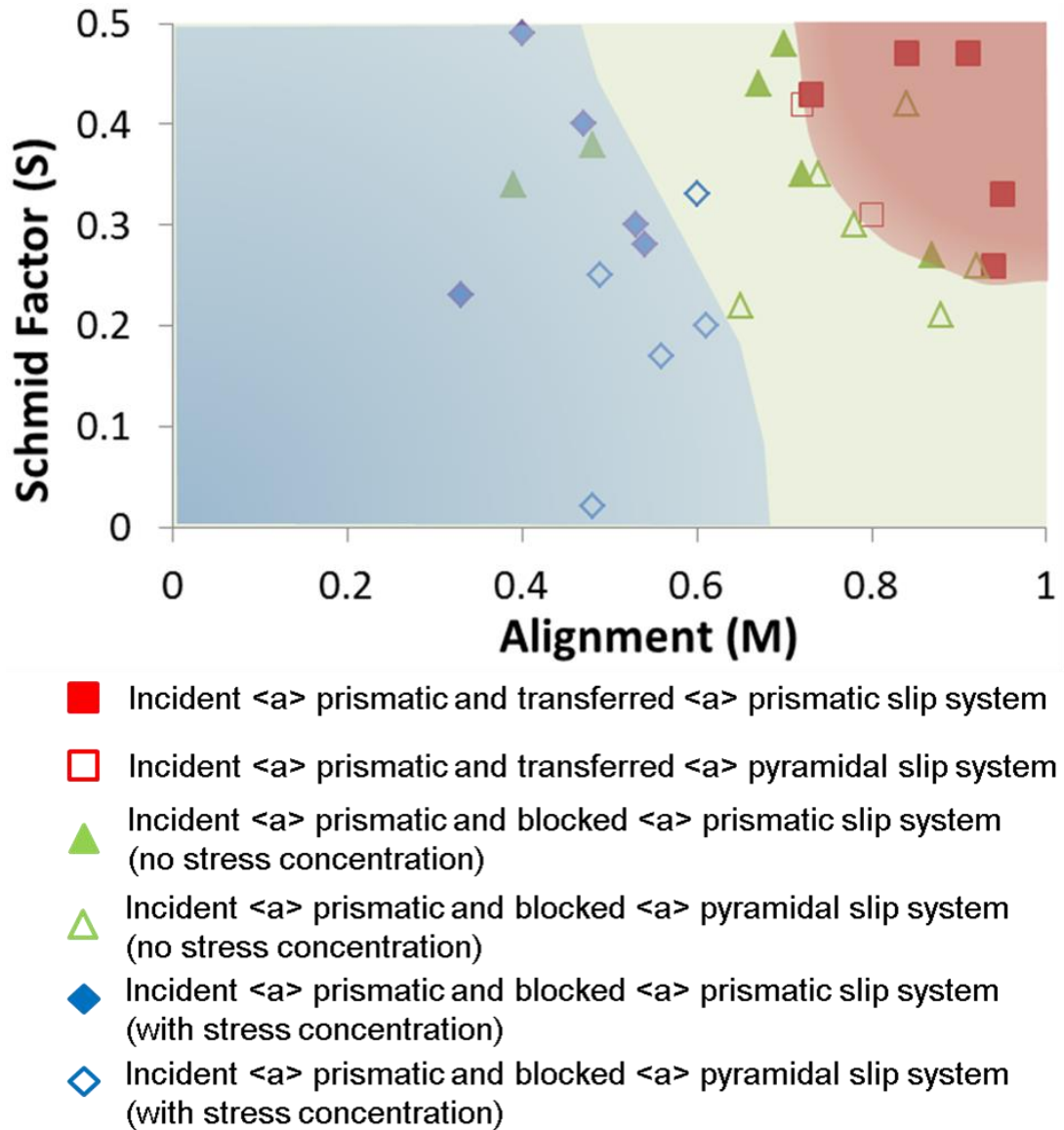


Figure 3.8: The plot of Schmid factor (S) against M factor. The left of the plot is coloured blue which represents the zone for the first type of interaction, the middle of the plot is coloured green to represent the zone for the third type of interaction, and the top right coloured red to represent the zone for the second type of interaction.

For the blocked slip bands that induced a stress intensity there was no obvious correlation between the M and K , this is revealed in Figure 3.9. However, K did tend to be smaller when the residual Burger's vector was larger assuming transfer to the best aligned prismatic slip systems. The most meaningful correlation was however between the magnitude of K and the extent of slip in the slip band (Figure 3.5).

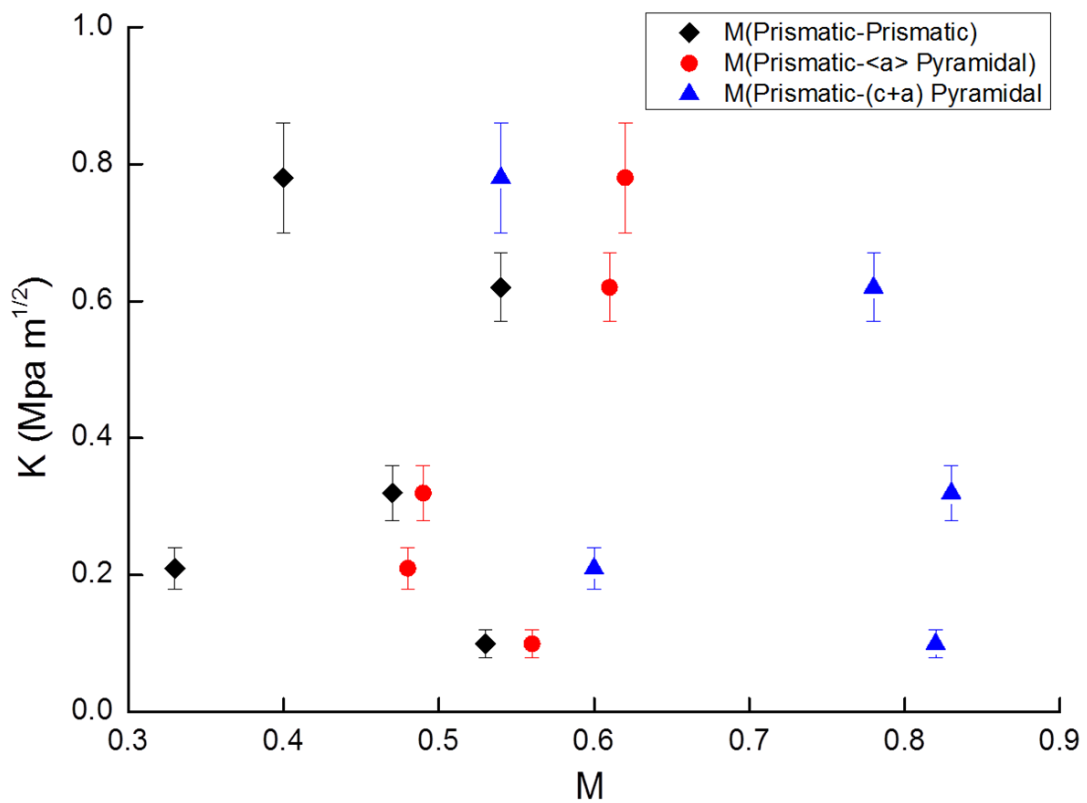


Figure 3.9: A plot of the alignment (M) between incoming prismatic slip system against prismatic, $\langle a \rangle$ pyramidal, and $\langle c+a \rangle$ pyramidal slip systems in the neighbouring grain. The data points are taken from the first category of interactions (Section 3.3.1).

3.4 Discussion

The slip band/grain boundary interactions in commercial purity titanium were studied with high resolution EBSD, slip trace analysis and AFM. The sample was pulled to 1% strain at low strain rate and no twinning activity was observed, therefore the observations at this stage are useful in analysing the condition for twin nucleation and the criteria for early stage slip transfer. Several blocked and transferred slip bands at randomly selected grain boundaries were studied and it was found that three categories of interactions can be observed.

The first category of interaction involves blocked slip bands leading to stress concentration in the neighbouring grain. Trace analysis shows that in each case the incoming slip band was due to dislocations on prismatic slip plane (marked red in Figure 3.2), which agrees well with the macroscopic resolved shear stress analysis (i.e. Schmid factor). From the grain orientations (Figures 3.2 and 3.4) it seems that all of the boundaries studied in this category have large rotations between the two grains causing the basal plane normal to be close to perpendicular to each other. This generates a large misalignment of the easy slip systems. Notably in the earlier work of Britton and Wilkinson [23] the significant misalignment of slip systems was achieved with the basal planes of the two grains close to parallel but with a rotation of $\sim 30^\circ$ about the almost shared c-axis.

For this case, the stress concentration resolved onto the pile-up plane of the incoming slip band agrees well with Eshelby's model (Figure 3.3). The stress intensity factor obtained by fitting the stress profile using equation 4 varies from

boundary to boundary in the range from 0.1 MPa $m^{1/2}$ to 0.78 MPa $m^{1/2}$ with an average value of 0.41 MPa $m^{1/2}$. The average value is in agreement with the Hall-Petch coefficient of 0.40 MPa $m^{1/2}$ obtained by Lederich et al. [105] and 0.40 MPa $m^{1/2}$ from Armstrong et al. [184] from macroscopic tensile testing of polycrystalline Ti of varying grain size. Lederich et al. [105] also examined tensile deformation of CP titanium at elevated temperature of 573K, where cross slip may facilitate slip transfer and obtained a lower Hall-Petch coefficient of 0.18 MPa $m^{1/2}$. Salem et al. [106] obtained a higher Hall-Petch coefficient of $K=0.67$ MPa $m^{1/2}$ from room temperature compression testing of titanium, and ascribed the higher value to the increased population of twin boundaries which generated large repulsive stress on incoming dislocations and therefore provides a harder barrier for slip transfer [185]. Our stress intensity values are in broad agreement with Hall-Petch coefficient obtained from tensile testing but of course can be assessed in terms of the slip resistance offered by individual boundaries of varying geometry where stress intensities were measured.

When large numbers of dislocations have passed along the slip band to pile-up at the grain boundary the analysis of Eshelby et al. [110] gives the stress intensity to be

$$\sigma'_{13}\sqrt{X} = \sqrt{\frac{N\mu b\tau}{\pi(1-\nu)}} \quad (3.9)$$

Where μ is the Shear modulus, ν Poisson's ratio and τ the shear stress resolved onto the slip system of the slip band. Figure 3.5 showed that there was a reasonable correlation between the stress intensity factors measured by HR-EBSD and $\sqrt{N}$ estimated from AFM measurements of the slip band height. The

blocked slip bands were all on slip system for which the Schmidt factor was close to 0.5 so τ is approximately half the applied tensile stress ($\tau = 115$ MPa). In every case the stress intensity measured by HR-EBSD was of the same order of magnitude but somewhat less than the value predicted from the theory of Eshelby et al. [110] using the estimated number of dislocations in the slip band. This implies that there has been some relaxation of the expected stress intensity factor which could be achieved by a combination of the following mechanisms: (i) some limited slip transfer with plastic flow in a small volume of material in the neighbouring grain as seen by Britton & Wilkinson [23] and Villechaise et al. [186], (ii) dissipation of some of the net Burger's vector by generation of grain boundary dislocations and their motion away from the head of the pile-up, or (iii) some cross slip across the slip band to generate a pile-up of finite width [187].

Figure 3.6 is an example of the slip band/grain boundary interactions which lead to slip transfer. It is not clear which slip band is the incoming one so the stress tensor is rotated onto each pile-up plane individually. As is shown in Figure 3.6, there is no localized stress concentration induced by shear stress on the active slip plane, which indicate that slip transfer is a highly effective stress relieving mechanism. For uniformity, prismatic slip band was selected as the incoming slip band and the other was treated as the transferred band and therefore the alignment factor M and Schmid factor between the 'incoming' slip band and various slip systems in the neighbouring grain can be calculated. Coloured cells in Table 3.3 indicate the pile-up plane of the transferred slip band. It can be seen that the transferred slip bands are on prismatic or $\langle a \rangle$

pyramidal slip system where the alignment is the best compared with other slip systems. For boundary 1 and 3, transferred slip systems are prismatic with almost perfect alignments with incoming slip plane while the RSS on those slip planes are small ($S_1=0.26, 0.33$, respectively). Contrary to this, the prismatic slip system that with highest Schmid factor ($S_2=0.5$) have the worst alignment with incoming slip plane ($M_2=0.08$ and 0.11 respectively). Figure 3.8 shows that the M factor effectively separates different types of interactions into their own regions. Generally speaking, slip transfer requires both high M and S (on prismatic and $\langle a \rangle$ pyramidal system), but M is the dominant factor to consider in predicting slip transfer. Although RSS plays less important role, it is important in determining how far the transferred slip band is able to propagate, a transferred slip band is likely to terminate at a short distance from grain boundary when RSS is not enough to sustain continuous dislocation motion. Recent work using digital image correlation and EBSD by Abuzaid et al. [188] showed that larger plastic strains accumulated in ‘mantle’ regions near grain boundaries for which slip transfer led to small residual Burger’s vectors compared to those in which slip transfer was inhibited by misalignment of the predominant slip systems to give a large residual Burger’s vector.

The third type of slip band/grain boundary interaction observed in this research was blocked slip bands that leads to neither stress concentration nor slip transfer. EBSD image quality map, geometrically necessary dislocation (GND) density, and σ_{13}^r shear stress map for two of such interactions are shown in Figure 3.7. From Figure 3.7 top, it can be seen that a small kink is formed at the intersection point of the slip band and the grain boundary. This kink is linked to

a localized region of GNDs reflected back from the grain boundary into the grain containing the slip band. Neither stress concentration nor GND concentration were induced in the neighbouring grain. This is likely to be a case when stress concentration is accommodated by localised slip close to the head of the blocked slip band. By contrast Figure 3.7 bottom is a case where there is no localized GND concentration but more diffuse GND distribution along the grain boundary, with again no stress concentration present around the interaction point but there seem to be a backward stress to the right of the grain boundary. In such cases it is suspected to involve absorption of lattice dislocations onto the grain boundary and the motion of grain boundary dislocations away from the head of the pile-up generating local grain boundary sliding. Figure 3.8 suggests that these interactions occur for intermediate values of slip system alignment. There is however considerable overlap between M factors observed for cases where a clear slip transfer occurs with the two significant slip bands intersecting at the grain boundary and M factors found for apparently blocked slip bands but where local deformation processes have successfully relaxed the local stress concentration.

It can be seen from Figure 3.9 that for the category of blocked slip band with stress concentration, the correlation is poor between incident prismatic slip system and various slip systems in the neighbouring grain. This may indicate that the geometric alignment factor M is not a sufficient indicator of the level of stress concentration compared to the number of dislocations within the pile up plane (Figure 3.5). However, this declaration is not conclusive as the slip plane normal was used to calculate the M factor (equation 2.16) rather than the line of

intersections between slip plane and grain boundary as was originally proposed in the LRB criteria [123]. A full three dimensional analysis of the boundary geometry, along with stress and dislocation density distributions below the sample surface might help resolve some of these issues.

3.5 Conclusion

The High resolution EBSD technique is used to study slip band and grain boundary interactions in CP titanium deformed in tension to 1% strain. The following conclusions may be drawn:

1. At this low strain level slip band/ grain boundary interactions can be ascribed to three types: blocked slip band that induces stress concentration in the neighbouring grain, blocked slip band that does not induce stress concentration in the neighbouring grain, and slip transfer.
2. Full slip transfer requires both good alignment (high $M > 0.7$) with incoming easy slip system and high resolved shear stress on the outgoing slip system. For selecting transferred slip system, local geometric alignment seems to play a more important role than the resolved shear stress.
3. In each case of slip transfer examined HR-EBSD showed that there was no significant stress intensity associated with the intersection of the two slip bands at the grain boundary.
4. When the easy slip systems in the neighbouring grain were badly aligned with the incoming slip system to give an M factor below ~ 0.7 blocked slip bands were observed. In some such cases HR-EBSD revealed significant local stress intensity at the head of the blocked slip band but in many cases the stresses were not significant. The high stress intensity cases tend to have worse slip plane alignment.

5. Where significant local stresses were found they were broadly consistent with the 'one over square root of distance' variation expected from Eshelby et al's model [25]. The stress intensity was larger for slip bands for which AFM revealed larger plastic strains.
6. For the blocked slip bands that had no obvious stress concentrations, lattice curvature analysis indicated that GND densities were increased either in a localised feature associated with the head of the pile-up, or in a more diffuse distribution along the grain boundary. These suggest that localised plastic flow was able to relieve any stresses developed by the blocked slip band.

Chapter 4 3D Measurements of Stress Fields near Grain Boundaries

For the research reported in Chapter 3, it is curious that if the blocked slip band leads to the localized stress concentration in the neighboring grain, what will the stress field look like beneath the sample surface and along the dislocation pile up plane? How does other strain measurement technique compares to our novel HR-EBSD? And is the grain boundary strength (K) quantification procedure persistent to stress measured using different approach? These can be answered by the serial sectioning + HR-EBSD measurement, however, this procedure suffers from the difficulty of quantifying damage induced by removing successive surface layers, the layer to layer alignment induces error, and further experiment on the material will not be possible as it will eventually disappear. Therefore, a non-destructive high energy X-ray approach was chosen—differential aperture X-ray Laue micro-diffraction (DAXM).

4.1 Experiments

The blocked slip band that leads to a stress concentration as shown in Figure 3.2 and Table 3.2 (boundary 1) was further investigated using differential aperture micro-beam Laue X-ray diffraction microscopy (DAXM), performed at beam line 34-ID-E at the Advanced Photon Source (APS), USA. Detailed overview of the technique, analysis method and some applications can be found in references [189, 190]. A brief outline of the specific procedure and analysis routine is provided here.

Polychromatic X-ray beam was focused to sub-micron size using two non-dispersive Kirkpatrick-Baez focusing mirrors. These mirrors produce a beam with a 'Lorentzian' profile with a full width half maximum of $\sim 0.5 \mu\text{m}$. Diffraction patterns were recorded on a Perkin-Elmer flat panel detector ($409.6 \times 409.6 \text{ mm}^2$, $2,048 \times 2,048$ pixels, amorphous Si, CsI scintillator, 16-bit dynamic range corresponding to 65,536 counts) mounted in 90° reflection geometry 510.3mm above the sample. The experimental geometry was calibrated using measurements of a thin stress free silicon sample ($<5 \mu\text{m}$ thickness). Knowledge of the energies of the reflections was subsequently used to calibrate the position and orientation of a large area X-ray detector with respect to the incident X-ray beam and the sample. Overlapping Laue patterns were generated from each voxel position along the direction of the beam/sample interaction. The depths of origin of different scattered contributions were determined by wire scanning, allowing the reconstruction of depth-resolved Laue patterns [189, 191]. Key to this process is the accurate positioning, alignment and scanning of the wire with respect to the sample surface. In this

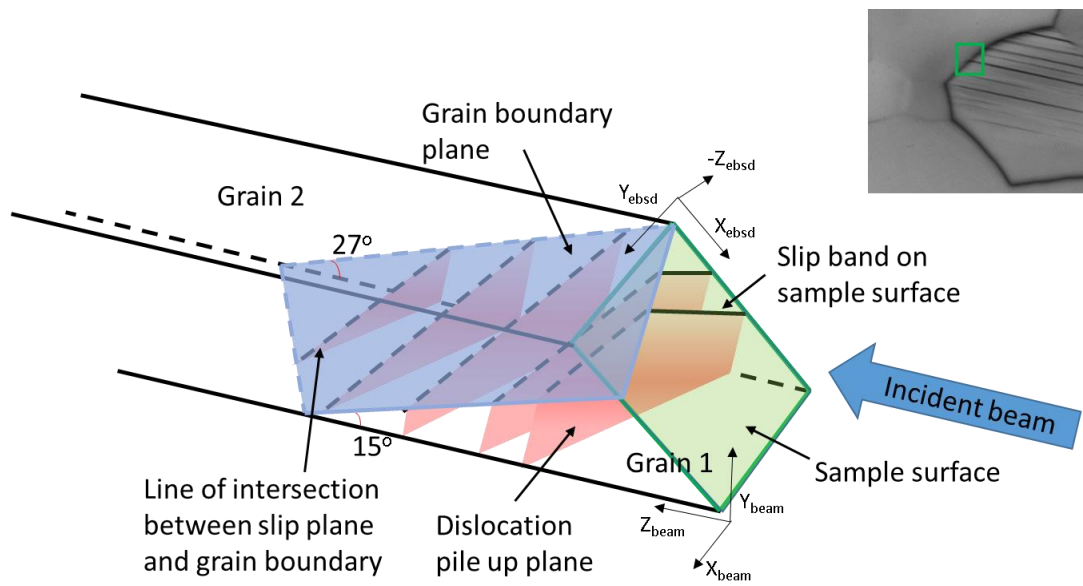
experiment, this was performed using careful calibration of the detector alignment to ensure that depth profiling produces reproducible results within the Si membrane reference sample.

The titanium sample was positioned in the X-ray beam through use of a low depth of field optical microscope, confining the inclined (45° with respect to horizontal plane) surface plane of the specimen to a specific region where the X-ray beam strikes. Regions of interest were marked using focused ion beam trenches and Pt depositions with an aim of using X-ray fluorescence (XRF) to determine the location. Unfortunately the volume of Pt present did not produce a significant signature in XRF and therefore only the optical images, and rapid assessment of diffraction patterns were used to choose the specific regions.

The sample surface was scanned at 1 μm intervals. At each interrogation point the wire was swept across the sample. Ray-tracing reconstruction of individual voxel Laue patterns using the wire-scans were performed using LaueGo software (developed by Jon Tischler: tischler@aps.anl.gov). Reconstructions were performed to a depth of 190 μm into the sample with a 1 μm depth spacing. This was repeated to generate a series of slices that form a volume map of the size 23 μm x 31 μm x 190 μm , as is shown in Figure 4.1, with 1 μm^3 voxel size. For each voxel, pattern indexing, strain refinement, and lattice rotation calculation were done using LaueGo software at 34-ID-E. As the volume map contained only two grains, example Laue patterns of the two crystal orientations were checked by hand. The lengths of the $\langle c \rangle$ and $\langle a \rangle$ axis for Ti were calibrated using a monochromatic beam and a c/a ratio of 1.588 was measured. Orientations and reference frames were directly compared between

EBSD measurements and the Laue diffraction analysis and were found to be consistent. In addition to extracting the strain and rotation tensors, a reliability factor based upon the number of spots used in the indexing is reported. This was used to filter out erroneous ‘ghost points’ from above the sample surface, observed due to the nature of the back-subtraction algorithm from the DAXM analysis procedure. Data were exported into Matlab for further computation and 3D volumes were rendered in Avizo.

Figure 4.1: Visualization of the probed volume. Grain boundary is approximated to be planar and colored light blue in this image. Dislocation pile up plane is colored red and



the dashed lines on the blue grain boundary plane represent the line of intersection between slip plane and grain boundary. The solid lines on sample surface plane (colored green) represent the slip band which is visible under optical microscopy and SEM. Coordinate systems X_{ebsd} , Y_{ebsd} , Z_{ebsd} and X_{beam} , Y_{beam} , Z_{beam} indicate the special arrangement of EBSD reference system and X-ray beam reference system with respect to the measured sample volume. The inserted image to the upper right corner is an

optical image of the sample surface, showing this specific case of slip band/grain boundary interaction and the approximate position of the experiments (green box).

4.2 DAXM data set analysis and 3D volume rendering

The indexed data set contains information about experimental geometry (3 sets of reference frames) strain free single crystal lattice constant referenced in reciprocal space, diffraction plane normal, and various other experimental set up related parameters. Different from Figure 3.1, the DAXM experiment have the following Cartesian reference frame relative to crystal reference frame:

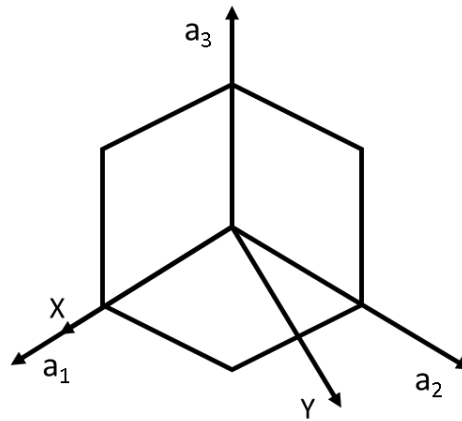


Figure 4.2: Cartesian reference frame definition relative to crystal reference frame

Under this configuration, the transformation matrix which converts vectors in crystal reference frame ($a_1 a_2 a_3$) to Cartesian reference frame ($X Y Z$) becomes:

$$T_2 = a \begin{pmatrix} 1 & -\cos 60^\circ & 0 \\ 0 & \cos 30^\circ & 0 \\ 0 & 0 & c/a \end{pmatrix} \quad (4.1)$$

Deformation free single crystal lattice constants can be found in literature [192], which lead to the following matrix that in effect define the crystal reference frame:

$$H = \begin{pmatrix} 0.29508 & 0 & 0 \\ 0 & 0.29508 & 0 \\ 0 & 0 & 0.46855 \end{pmatrix} \quad (4.2)$$

H is first normalized and then transform into the Cartesian reference frame shown in Figure 4.2 by:

$$H_c = T_2 * H \quad (4.3)$$

H_c is then transformed in to reciprocal space, H_c^* , following the procedure described in Chapter 3.3.2. As the experiment reports reciprocal lattice parameters, H_{DAXM}^* expressed in beamline reference frame (Figure 4.1) therefore by comparing H_c^* and H_{DAXM}^* a rotation matrix R_{beam} can be found:

$$R_{beam} = H_{DAXM}^* * H_c^* \quad (4.4)$$

Single crystal stiffness matrix for pure titanium was chosen from literature [193] and takes the following form (units in GPa):

$$C = \begin{pmatrix} 162.4 & 92 & 69 & 0 & 0 & 0 \\ 92 & 162.4 & 69 & 0 & 0 & 0 \\ 69 & 69 & 180.7 & 0 & 0 & 0 \\ 0 & 0 & 0 & 46.7 & 0 & 0 \\ 0 & 0 & 0 & 0 & 46.7 & 0 \\ 0 & 0 & 0 & 0 & 0 & 35.2 \end{pmatrix} \quad (4.5)$$

As C is defined in crystal reference frame, it need to be rotated into beamline reference frame to calculate stress (as the strain output from LaueGo is

referenced in beamline reference frame), to do so two matrices can be defined following Dunne et al [194]:

$$T_{\sigma} =$$

$$\begin{pmatrix} R_{11}^2 & R_{21}^2 & R_{31}^2 & 2R_{11}R_{21} & 2R_{21}R_{31} & 2R_{31}R_{11} \\ R_{12}^2 & R_{22}^2 & R_{32}^2 & 2R_{12}R_{22} & 2R_{22}R_{32} & 2R_{32}R_{12} \\ R_{13}^2 & R_{23}^2 & R_{33}^2 & 2R_{13}R_{23} & 2R_{23}R_{33} & 2R_{33}R_{13} \\ R_{11}R_{12} & R_{21}R_{22} & R_{31}R_{32} & R_{11}R_{22} + R_{12}R_{21} & R_{21}R_{32} + R_{22}R_{31} & R_{31}R_{12} + R_{32}R_{11} \\ R_{12}R_{13} & R_{22}R_{23} & R_{32}R_{33} & R_{23}R_{12} + R_{13}R_{22} & R_{22}R_{33} + R_{23}R_{32} & R_{32}R_{13} + R_{33}R_{12} \\ R_{13}R_{11} & R_{23}R_{21} & R_{33}R_{31} & R_{13}R_{21} + R_{11}R_{23} & R_{23}R_{31} + R_{21}R_{33} & R_{33}R_{11} + R_{31}R_{13} \end{pmatrix}$$

(4.6)

$$T_{\varepsilon} =$$

$$\begin{pmatrix} R_{11}^2 & R_{21}^2 & R_{31}^2 & R_{11}R_{21} & R_{21}R_{31} & R_{31}R_{11} \\ R_{12}^2 & R_{22}^2 & R_{32}^2 & R_{12}R_{22} & R_{22}R_{32} & R_{32}R_{12} \\ R_{13}^2 & R_{23}^2 & R_{33}^2 & R_{13}R_{23} & R_{23}R_{33} & R_{33}R_{13} \\ 2R_{11}R_{12} & 2R_{21}R_{22} & 2R_{31}R_{32} & R_{11}R_{22} + R_{12}R_{21} & R_{21}R_{32} + R_{22}R_{31} & R_{31}R_{12} + R_{32}R_{11} \\ 2R_{12}R_{13} & 2R_{22}R_{23} & 2R_{32}R_{33} & R_{23}R_{12} + R_{13}R_{22} & R_{22}R_{33} + R_{23}R_{32} & R_{32}R_{13} + R_{33}R_{12} \\ 2R_{13}R_{11} & 2R_{23}R_{21} & 2R_{33}R_{31} & R_{13}R_{21} + R_{11}R_{23} & R_{23}R_{31} + R_{21}R_{33} & R_{33}R_{11} + R_{31}R_{13} \end{pmatrix}$$

(4.7)

Where R_{ij} is the rotation matrix that maps from crystal reference frame to local reference frame, in this the rotation matrix is R_{beam} . The stiffness matrix C can then be rotated to beamline reference frame C_{beam} through:

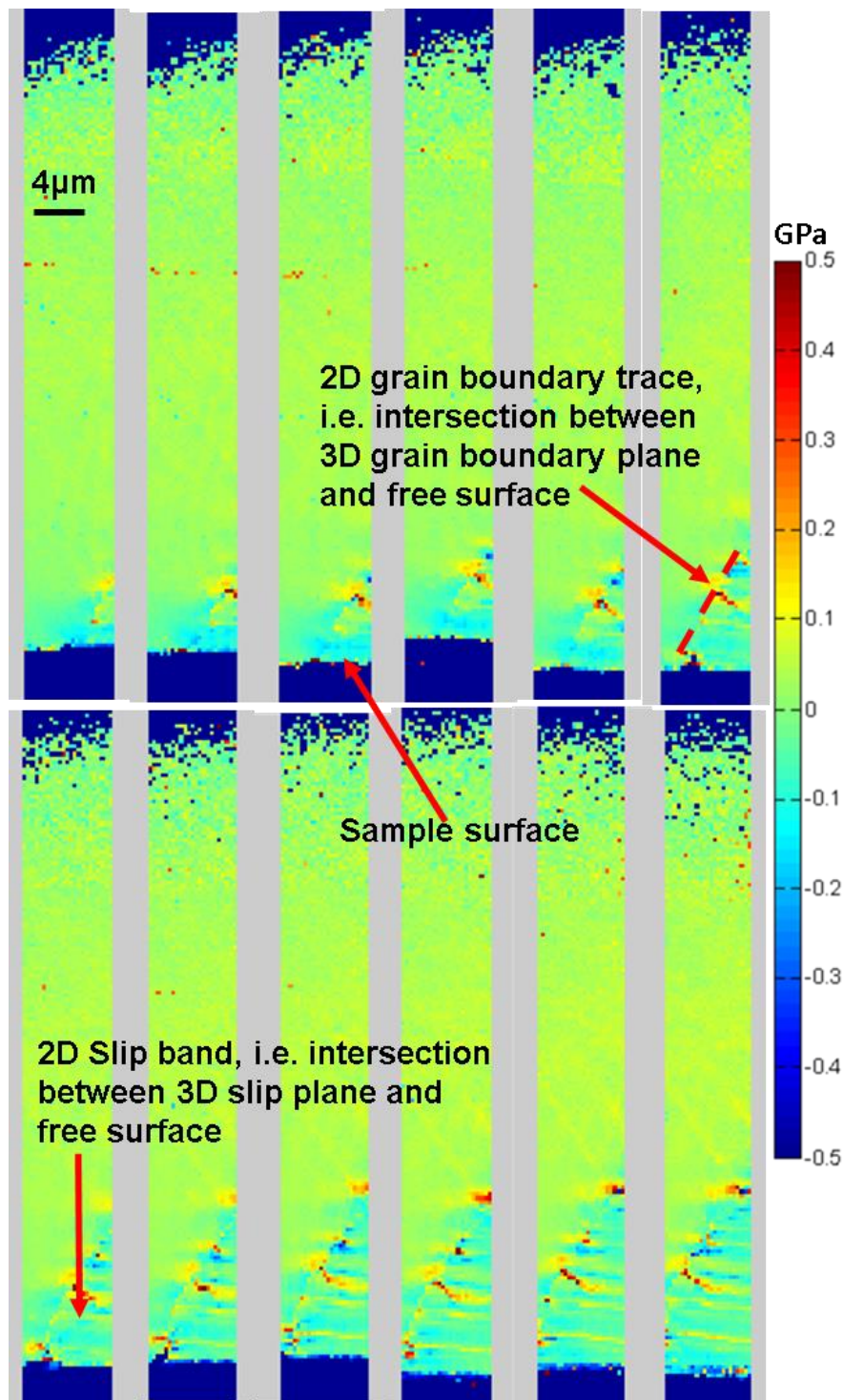
$$C_{\text{beam}} = T_{\sigma}^{-1} C T_{\varepsilon} \quad (4.8)$$

Stress is calculated using anisotropic Hook's law [195]:

$$\sigma_{\text{beam}} = C_{\text{beam}} \begin{pmatrix} \varepsilon_{\text{beam}}^{11} \\ \varepsilon_{\text{beam}}^{22} \\ \varepsilon_{\text{beam}}^{33} \\ 2\varepsilon_{\text{beam}}^{23} \\ 2\varepsilon_{\text{beam}}^{13} \\ 2\varepsilon_{\text{beam}}^{12} \end{pmatrix} \quad (4.9)$$

This experiment employ white beam diffraction which is not sensitive to lattice dilation, therefore, strain and stress obtained are deviatoric, reflecting the shape change of the crystal. The hydrostatic component (volume change) can be found by measuring the energy of one of the reflections [196], which was not performed for the current investigation.

The experimental results were indexed on-site at beamline 34-ID-E and stored in single column. These were imported into Matlab and sorted into 31 slices of images according to the periodicity of their associated 3D coordinates. The stress maps as calculated in beamline reference frame were rotated onto the active slip plane by first rotate into EBSD reference frame as defined in Figure 4.1. As now the stress is in the same reference frame as that shown in Figure 3.2b, the stress tensor can be resolved onto the active slip plane using the same rotation matrix as have been used to rotate stress tensor in Figure 3.2b. Again the σ_{13} component of the rotated stress tensor reflect the active shear on the dislocation pile up plane. All 31 slices of the rotated σ_{13} shear stress maps are shown in Figure 4.3.



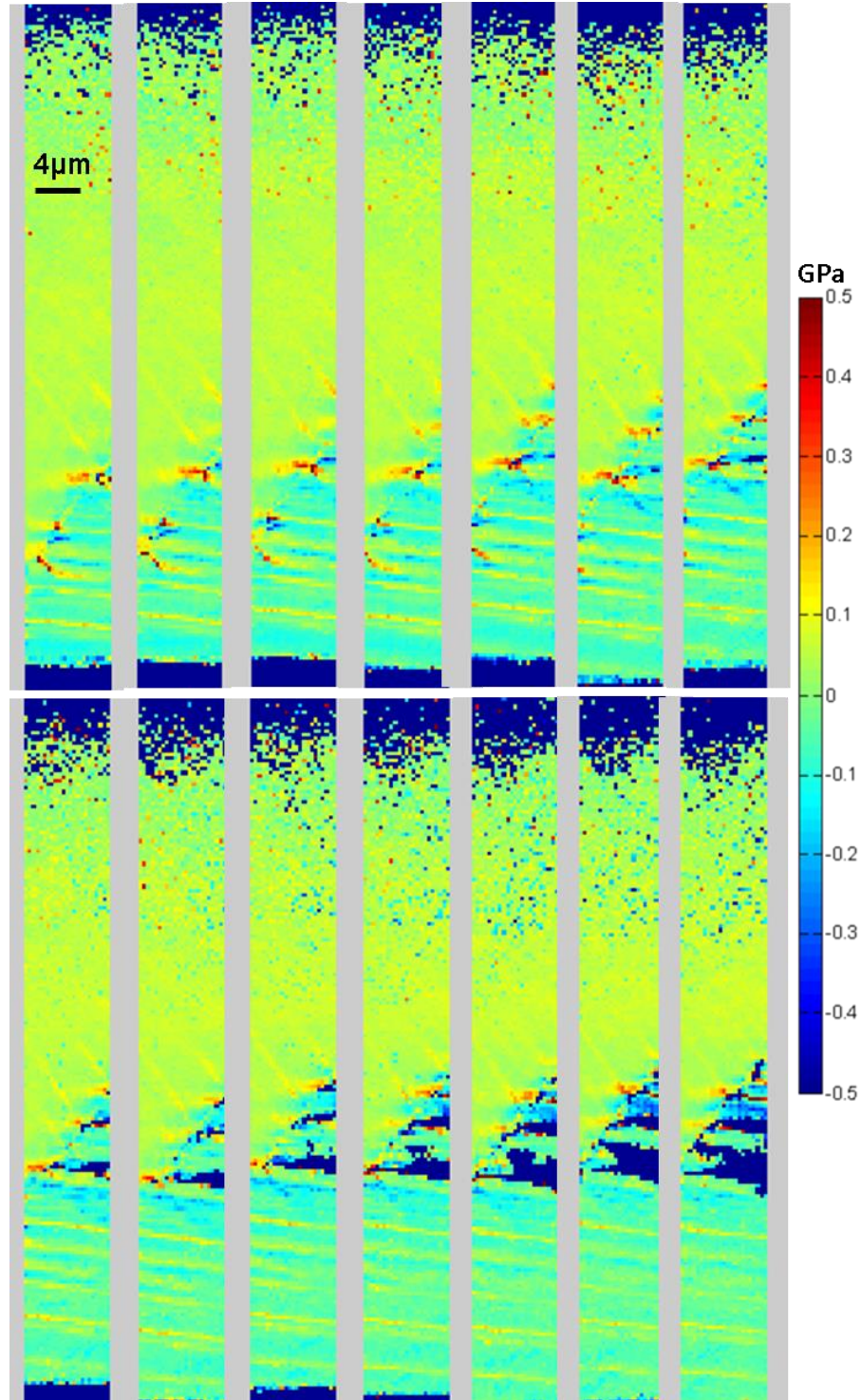


Figure 4.3: σ_{13} deviatoric stress component for all 31 slices of data probed using DAXM. The first image (top left) is the top slice of the 3D volume and the last image (bottom right) is the bottom slice of the 3D volume (refer to Figure 4.1).

For images in Figure 4.3, synchrotron beam direction is from bottom to top vertically, the noisier data towards the top of each slice indicate that beam intensity decreased as it travel deep into bulk of the sample. Generally speaking the data quality is good ~90 μm into the sample surface, within which the slip band and grain boundary interactions were analysed. As is revealed in Figure 4.3 the bottom grain (grain 1 in Figure 4.1) contains multiple slip band features as is indicated by the arrow, some of them intersect with the 2D trace of the grain boundary, and the stress associated with which were analysed in the next section. There are also other slip band that don't intersect with the grain boundary within the probed volume of data but will intersect with either the boundary of the same grain or other neighbouring grains in the extended region of the image.

A simple 3D stack of all the 31 slices of images shown in Figure 4.3 is demonstrated in Figure 4.4. This volume reconstruction was done in Avizo and can equally be done using MTEX open toolbox in Matlab, however, the latter either show the whole 3D image as in Figure 4.4 or show one internal slice at a time. This does not give a direct overall visualisation of the feature of interest in 3D, in this case, the stress field.

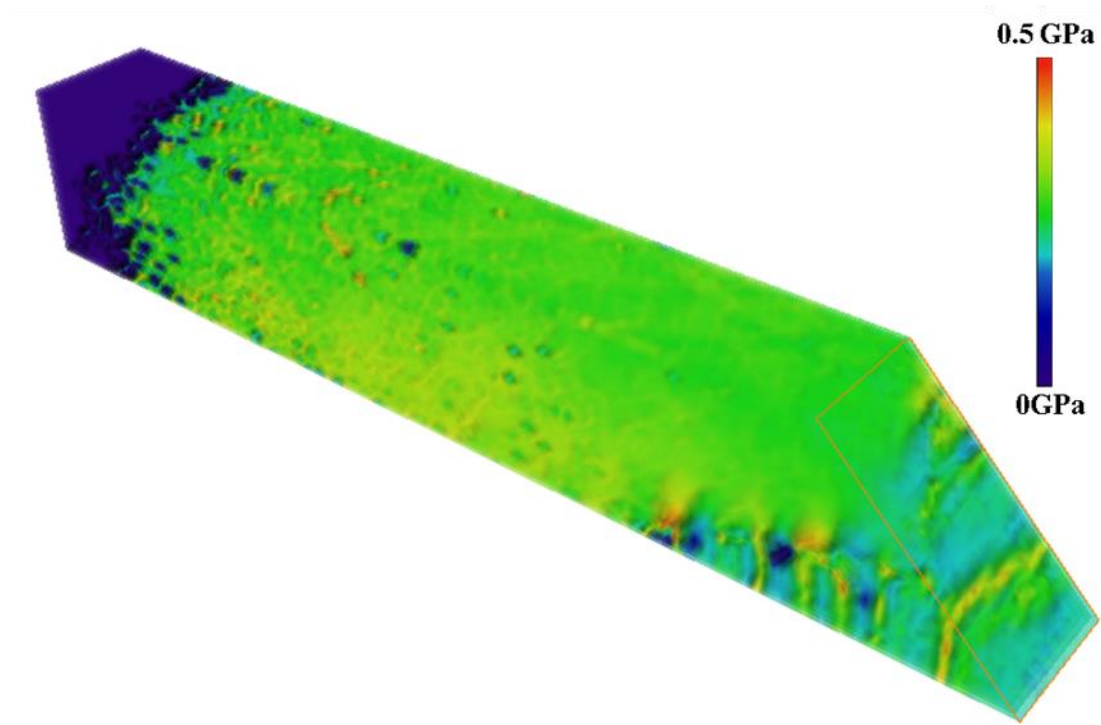
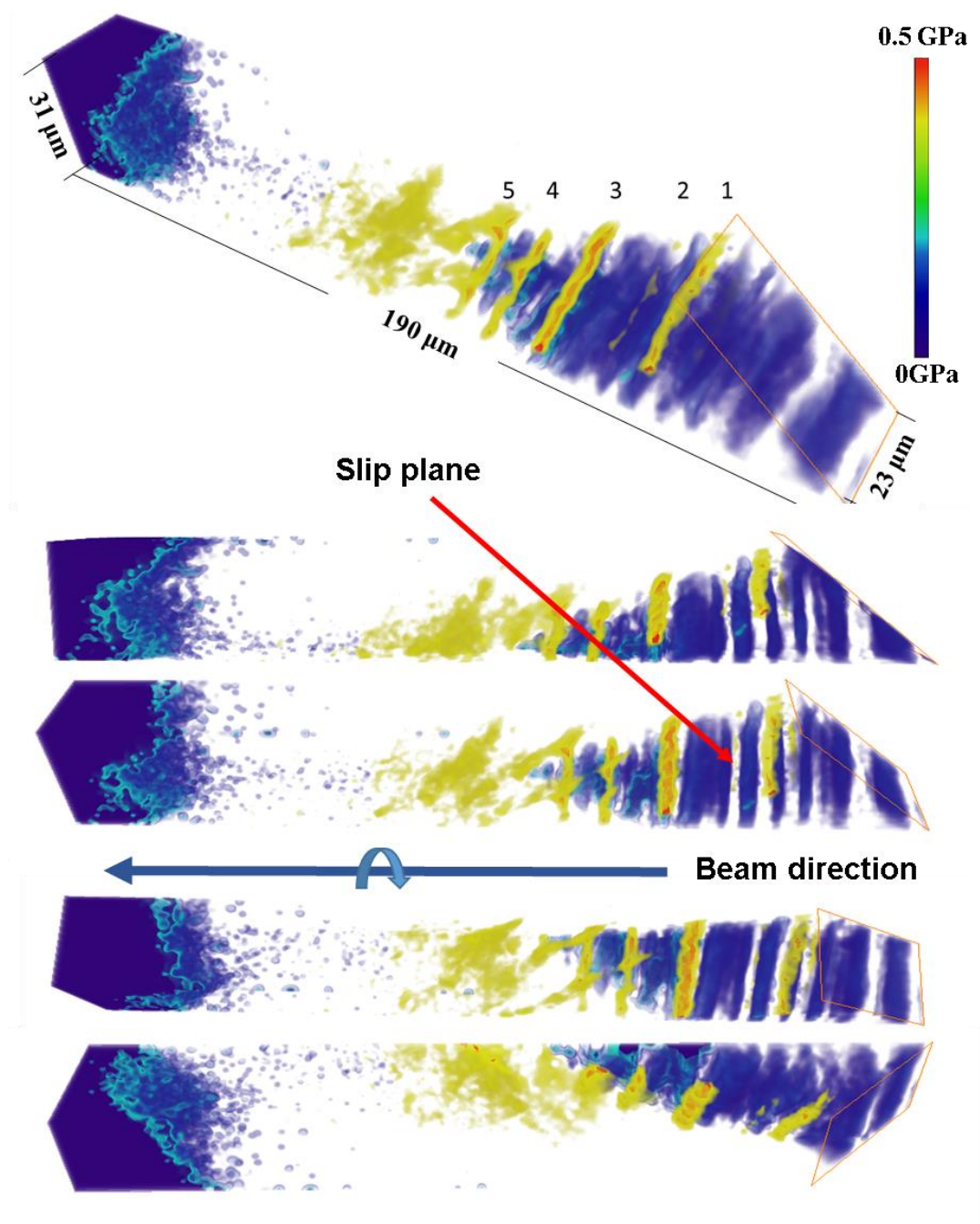


Figure 4.4: The probed volume of data visualized by stacking images in Figure 4.3 together.

The problem of visualizing only the stress concentration caused by the blocked slip planes can be solved by inspecting the colour coding in Avizo. From the colour gradients of Figure 4.4 it can be seen that the points of higher stress concentration, i.e. the place where slip plane intersects the grain boundary, generally have higher pixel intensity (yellow/red), and therefore these can be visualized independently by making pixels with lower intensity transparent, as

can be seen from the opacity histogram in Figure 4.5.. The rendered data volume is shown in Figure 4.5.



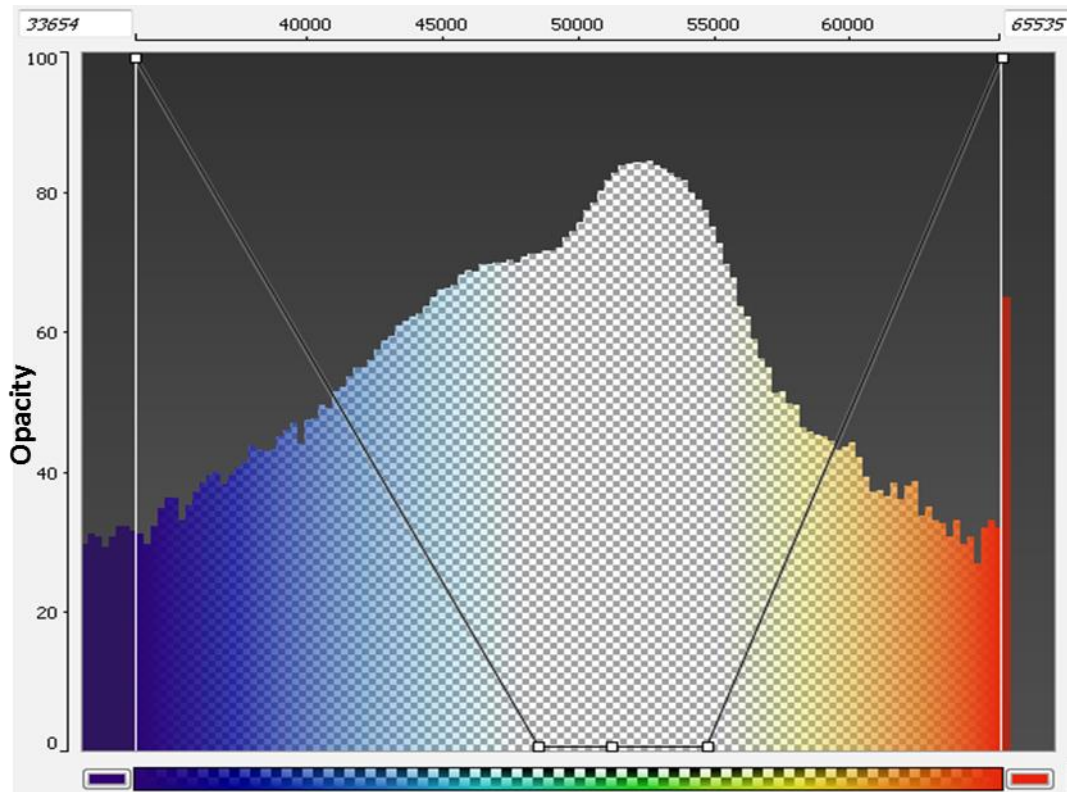


Figure 4.5: A 3D visualization of stress field associated with the blocked arrays of dislocations. First image is brought to the same perspective view as Figure 4.4 and Figure 4.1 for comparison. The other four images show a rotational view about the incident X-ray beam direction. The opacity histogram of the color coding is shown at the bottom of the figure.

Figure 4.5 effectively reveals the realistic 3D configuration of the stress field as a result of deformation incompatibility between grains. As can be seen that the blob of stress concentration which was picked up by HR-EBSD in 2D (Figure 3.2) is in 3D space a ribbon. The ribbons follow the trace of the line of intersection between slip plane and grain boundary therefore it is not difficult to imagine that this ribbon could be extended to a ring (or interrupted ring) if the whole grain sphere were probed. It is also qualitatively evident that the intensity of stress is not uniform along each ribbons. There are totally five analysable stress ribbons

as numbered in Figure 4.5 and the stress intensity along each ribbon and between the five ribbons will be quantified and compared in the next section.

4.3 Quantifying the stress intensity distribution

The stress intensity factor K were calculated using the Eshelby, Frank, and Nabarro (EFN) model similar to the procedures described in Chapter 3.3.1. For the 3D volume, stress line profile ahead of each blocked slip band was taken on the slices of images shown in Figure 4.3. An example is shown in Figure 4.6.

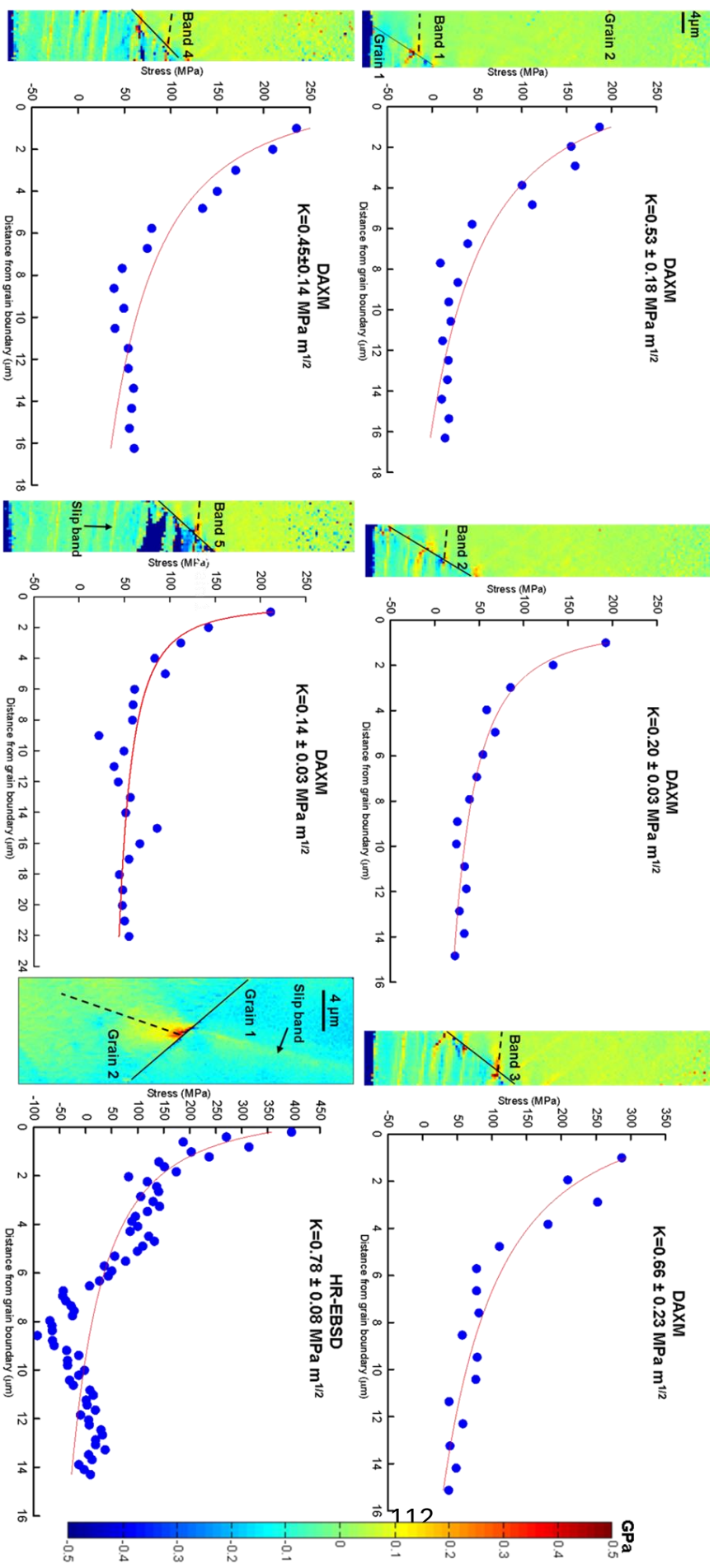


Figure 4.6: $X_{\text{beam}}\text{-}Z_{\text{beam}}$ (defined in Figure 1) slice of the deviatoric residual elastic stress maps of the 3D volume. Numbering of slip bands corresponds to the numbering in Figure 4.5. Line profile of the stress distribution was taken along each of the dashed line, the direction of which is along the slip band. As comparison, the stress intensity profile from HR-EBSD in Figure 3.2 is also shown (final image).

It can be seen from Figure 4.6 that the stress distribution in front of each of the five blocked slip plane is consistent with the EFN model. Differences can be seen in both the magnitude of the maximum stress and how these decay over distance. These differences are reflected by values of the stress intensity factor (K). To account for the uncertainties in the position of grain boundary, the place of intersection between slip band and grain boundary, and the direction of slip plane/free surface intersection (slip band), multiple line profiles were taken at each stress 'hot spot', and the K values obtained by fitting these curves were averaged. Results for the stress intensity distribution along all five line of intersections between slip plane and grain boundary are shown Figure 4.7 (a). Figure 4.7 (b) shows the average K values for each stress band. The horizontal axis represents the distance estimated relative to the first band along the grain boundary plane.

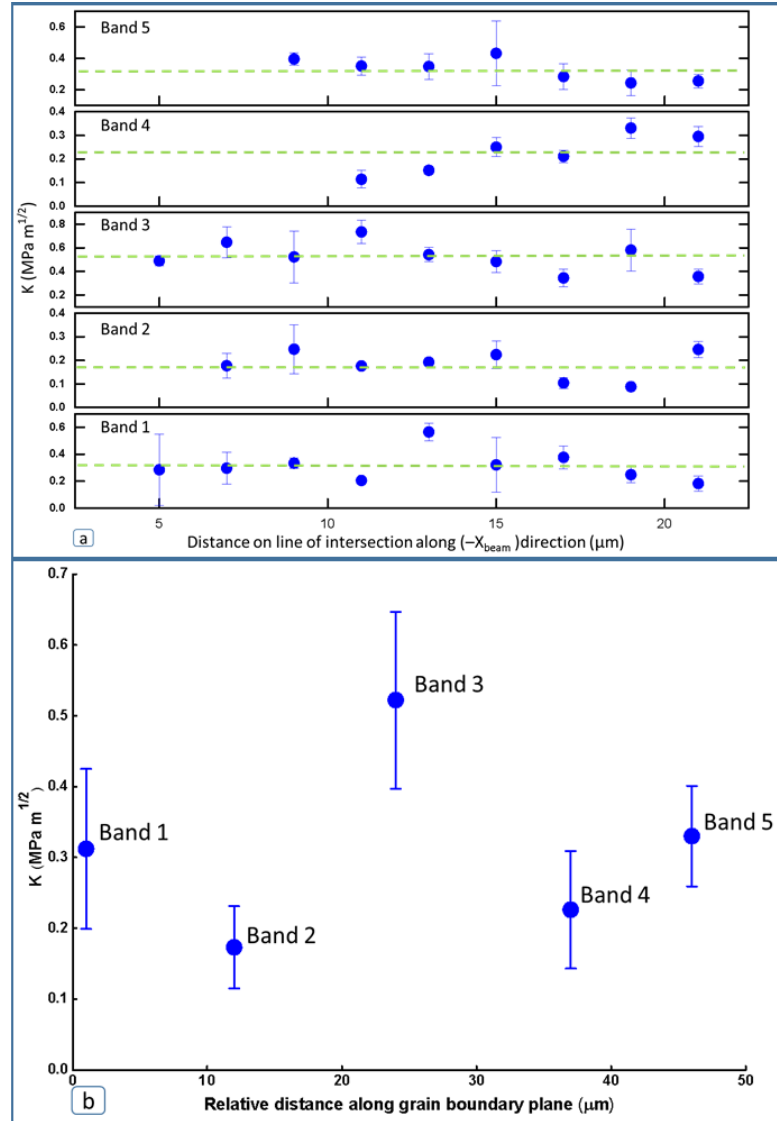


Figure 4.7: (a) stress intensity factor (K) variation along the line of intersection between slip plane and grain boundary. For each band, data points from left to right corresponds to bottom left to top right in the first image of Figure 4.5. Green dashed line indicates the average K value of each band. (b) Shows the average K value of each band plotted against approximate distance from the first band along grain boundary surface plane.

4.4 Discussion

The DAXM results capture the 3D nature of slip-plane grain boundary interactions and underpin our mechanical understanding of the effect of grain

boundaries at blocking dislocation transfer between grains. This information is important as we move away from simple empirical 'Hall-Petch' approaches which have been recently discussed by Dunstan et al [97], which suggest that the Hall-Petch relationship may not be most appropriate at describing the mechanical role of smaller grains on the strength of polycrystalline aggregates.

In the present work, the HR-EBSD and DAXM results represent a bottom-up mesoscale understanding of local stress rises associated with these interactions which now must be captured by modelling efforts more accurately to outline the contribution of individual grain boundaries on mechanical strength and performance. This is necessarily involved, as crystal plasticity approaches may 'smear out' the local stress fields associated with individual dislocation pile ups. Furthermore, as an industrial challenge these observations and modelling approaches must be homogenized appropriately to capture many grains in larger component scale analyses to be used by design engineers. In particular, contributions from a similar slip system interacting with subtle variations in the grain boundary geometry show some variation in local stress intensity (Figure 4.7) which is likely associated with both the number of dislocations within the pile-up [197] as well as the local grain boundary structure [80].

In this current work, both the HR-EBSD measurement of slip band/grain boundary interaction and DAXM measurement of slip plane/grain boundary interaction confirm that the stress state due to blocked array of dislocations is consistent with the description of the EFN model (Figure 4.6). Indexing of the DAXM pattern from near the free surface of the sample was poor, therefore direct comparison between the HR-EBSD stress intensity and that measured by

DAXM cannot be made. Slightly less direct routes of assessing the consistency of the data obtained by the two methods must be used. Potential issues with the EBSD data are the unknown stress states at the reference point and the restriction to measurements very close to the surface. By comparison, DAXM allows probing over a range of depths but not near the free surface and gives stresses averaged over substantially larger volumes than does EBSD. The DAXM measurements give absolute (deviatoric) strains rather than relative to some reference states. In the undeforming grain 2, DAXM shows that the strain values away from the localized concentrations at the grain boundary are small and show little variation. This gives reasonable confidence that the reference points used in the EBSD analysis corresponds to relatively low stress regions and so would not adversely affect the analysis.

Band 1 (Figure 4.5) captures the stress variation from close to the free sample surface to bulk (in the absence of a suitable DAXM surface measurement) as its rightmost data point (Figure 4.7(a)) is about only 5 micro-meters away from the free surface. The measured stress intensity value at this point is 0.18 ± 0.01 (MP m^{1/2}), about 40% lower than the average value of 0.31 ± 0.11 (MP m^{1/2}). Moving away from this point to the bulk of the sample (to the left of the graph), the stress intensity fluctuates around the mean value, reflecting the local grain boundary effect, most notably the local curvature which have been confirmed to present in various alloy systems through field ion microscopy [81, 198] and more advanced high precision focused ion beam serial sectioning technique [199]. It is also possible that intrinsic grain boundary defects as well as local chemical variations could adjust the pile-up strength and these have not been

sampled here. These fluctuations of K are, however, of similar magnitude to the error bars associated with determining K from the stress profile. The variation of K along slip band 1 (and indeed for the other slip bands) does not suggest any strong systematic variation with depth from the free surface over length scales of several microns. Moreover, the fluctuation of stress intensity over a single line of intersection between slip plane and grain boundary (Figure 4.7 (a)) is within similar range with those obtained from HR-EBSD on different grain boundaries [197], this seems to suggest that for higher length scale models the slip transmission criteria can be treated as a probabilistic process.

Variations of stress intensity are seen both along the same line of intersection between slip plane and grain boundary (Figure 4.7(a)) and between the different bands (Figure 4.7(b)). The range of K is as high as 68% of the mean along band 1 and 67% between band 2 and band 3. This large variation of stress intensity magnitude on the grain boundary poses a question of to what extent could the damage nucleation site be predicted with accuracy. While it is relatively easy to identify the most damage prone site (band 3 in this case), the exact position for the most probable damage nucleation along band 3 (or any other line of intersections) is likely to be stochastic, depending on the interactions between dislocations and local grain boundary features. This effect is likely to lead to two scenarios which requires 3D characterization, and 2D observations will be less than conclusive:

1. If damage nucleation is observed as a surface stress riser in 2D, there is a possibility that large population of damage already exist beneath the sample surface;

2. Damage not observed in 2D does not mean it is not nucleated along the same microstructural feature in 3D.

The angle between the grain boundary plane and the bottom plane of the data volume was measured to be about 15° to the left and 27° to the right (as shown in Figure 4.1), indicating the presence of grain boundary curvature. The M factor that represent local alignment of slip system across the grain boundary was calculated at both left and right side of the data volume and two approaches were used: the first one utilizing the angle between slip plane normal as was suggested by Werner et al. [127] and the second one using the angle between the line of intersections between slip planes and the grain boundary as was suggested in the original LRB criteria [200]. The reason Werner et al. propose to use slip plane normal is that it is more experimentally accessible and is independent of grain boundary curvature, while the knowledge of the line of intersection between slip plane and grain boundary usually require either synchrotron or serial sectioning experiment. The results are shown in Table 4.1.

	Basal			Prismatic		
	$\langle 11-20 \rangle (0001)$	$\langle 1-210 \rangle (0001)$	$\langle -2110 \rangle (0001)$	$\langle 11-20 \rangle (1-100)$	$\langle 1-210 \rangle (-1010)$	$\langle -2110 \rangle (01-10)$
M(Werner)	0.072	0.005	0.067	0.13	0.011	0.286
M(R-LRB-left)	0.054	0.004	0.05	0.257	0.02	0.261
M(R-LRB-right)	0.016	0.001	0.015	0.308	0.02	0.243
$\langle a \rangle$ Pyramidal						
	$\langle 11-20 \rangle (1-101)$	$\langle 1-210 \rangle (-1011)$	$\langle -2110 \rangle (01-11)$	$\langle 11-20 \rangle (1-10-1)$	$\langle 1-210 \rangle (-101-1)$	$\langle -2110 \rangle (01-1-1)$
M(Werner)	0.035	0.004	0.243	0.141	0.011	0.144
M(R-LRB-left)	0.078	0.016	0.102	0.235	0.012	0.133
M(R-LRB-right)	0.159	0.013	0.125	0.172	0.015	0.063
$\langle c+a \rangle$ Pyramidal						
	$\langle 11-23 \rangle (1-101)$	$\langle -1-123 \rangle (10-11)$	$\langle 1-213 \rangle (0-111)$	$\langle -12-13 \rangle (1-10-1)$	$\langle -2113 \rangle (10-1-1)$	
M(Werner)	0.397	0.258	0.435	0.447	0.616	
M(LRB-left)	0.783	0.509	0.774	0.795	0.561	
M(LRB-right)	0.937	0.609	0.778	0.799	0.523	
$\langle c+a \rangle$ Pyramidal						
$\langle 2-1-13 \rangle (0-11-1)$	$\langle 11-2-3 \rangle (1-101)$	$\langle -1-12-3 \rangle (10-11)$	$\langle 1-21-3 \rangle (0-111)$	$\langle -12-1-3 \rangle (1-10-1)$	$\langle -211-3 \rangle (10-1-1)$	$\langle 2-1-1-3 \rangle (0-11-1)$
0.921	0.258	0.397	0.447	0.435	0.921	0.616
0.839	0.509	0.783	0.795	0.774	0.839	0.561
0.781	0.609	0.937	0.799	0.778	0.781	0.523

Table 4.1: The geometric alignment factor M calculated from LRB criteria using the angle between line of intersections between slip plane and grain boundary and from the Werner approximation which uses the angle between slip plane normal instead of that between lines of intersections.

It can be seen from Table 4.1 that both the true LRB criteria and Werner approximation are able to indicate which slip system in the neighbouring grain have the maximum alignment factor with the incoming slip system. However maximum alignment appears for different variant of the same slip system. This variation of slip system alignment along grain boundary plane is consistent with the note made by Clark et al. [201] which says that slip transfer to different slip systems in the neighbouring grain is expected when grain boundary curvature is present. It follows that the M factor shifting as shown in Figure 4.1 would be more significant when the sampled volume is bigger, i.e. when more grain boundary curvatures are captured.

The variations of stress intensity over single line of intersections is not likely to reflect the effect of global grain boundary curvature, as their maximum span over the measured volume of data is only about 18 μm (Figure 4.5) yet variation of the average stress intensity of each band along the grain boundary plane, spanning about 50 μm , is likely to be affected by the global grain boundary curvature. The most direct effect of global grain boundary curvature is on the mean free path of the dislocation pile up and as the more dislocation is present in the pile up plane the higher the stress intensity this will result in the neighbouring grain, as is shown in Figure 3.5 in Chapter 3.3.1. Variation in the average stress intensity for different slip plane/grain boundary interaction also indicates that different part of grain boundary have different barrier strength to dislocation transmission (direct or indirect).

Figure 4.6 compares the stress profile in front of the five blocked slip bands extracted from five slices in the X_{beam} and Z_{beam} plane of the DAXM measurement together with the one from HR-EBSD measurement. It can be seen that the stress profiles captured from both techniques converge with the EFN model [110], but HR-EBSD offers more detail of the stress distribution than the DAXM technique due to better spatial resolution achievable in SEM. This difference in spatial resolution between the two techniques may affect the measured stress profiles, especially the stress distribution within the initial 2 μm from grain boundary. The data point $\sim 0.8\mu\text{m}$ away from the grain boundary shows a markedly higher stress value for HR-EBSD than the DAXM measurement, which is likely due to positioning of the electron beam near a discrete dislocation toward the head of the pile-up [129] or possibly as a result

of local chemical effect [202], while the DAXM measurement maybe averaged over too large a volume to capture such local effects. Moving away from the locations very close to grain boundary ($> 1 \mu\text{m}$), the stress field probed by HR-EBSD and DAXM have shown good accordance in both magnitude and distribution. The variation of stress intensity between different slip band/grain boundary interactions result in the difference in the fitted K value between HR-EBSD and DAXM as shown in Figure 4.6.

4.5 Conclusion

This work presents the first three dimensional measurement and visualization of the local stress elevations due to dislocation-grain boundary interactions. The stress field due to slip band and grain boundary interaction was probed using DAXM and were compared with previous HR-EBSD investigation. The results demonstrates a similar strain/stress measurement capability between the two techniques. While the HR-EBSD technique provides more information in a much more local length scale, the stress field is based on a reference whose stress state is unknown. As a comparison, DAXM has a coarser spatial resolution but offers absolute stress state measurement and information in 3D.

The stress intensity was found to vary significantly along individual line of intersection between slip plane and grain boundary as well as between different lines of intersections. The variations of stress intensity is likely due to local irregularities and global curvatures along grain boundary plane and indicate

more than five parameters to completely describe the characteristics of grain boundary.

These discoveries allow us to conclude that it is now possible to pin point which dislocation pile up is likely to lead to material damage initiation (void, crack, etc.) by a systematic assessment of position dependent strength of a single grain boundary. However, a stochastic approach might be needed to capture in more detail the damage nucleation behaviour of the most susceptible dislocation pile ups. This work points to a critical need for physically based dislocation based models that incorporate the realistic shape of the grain boundary, which may be captured by high fidelity 2D and 3D techniques or novel test strategies.

Chapter 5 Geometrically necessary dislocation accumulation due to blocked array of dislocations

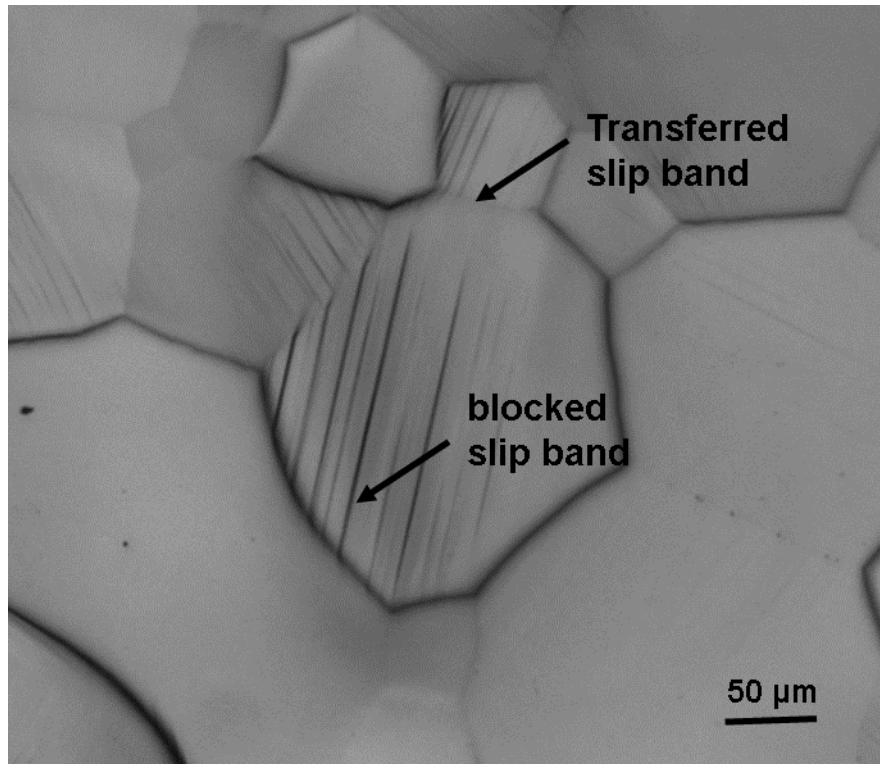


Figure 5.1 Optical image showing examples of both blocked slip band and transferred slip band. The blocked slip band as indicated by the arrow was studied using both HR-EBSD and DAXM.

In this chapter the GND distribution generated as a result of slip band been blocked at a grain boundary (indicated by the arrow in Figure 5.1) will be demonstrated and compared between 2D EBSD data discussed in Chapter 3.3.1 (Figure 3.2) and the 3D DAXM investigation of the interactions in the same grain pair as is presented in Chapter 4. Although the exact slip band and grain boundary interaction case shown in Figure 3.2 was not captured by DAXM due to poor diffraction pattern indexing near the free surface, they are

from the same grain pair and are experiencing the similar meso scale geometric constraints.

It has been noticed [111] that in case of no meso-scale slip transfer, discrete dislocation accumulation were found with particular type of Burger's vector higher in population than others. This indicates that in some cases even the incident slip band is observably blocked, dislocation transfer could happen in micro or nano scale. The word 'transfer' here could be direct transfer or nucleation of new dislocation sources. The density of the transferred dislocation and their arrangement relative to the local crystal lattices have implications on the slip transfer mechanism and subsequent microstructure evolution.

5.1 3D rotation gradient and GND density calculation

The method for this calculation follows the bloodline of HR-EBSD [111, 173, 176, 178] but with extension to incorporate the gradients in the third direction and therefore including all nine component of Nye's tensor [174].

The DAXM data set was input into Igor Pro (www.wavemetrics.com/) in which a finite lattice rotation matrix for each data point relative to a hand-picked point in each grain was calculated. To get the infinitesimal rotation tensor, a local cubic kernel containing 27 voxels was extracted from the data volume centred at each interrogation point. The data in the kernel was filtered to eliminate those voxels from outside the data volume and from different grain. After filtering, a 'useful' kernel should contain, in addition to the central voxel, at least one voxel in each of the X_{beam} , Y_{beam} and Z_{beam} (figure 4.1) directions to capture lattice

rotations associated with that direction. Then the disorientation matrix [111] was calculated between each voxel in the kernel and the centre voxel of the kernel:

$$\omega = R_{\text{centre}}^T * R_{\text{kernel}} \quad (5.1)$$

This gives the disorientation matrix for all kernel voxels in the central voxel reference frame and then the infinitesimal lattice rotation components (ω_{12} ω_{13} ω_{23}) can be approximated by: $(\omega_{ij} - \omega_{ji})/2$.

The rotation gradient in all three directions in the kernel can be found by fitting the kernel to a hyper plane:

$$\omega_{ij} = g_x X + g_y Y + g_z Z + C \quad (5.2)$$

Where $g_x = \frac{\partial \omega_{ij}}{\partial x}$ $g_y = \frac{\partial \omega_{ij}}{\partial y}$ $g_z = \frac{\partial \omega_{ij}}{\partial z}$ and C is constant. Equation 5.2 can be written in full as:

$$\begin{pmatrix} \omega_{ij}^1 \\ \vdots \\ \omega_{ij}^n \end{pmatrix} = \begin{pmatrix} X^1 Y^1 Z^1 1 \\ \vdots \\ X^n Y^n Z^n 1 \end{pmatrix} (g_x \quad g_y \quad g_z \quad C) \quad (5.3)$$

In which ω_{ij}^n refer to the infinitesimal rotation tensor of the n^{th} kernel and X^n Y^n and Z^n are its associated spatial coordinates. The fitting was performed using least square minimization process within Matlab. The measured 9 curvature components, $\frac{\partial \omega_{jk}}{\partial x_i}$ (neglecting elastic strain gradient) are linked to dislocation density by expanding equation 2.52 (Chapter 2.3.3) into:

$$\begin{pmatrix} b_1^1 l_1^1 - \frac{1}{2} \mathbf{b}^1 \mathbf{l}^1 & \cdot & b_1^s l_1^s - \frac{1}{2} \mathbf{b}^s \mathbf{l}^s \\ b_1^1 l_2^1 & \cdot & b_1^s l_2^s \\ b_1^1 l_3^1 & \cdot & b_1^s l_3^s \\ b_2^1 l_1^1 & \cdot & b_2^s l_1^s \\ b_2^1 l_2^1 - \frac{1}{2} \mathbf{b}^1 \mathbf{l}^1 & \cdot & b_2^s l_2^s - \frac{1}{2} \mathbf{b}^s \mathbf{l}^s \\ b_2^1 l_3^1 & \cdot & b_2^s l_3^s \\ b_3^1 l_1^1 & \cdot & b_3^s l_1^s \\ b_3^1 l_2^1 & \cdot & b_3^s l_2^s \\ b_3^1 l_3^1 - \frac{1}{2} \mathbf{b}^1 \mathbf{l}^1 & \cdot & b_3^s l_3^s - \frac{1}{2} \mathbf{b}^s \mathbf{l}^s \end{pmatrix} \begin{pmatrix} \rho_1 \\ \cdot \\ \rho_s \end{pmatrix} = \begin{pmatrix} \frac{\partial \omega_{23}}{\partial x_1} \\ \frac{\partial \omega_{31}}{\partial x_1} \\ \frac{\partial \omega_{12}}{\partial x_1} \\ \frac{\partial \omega_{23}}{\partial x_2} \\ \frac{\partial \omega_{31}}{\partial x_2} \\ \frac{\partial \omega_{12}}{\partial x_2} \\ \frac{\partial \omega_{23}}{\partial x_3} \\ \frac{\partial \omega_{31}}{\partial x_3} \\ \frac{\partial \omega_{12}}{\partial x_3} \end{pmatrix} \quad (5.4)$$

Where ρ_i is the density of the i^{th} type of dislocation and b_i^s and l_i^s are the components of the Burger's vector and line direction of the s^{th} type of dislocation in the x_i direction.

For titanium in this analysis, dislocations were assumed to be either edge or screw type. This gives rise to potentially 33 types of dislocation consisting of 3 <a> screw, 3 <a> basal, 3 <a> prismatic, 6 <a> pyramidal, 6 <c+a> screw, 12 <c+a> pyramidal. The use of 33 dislocations to support 9 lattice curvatures rendering dislocation densities under determined and therefore other supporting parameters need to be used to obtain a physically based solution. This problem can be solved by employing a L^1 minimization scheme and minimizing the total line energy of dislocations by using a weighing factor w_s [111, 176]:

$$w_s(\text{screw}) = b_s^2 \quad (5.5)$$

$$w_s(\text{edge}) = \frac{b_s^2}{(1-\nu)} \quad (5.6)$$

Where b_s is the Burger's vector length of the s^{th} dislocation and ν is Poisson's ratio. For GND density calculation the number of dislocations are doubled (66 dislocations) to account for negative Burger's vectors but with the constraint that the total densities must be positive. For figures in the following section, the positive and negative dislocations are shown together and a sign applied to the densities to indicate the sense of the dislocation.

5.2 3D GND density distribution

The average GND density for all 33 types of dislocations in front of slip band 3 (Figure 4.5) in both grain 1 and grain 2 are demonstrated in the following diagram:

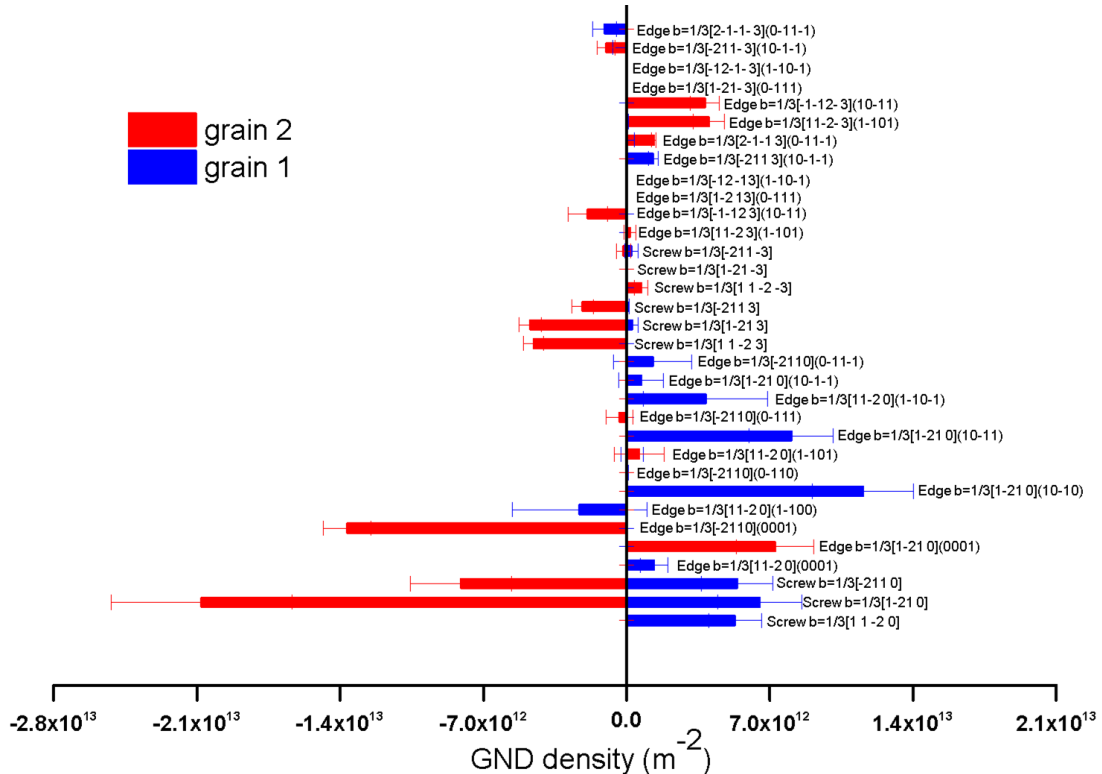
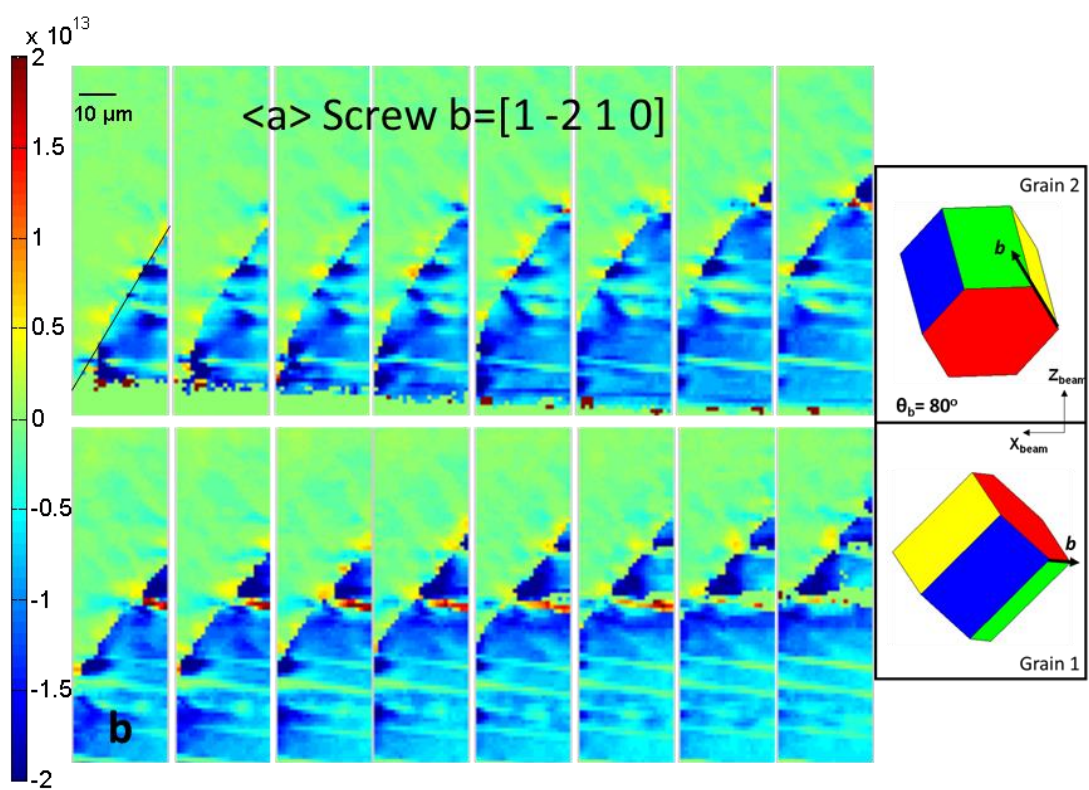
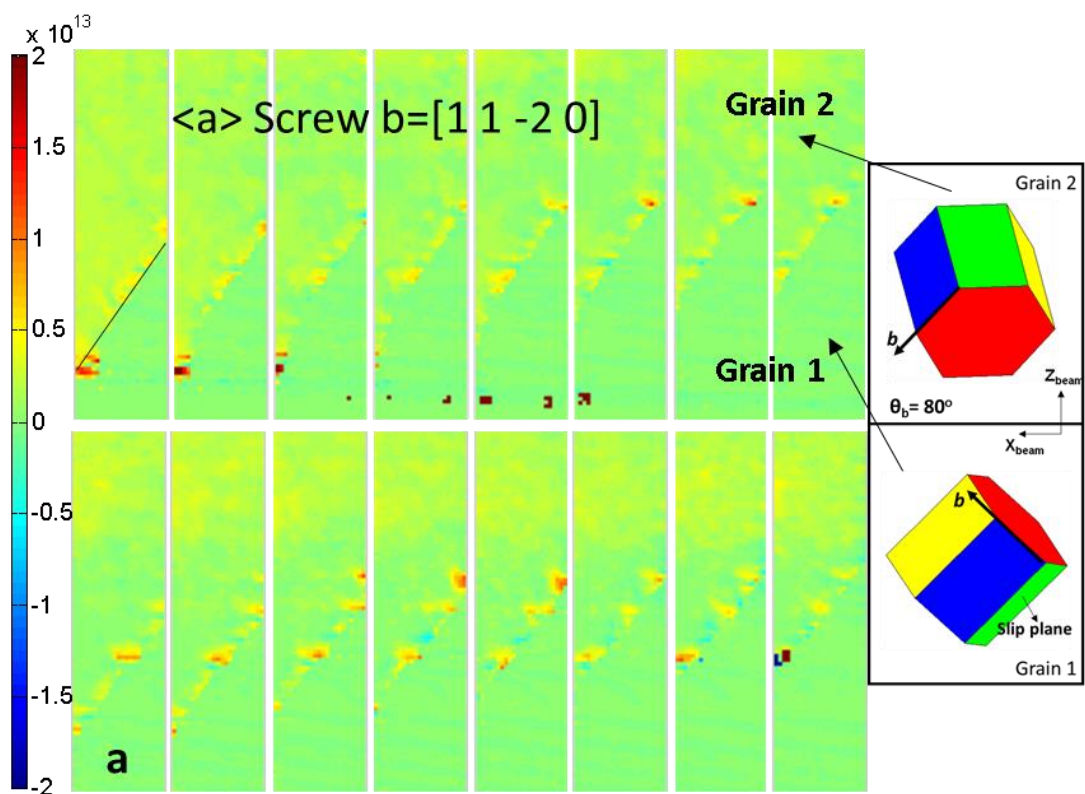
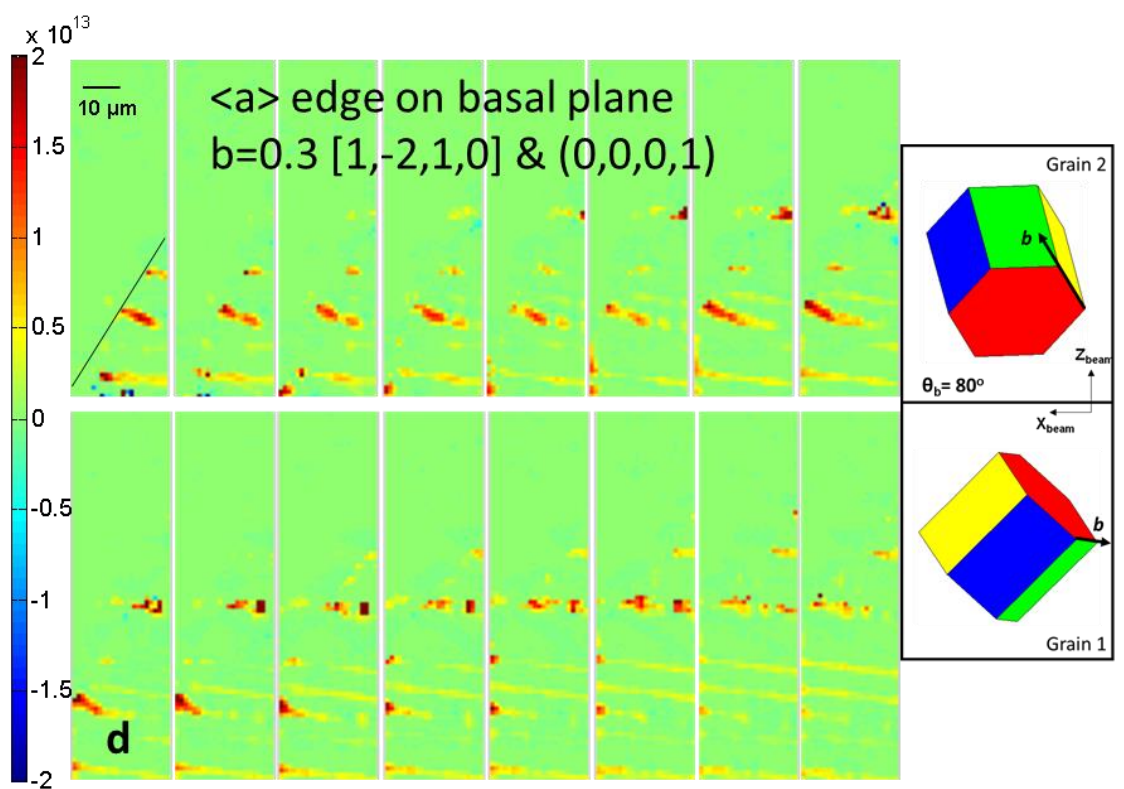
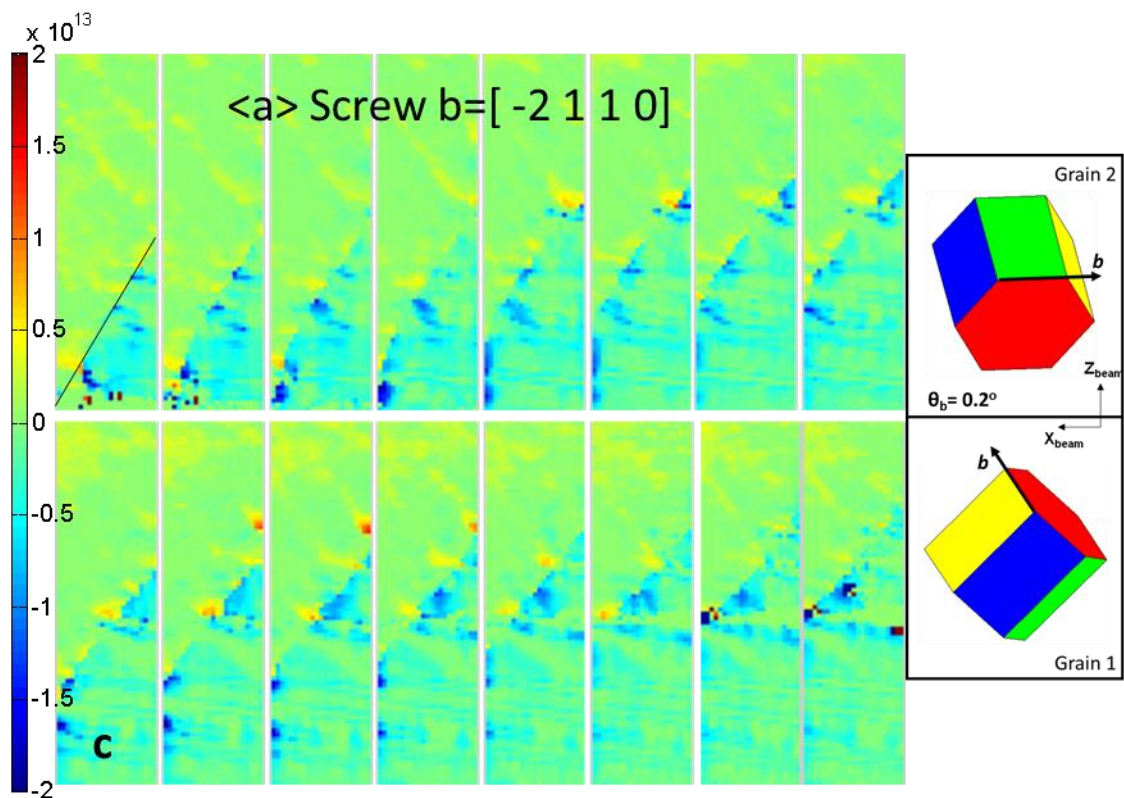
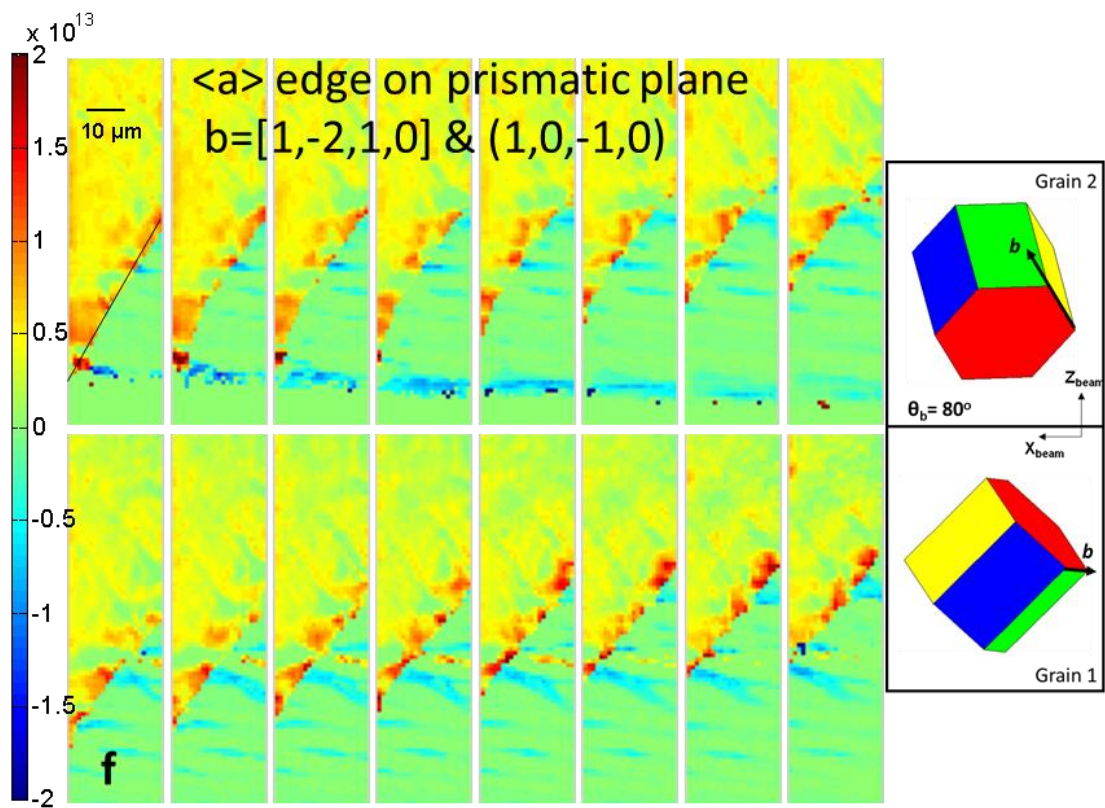
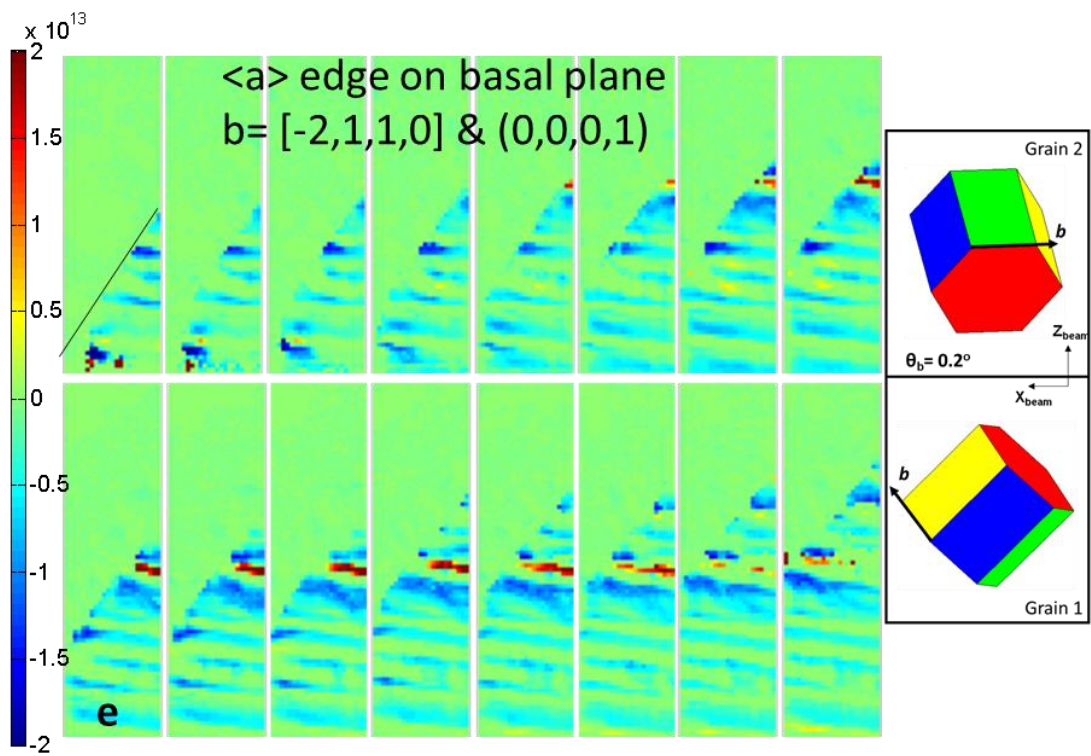


Figure 5.2: Average GND density for all 33 dislocation types in titanium at slip plane 3 and grain boundary intersection, measured from the DAXM experiment.

Dislocation types giving rise to significant lattice curvatures and therefore discernible GND densities, i.e. 3 $\langle a \rangle$ screw, 2 $\langle a \rangle$ edge on basal plane, 1 $\langle a \rangle$ edge on prismatic plane, and 2 $\langle c+a \rangle$ edge on pyramidal plane, are shown in Figure 5.3. The maps show slices 12 to 27 of the data volume and in order to save space each slice was cropped to incorporate only the regions around grain boundary. Together with the GND maps are the schematics of the HCP crystal cell in the X_{beam} and Z_{beam} plane, i.e. viewing top-down from the positive axis of the Y_{beam} direction (Figure 4.1). Slip trace analysis was performed in the X_{beam} and Z_{beam} plane, it was found that the traces of slip bands in grain 1 as can be observed from stress maps in Figure 4.3 are due to dislocation pile up on the green prismatic plane of the HCP crystal schematic for the lower grain. This is highlighted in Figure 5.3 (a). The Burger's vector corresponding to the GND type was indicated by a black arrow on the HCP crystal. On the first image of each set of maps the grain boundary position is indicated by a solid line, but bear in mind that the position of which shifts upward gradually on successive slice of images due to inclination of the grain boundary plane. The angle θ_b represents the angle between the $\langle a \rangle$ type Burger's vector on the active slip plane (green plane in the HCP crystal of grain 1) and the indicated Burger's vector in grain 2 (θ_b is not the angle between the two indicated Burger's vectors on the HCP crystal schematics).







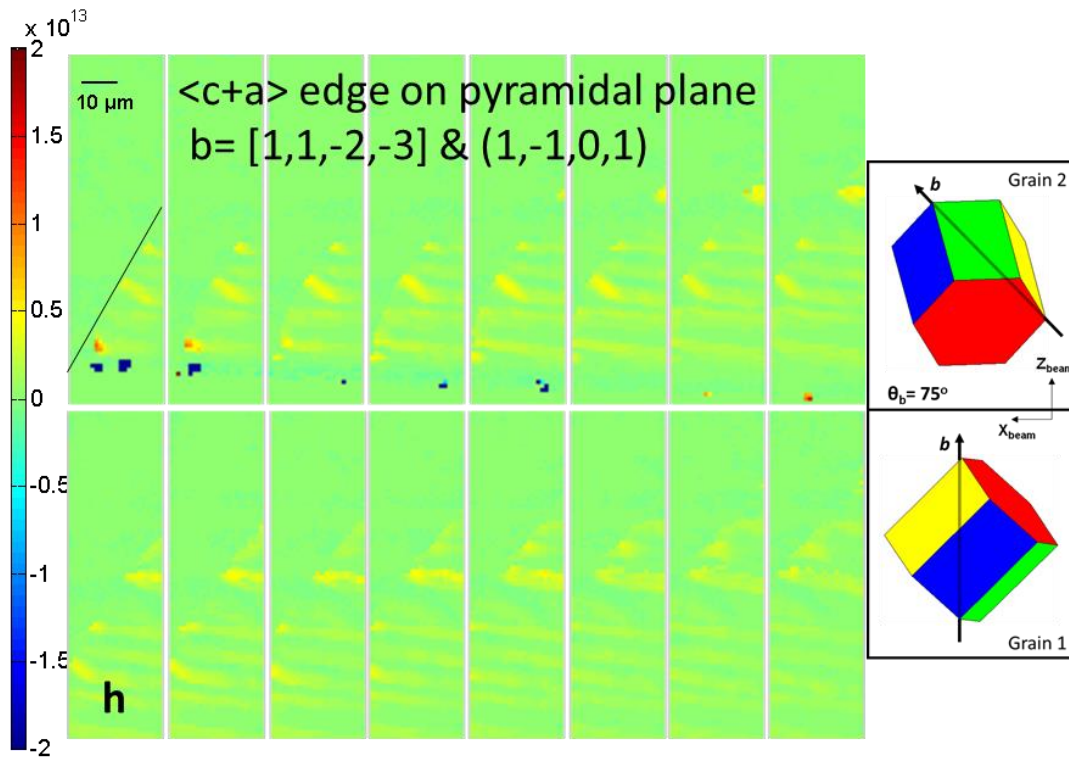
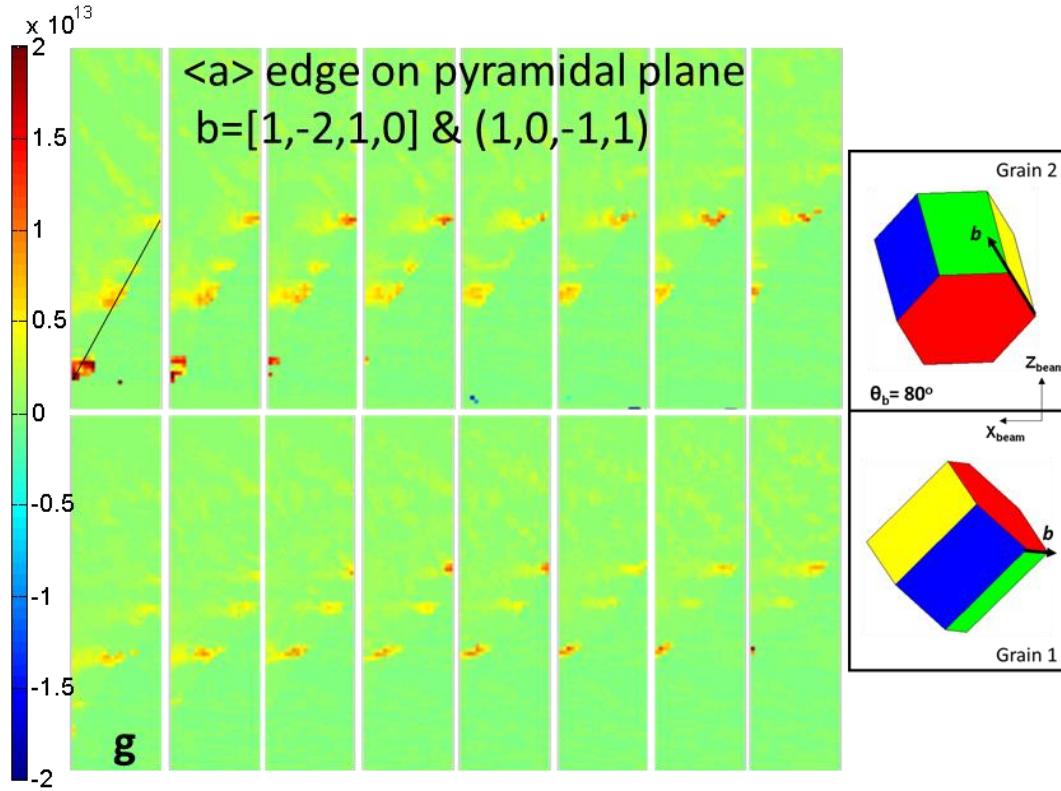


Figure 5.3: GND maps of each dislocation type giving rise to the experimentally captured lattice curvatures. Grain boundary position is indicated by a solid line. Below

the solid line is grain 1 and above it is grain 2 as is indicated in (a). Dislocation pile up plane is coloured blue in the HCP crystal representing grain 1 as is indicated in (a). Burger's vector indicates the corresponding dislocation type as labelled on top of each set of images.

As is revealed by Figure 5.3 totally 8 dislocation types are required in significant density to account for the measured lattice curvature, these are: 3 $\langle a \rangle$ screw, 2 $\langle a \rangle$ edge on basal plane, 1 $\langle a \rangle$ edge on prismatic plane, 1 $\langle a \rangle$ edge on pyramidal plane, and 1 $\langle c+a \rangle$ edge on pyramidal plane. Among these dislocations, all three $\langle a \rangle$ screw types of dislocation seems to be generated in grain 2 at the places where slip bands in grain 1 intersects the grain boundary (Figure 5.3 (a-c)). The $[11\bar{2}0]$ type screw dislocation density in Figure 5.3 (a) have about $8.85 \times 10^{12} \text{ m}^{-2}$ at the highest density site, the $[1\bar{2}10]$ screw in Figure 5.3(b) have about $7.43 \times 10^{12} \text{ m}^{-2}$, and the $[\bar{2}110]$ screw in Figure 5.3(c) have about $7.72 \times 10^{12} \text{ m}^{-2}$.

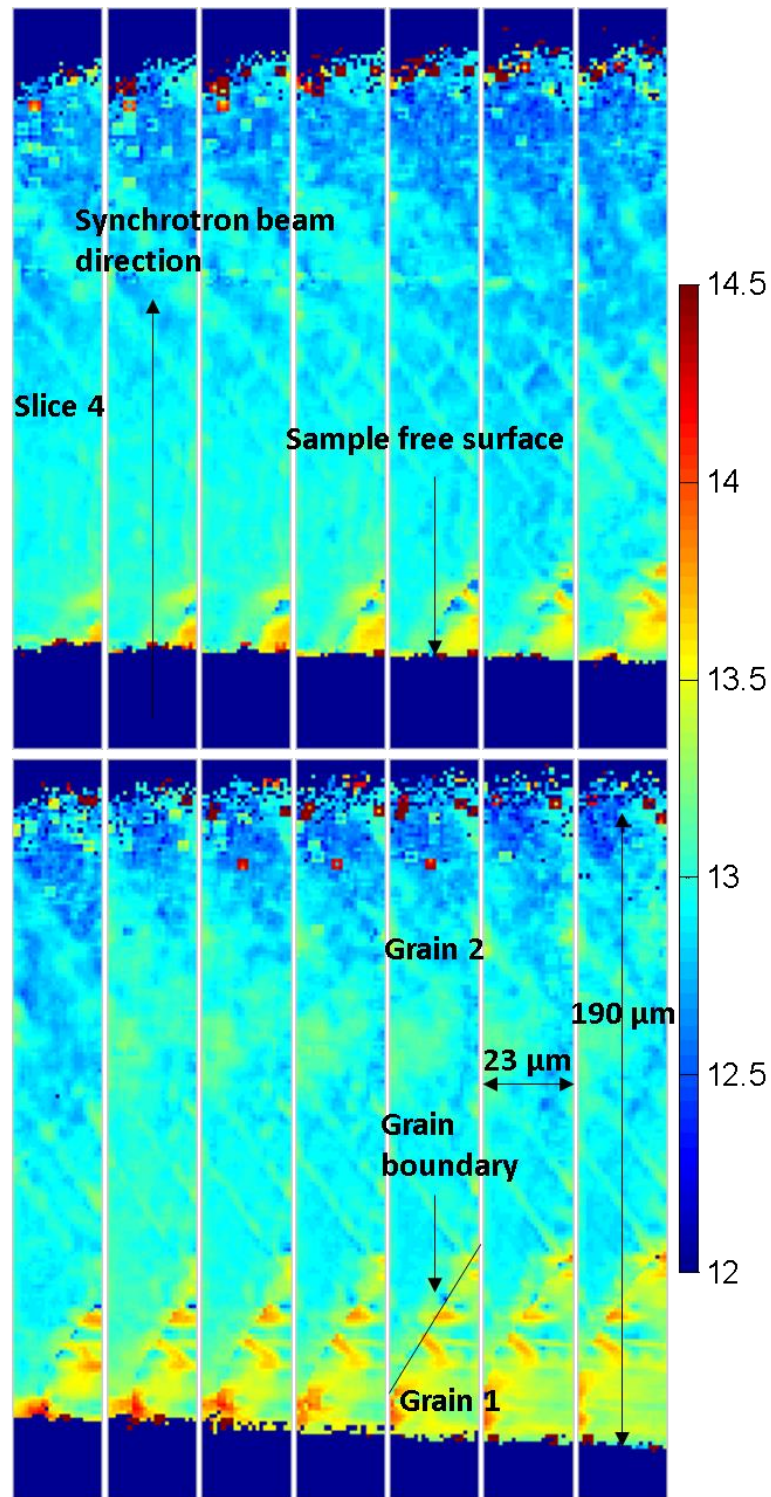
The $[1\bar{2}10]$ edge dislocation on the $(10\bar{1}0)$ prismatic slip plane and the $[1\bar{2}10]$ edge dislocation on the $(10\bar{1}1)$ pyramidal slip plane (Figure 5.3 (e) and (f) respectively) also have shown similar 'dislocation transferring' behaviour, with density at about $1.2 \times 10^{13} \text{ m}^{-2}$ and $8.77 \times 10^{12} \text{ m}^{-2}$ respectively. In terms of dislocation density, the 5 'transferred' cases have similar level, but the $[1\bar{2}10]$ edge dislocation (Figure 5.3 (e)) have shown more wide spread distribution with GND density both along grain boundary plane and extended into the bulk of grain 2, while the other 4 cases show dislocation distribution immediately next

to the point of intersection between slip band and grain boundary very similar to the stress distribution shown in Figure 4.3.

Apart from been 'transferred' the $[\bar{1}\bar{2}\ 10]$ and $[\bar{2}\ 110]$ type screw dislocation also have shown pile-ups in grain 1 with negative Burger's vectors. The densities are $\sim 2.6 \times 10^{13}$ for the $[\bar{1}\bar{2}\ 10]$ type and $\sim 1.0 \times 10^{13}$ for the $[\bar{2}\ 110]$ type at a generally higher density than for the dislocations with neighbouring grain.

For the $[\bar{1}\bar{2}\ 10]$ edge dislocation on basal plane (Figure 5.3 (d)), $[\bar{2}\ 110]$ edge dislocation on basal plane (Figure 5.3 (e)), and $[\bar{1}\bar{1}\bar{2}\ \bar{3}]$ edge dislocation on $(\bar{1}\bar{1}\ 01)$ pyramidal plane, dislocations were mainly blocked within the deforming grain (grain 1), little dislocation transfer could be observed in grain 2. The $[\bar{1}\bar{2}\ 10]$ edge dislocation on basal plane have dislocation density at $\sim 1.7 \times 10^{13}$, the $[\bar{2}\ 110]$ edge dislocation on basal plane at $\sim 1.0 \times 10^{13}$, and $[\bar{1}\bar{1}\bar{2}\ \bar{3}]$ edge ($\langle c+a \rangle$ type) on $(\bar{1}\bar{1}\ 01)$ pyramidal plane at $\sim 4.5 \times 10^{12}$.

The absolute density of all 33 dislocation types were add together to give the total GND density, the results of which are shown in Figure 5.4. Images in Figure 5.4 starts from slice 4 of the totally 31 slices of images since the first 3 slices show no GND feature as they do not contain information on grain boundary. Also, different to Figure 5.3, these total GND maps were not cropped and so show the variations in the whole volume probed in the experiment.



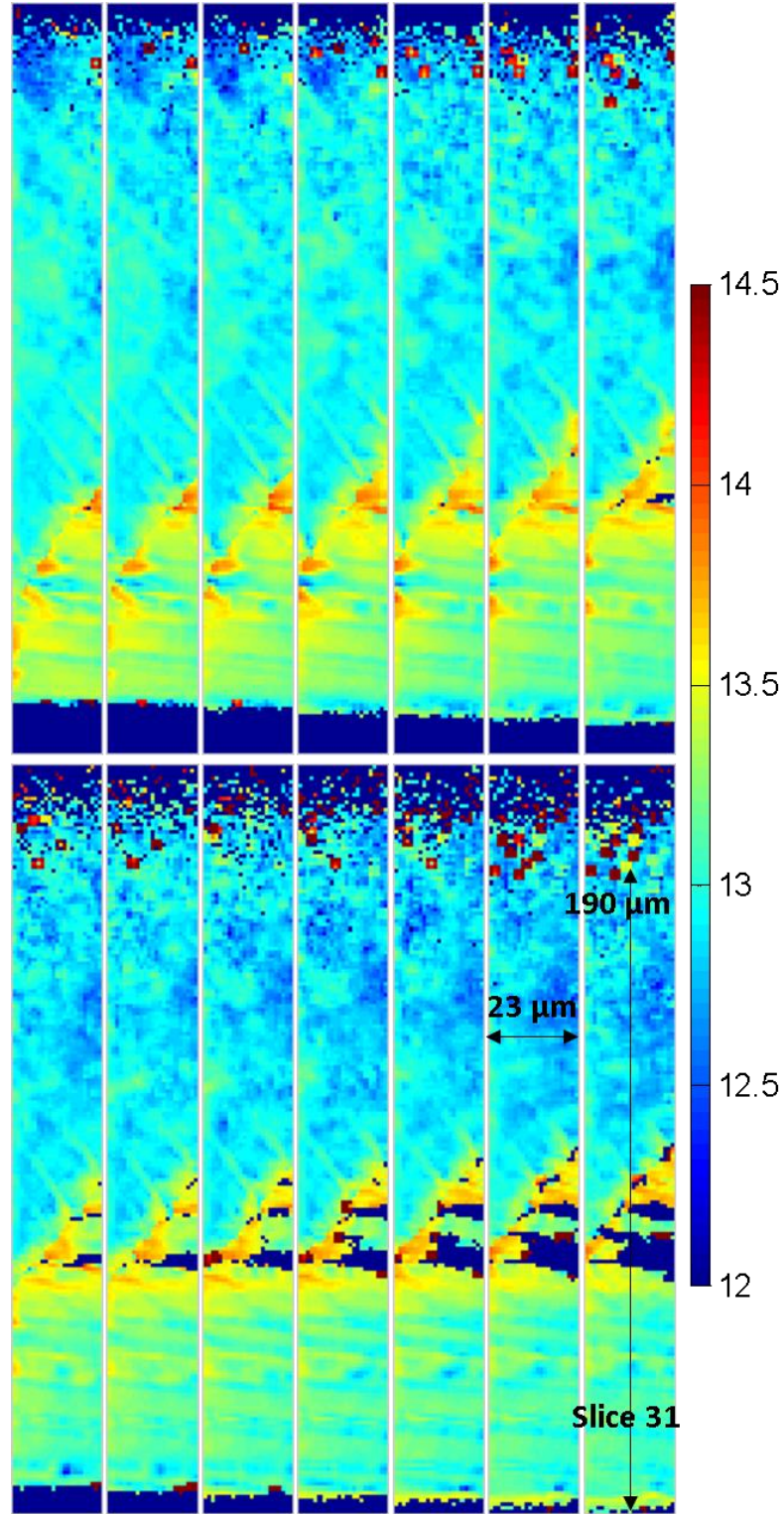


Figure 5.4: GND density of all dislocation types assumed. Images show variation of GND density around grain boundary from slice 4 to slice 31 (totally 31 slices). The numbers on the scale bar are on $\log_{10}$ scale.

Slice 1 corresponds to the topmost slice of the probed volume and slice 31 corresponds to the bottom slice of the volume (see Figure 4.1). The grain boundary position can be revealed by the GND density contrast and unindexed points in some images and is indicated on one of the images by a solid line. The position of the grain boundary changes on each slice as a result of the grain boundary inclination beneath sample surface.

As can be seen from Figure 5.4, the interior of grain 2 generally contains lower level of GND density at $\sim 12 \times 10^{12} / \text{m}^2$ than grain 1. Highest GND density is mainly distributed along the grain boundary plane with small amount been transferred into grain 2 and majority blocked in grain 1. The GND 'hot spots' span about $5 \mu\text{m}$ away from the grain boundary and are located at the places where slip band intersects grain boundary. Lower level diffuse GND density can also be observed between these hot spots. The large patches of vacant data points seen on the images of the last few slices are the place where diffraction patterns could not be indexed.

A comparison between Figure 5.3 and Figure 5.4 reveals that the GND density in grain 2 near grain boundary are due to $\langle a \rangle$ screw, $\langle a \rangle$ edge on prismatic plane, and $\langle a \rangle$ edge on pyramidal plane. While the GND density in grain 1 near grain boundary plane are due to $\langle a \rangle$ screw, $\langle a \rangle$ edge on basal plane, and $\langle c+a \rangle$ edge on pyramidal plane.

The 3D volume rendering technique as applied to the stress maps shown in Figure 4.5 can also be applied to the GND slices in Figure 5.4 to visualize the distribution of GND in 3D. The result is shown in Figure 5.5.

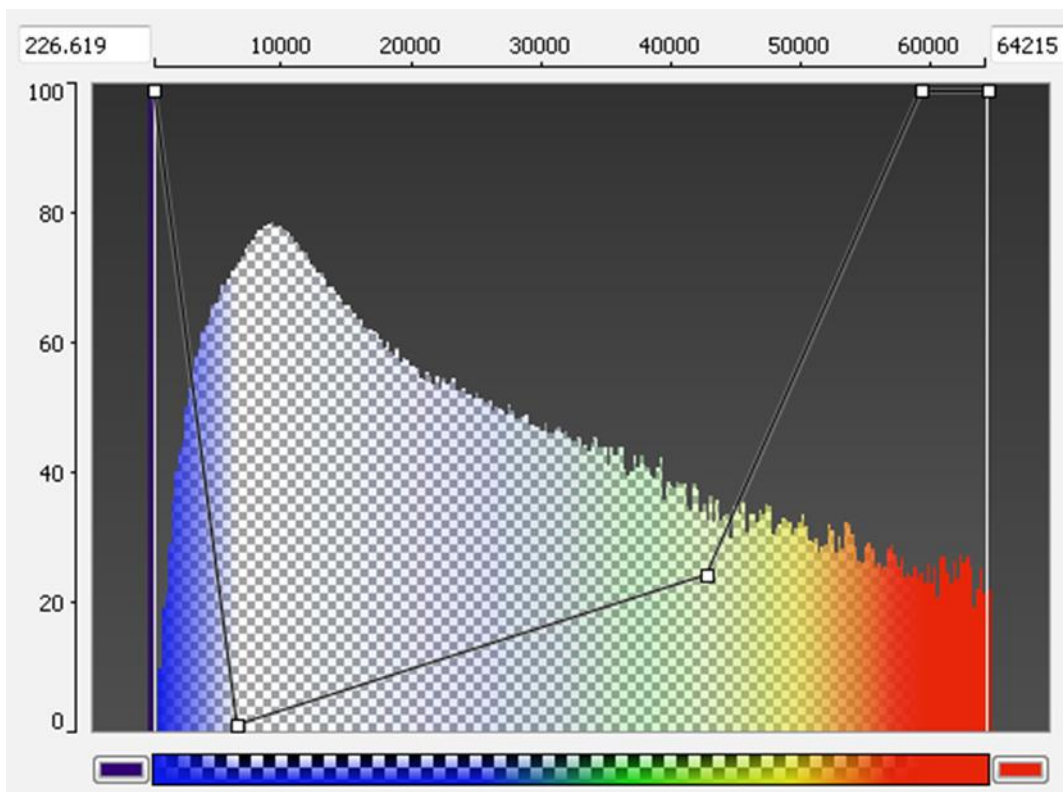
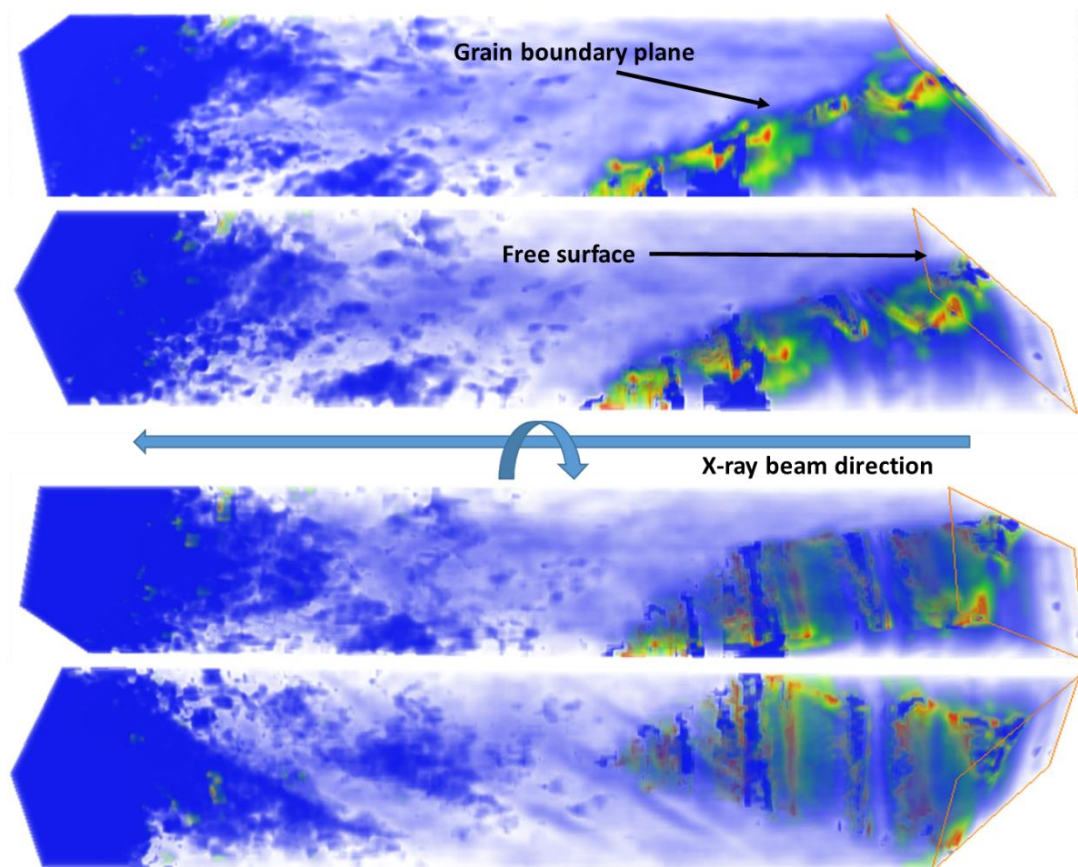


Figure 5.5: Visualization of the distribution of GND densities in 3D. Four images are viewed from different perspectives indicated by the curved arrow. The orange box to the right of each image indicate where the free surface is. Bottom of figure is the opacity histogram associated with the volume rendering.

It can be seen that the place of GND concentration corresponds to the place of stress concentration in Figure 4.5. However, the distribution of GND density is more diffuse compared with stress distribution. Moreover, GND density seems to be spread along the grain boundary plane.

5.3 2D GND density from HR-EBSD analysis

The density of GND as a result of the same slip band and grain boundary interaction described in Section 5.2 was also calculated in the X_{ebad} and Y_{ebad} reference frame (Figure 4.1) using HR-EBSD. The experimental details were described in Chapter 3.1. The step size used here is 0.2 μm which is significantly smaller than the 1 μm step size used for DAXM. A histogram on the average GND density around the slip band/grain boundary interaction for all 33 types of dislocations is shown in Figure 5.6.

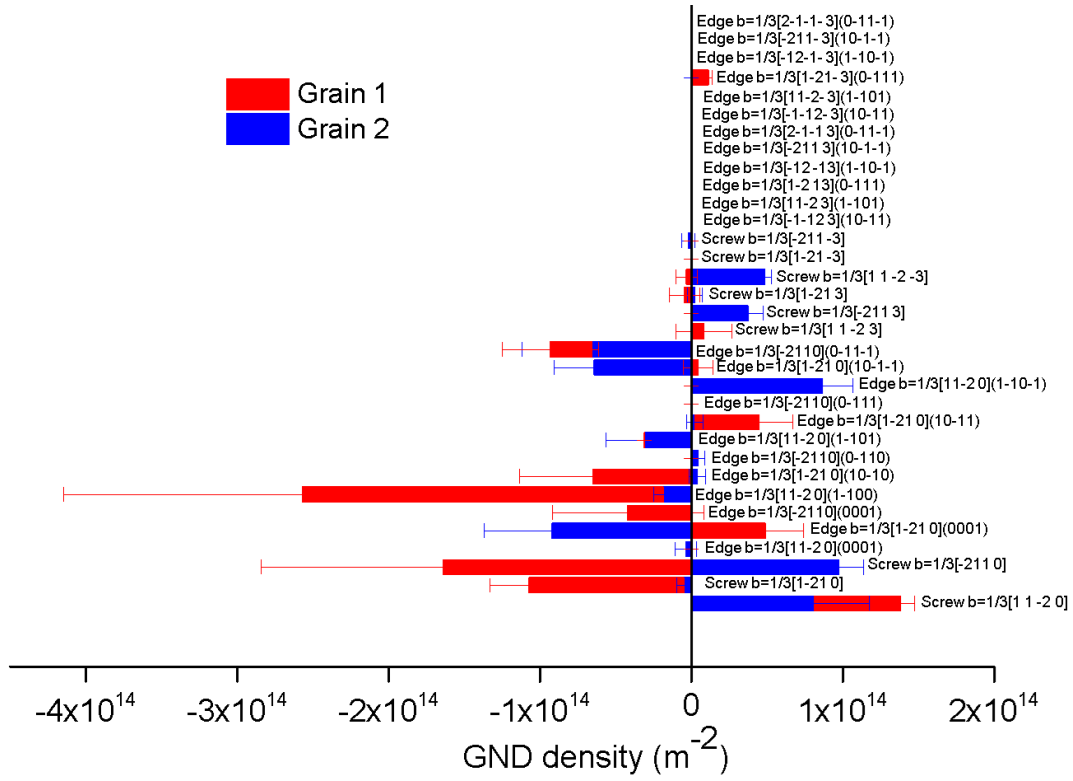


Figure 5.6: Average GND density for all 33 dislocation types in titanium at slip band and grain boundary intersection, measured from the DAXM experiment.

The Burger's vector types giving rise to appreciable lattice curvature were identified and the corresponding GND density maps are shown in Figure 5.7 (a) and the total GND density is shown in Figure 5.7 (b).

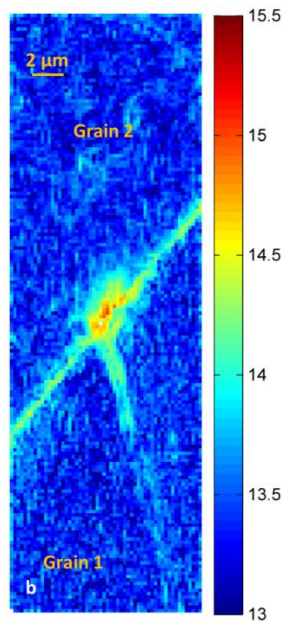
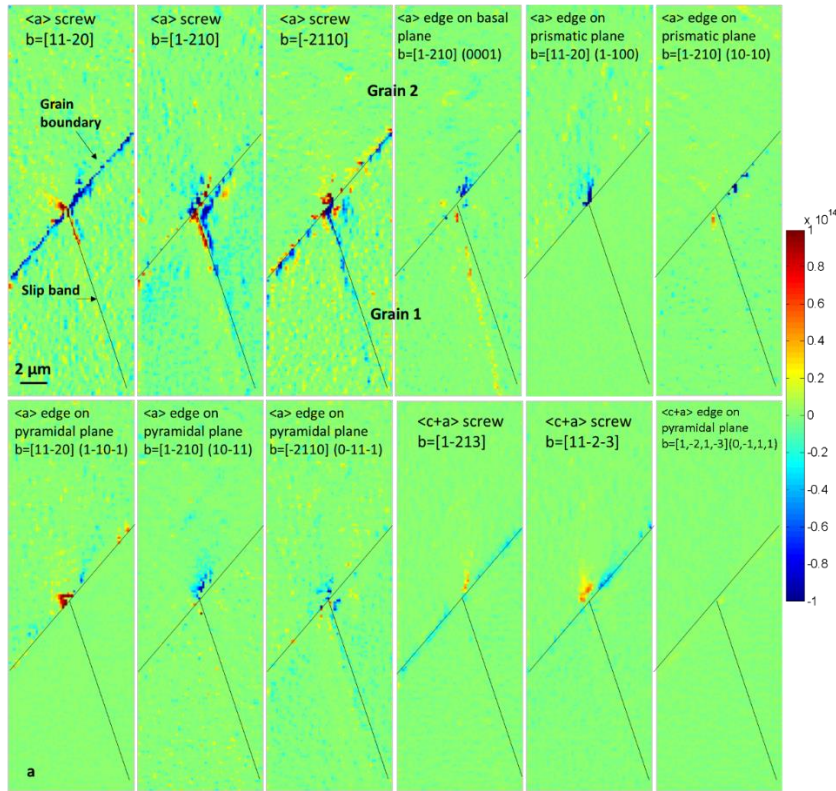


Figure 5.7: HR-EBSD maps showing GND distribution as a result of slip band and grain boundary interactions. (a) GND density maps for the type of dislocations giving rise to significant lattice curvature. (b) Total density of GND as a summation of all 33 types of dislocations used in the calculation.

It is evident from Figure 5.7 (a) that there are 12 types of dislocations supporting the captured lattice curvatures. Almost all dislocation types can be found 'transferred' to grain 2 immediately ahead of the blocked slip band in grain 1, except for $[1\bar{2} 10]$ edge dislocation on $(10\bar{1} 0)$ prismatic slip plane and $[1\bar{2} \bar{1}\bar{3}]$ edge on $(0\bar{1} 11)$ pyramidal slip plane, which only seem to concentrate in grain 1 around the vicinity of slip band and grain boundary intersection. Apart from the just mentioned two types of 'blocked' dislocations, all three $\langle a \rangle$ screw dislocation, the $\langle a \rangle$ edge dislocation on basal plane, as well as the $[\bar{2} 110]$ edge on $(0\bar{1} \bar{1}\bar{1})$ pyramidal plane were also found in grain 1 near the point of interaction. Very similar to what was revealed by the DAXM measurement (Section 5.2), the total GND density mapped from HR-EBSD experiment also show localized GND distribution in both grains but with the range of distribution in grain 1 (neighbour grain) much more extended than that in grain 2 (slipped grain).

5.4 Discussion

This chapter presented a comparison between the two state of the art electron (HR-EBSD) and X-ray (DAXM) based GND measurement techniques. There are other dislocation characterisation techniques available for various purposes such as transmission electron microscopy (TEM), which is excellent at capturing very local dislocation morphology, dissociation and reaction behaviour [203] but suffers from the limited sample volume that can be analysed and laborious sample preparations as well as the image stress issue

[204]. Traditional X-ray line profile analysis is relatively quick in demonstrating evolution of dislocation density linked with various processing history but doesn't provide spatially resolved point to point comparison and local distribution of dislocation densities [205]. Far-field 3D XRD technique [204, 205] offers high angular resolution ($\sim 0.004^\circ$) and good time resolution and therefore enables in-situ testing, but the spatial resolution ($\sim 5 \mu\text{m}$) is not sufficient to capture local intragranular deformation structures. Although not applicable for dynamic deformation studies, the DAXM method offers excellent spatial resolution ($> 0.5 \mu\text{m}$) and competitive angular resolution ($\sim 0.01^\circ$). The cross-correlation based EBSD technique (HR-EBSD) offers better spatial resolution ($> 0.05 \mu\text{m}$) similar strain sensitivity to DAXM (10^{-4}) and but is limited to 2D surface measurements. In terms of capturing the GND density distribution, both HR-EBSD and DAXM offer better sensitivity than Hough-based EBSD technique, which has angular resolution of 0.5° or worse [176].

A lower bound GND noise level for each of HR-EBSD, DAXM, and Hough-based EBSD can be estimated using:

$$\Delta\rho = \frac{\delta}{b\lambda} \quad (5.7)$$

Where δ is angular resolution (in radian), b is Burger's vector magnitude, and λ is the step size. Using $b=0.295 \text{ nm}$ and assume step size of $0.2 \mu\text{m}$, the GND noise level can be estimated to be $\approx 2 \times 10^{12} \text{ m}^{-2}$ for HR-EBSD, $\approx 3 \times 10^{12} \text{ m}^{-2}$ for DAXM, and $\approx 2 \times 10^{14} \text{ m}^{-2}$ for Hough. It is then obvious that both HR-EBSD and DAXM captures GND density with significantly lower noise level than the Hough-based EBSD technique and therefore are able to reveal GND

distribution even at low level of plastic deformation. In this research, the step size used for HR-EBSD was indeed 0.2 μm , but the one used for DAXM was 1 μm , this brings the noise level of DAXM experiment down to $6 \times 10^{11} \text{ m}^{-2}$. With the increased step size in DAXM, the captured GND density was on average an order of magnitude lower than the HR-EBSD measurement. This effect can be explained by the size of Burger's circuit captured by the experimental step size. As the Burger's circuit becomes bigger, a larger fraction of the dislocation densities is counted as statistically stored dislocations (SSD) as their short range rotation gradient cancel out with each other (i.e. more dipoles in larger Burger's circuit) [174, 206], while as Burger's circuit was sampled smaller, more dislocations can be counted as GND. The effect of step size on experimentally measured GND density were well explained by Jiang et al [207]. The average GND density near the grain boundary investigated in detail here was estimated to be $5.3 \times 10^{13} \text{ m}^{-2}$ by DAXM over step size of 1 μm and $2.2 \times 10^{14} \text{ m}^{-2}$ for HR-EBSD over step size of 0.2 μm . The effect of step size on the ability to differentiate GND from SSD is consistent with previous report [207]. A factor of 4 decrease in the measured GND density over a factor of 5 decrease in step size is dramatic compared with GND measured in copper [207], which have demonstrated a decrease of density from 10^{14} to $3 \times 10^3 \text{ m}^{-2}$ (a factor of 5) over step size change from 0.5 to 10 μm (a factor of 20). The noise in GND measurement is unlikely to be associated with this observation as the average captured GND density for both measurements are well above the noise level assessments made above. Therefore, other factors should be responsible for this sharp GND density decrease. One possible factor could be the size of the

sampled region, the GND density in the copper case were averaged over large population of grains while in this study only a portion of a single grain boundary was compared. Another possible factor is that there are more grain to grain variation due to anisotropy in the HCP titanium than BCC copper. It is likely that larger strain gradients between hard-soft grain neighbours in this case results in a less uniform distribution of dislocations and therefore a magnified step size effect. The fact that totally 8 dislocation types were found to support the measured lattice curvature in DAXM measurement (Figure 5.3) while 12 dislocation types were found in HR-EBSD measurement (Figure 5.7 (a)) is also possibly related to the effect of step size, as the greater spatial detail revealed by HR-EBSD requires more dislocation types to represent it.

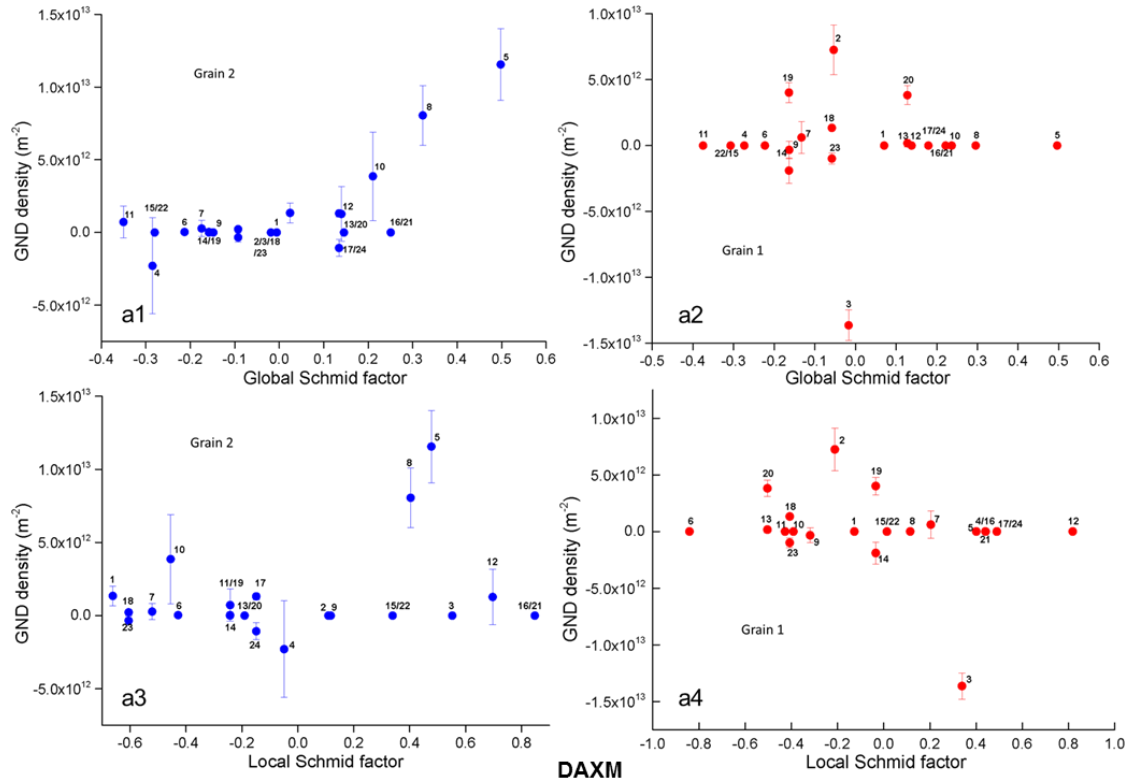
The distribution of dislocation density has been shown to be heterogeneous from both DAXM and HR-EBSD measurements. In both cases, GNDs were found on both side of the grain boundary but mainly confined in the vicinity of the location where slip bands intersect the grain boundary. Apart from the localized GND density near grain boundary, dislocation cell structures within the interior of grain 2 were also revealed by DAXM as is shown in Figure 5.4. The HR-EBSD revealed a smaller area focused on the lead of the blocked slip band and did not shown the cell structure away from the grain boundary.

The angle between Burgers' vector of the active slip system (green plane of the HCP crystal in grain 1) with the identified Burger's vector for each map for the specific GND types were calculated and given as θ_B with the crystal orientation schematics in Figure 5.3. Higher angle indicates a higher magnitude of residual Burger's vector if slip with the indicated Burger's vector is to be

transferred into grain 2. It can be seen that there is poor correlation between the magnitude of residual Burger's vector and whether dislocation can be found in grain 2 or the magnitude of dislocation 'transferred' to grain 2. It is therefore evident that residual Burger's vector might not be a sufficient indicator of GND transfer although previously it has been taken as an important indicator for slip transfer [117, 123, 124, 197, 208]. The observation that the total GND density is partly transferred (or independently nucleated) in grain 2 and partly reflected back into grain 1 from the point of slip band/grain boundary intersection could be accounted for by Li's theory [84], in which it was proposed that grain boundary ledges serve as dislocation sources that emit dislocations into adjacent grains. In case of no slip transfer, the out of grain boundary plane component of the leading dislocation in the pile-up plane could lead to ledge formation in the grain boundary plane, this effect together with Li's theory explain why GND is found at both side of the boundary and mainly concentrated near the point slip band and grain boundary intersects.

Apart from the very local high density GND concentration, the diffuse density along grain boundary are also evident from both DAXM and HR-EBSD experiments, as can be seen from Figure 5.3, Figure 5.4, and Figure 5.7. The spreading of dislocation along the vicinity of grain boundary plane reflect the general deformation compatibility requirement at the interface of two differently oriented anisotropic crystals [27] as well as, more generally, the grain boundary (or phase boundary) being a source for deformation heterogeneity compared with grain interior [87, 207].

The average GND density at the neighbourhood of slip band grain boundary interactions in grain 1 and grain 2 from both DAXM and HR-EBSD are plotted against Schmid factor each slip system were experiencing, utilizing both far field external loading (Global Schmid factor) and local stress tensor (Local Schmid factor) measured from DAXM and HR-EBSD technique. The results are shown in Figure 5.8. For local Schmid factor calculation, please refer to equation 6.2 in Chapter 6.2.



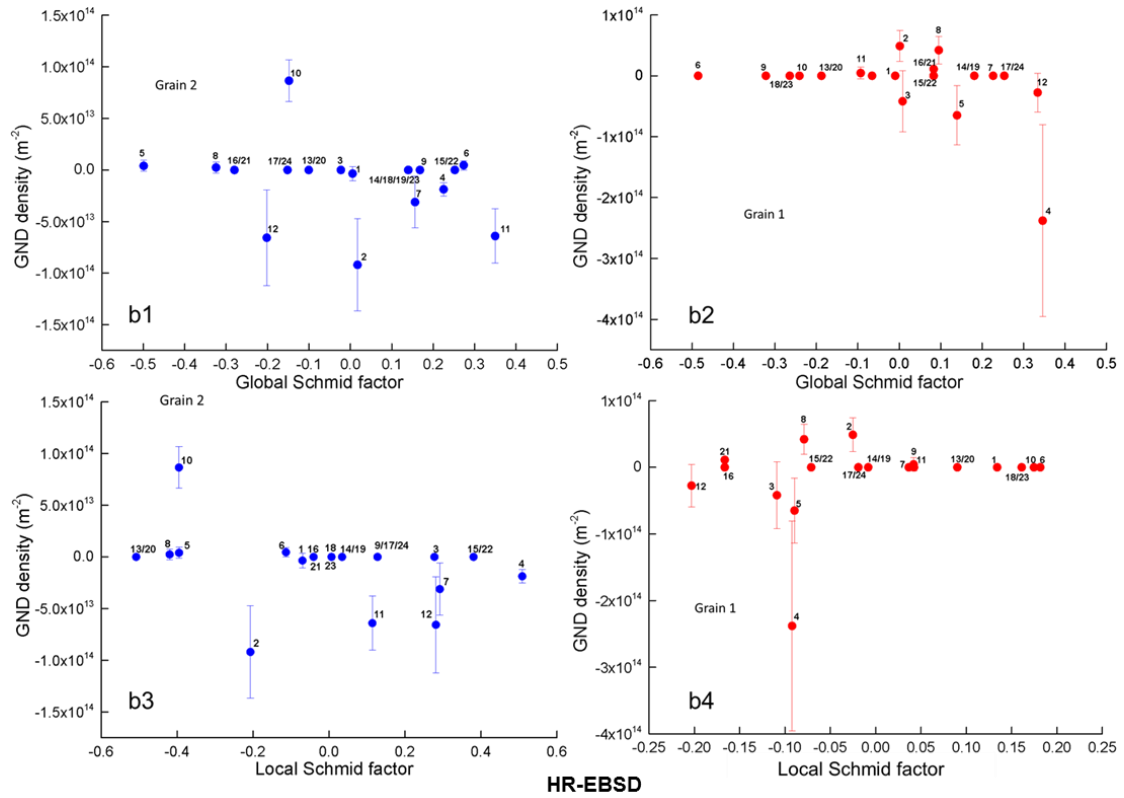
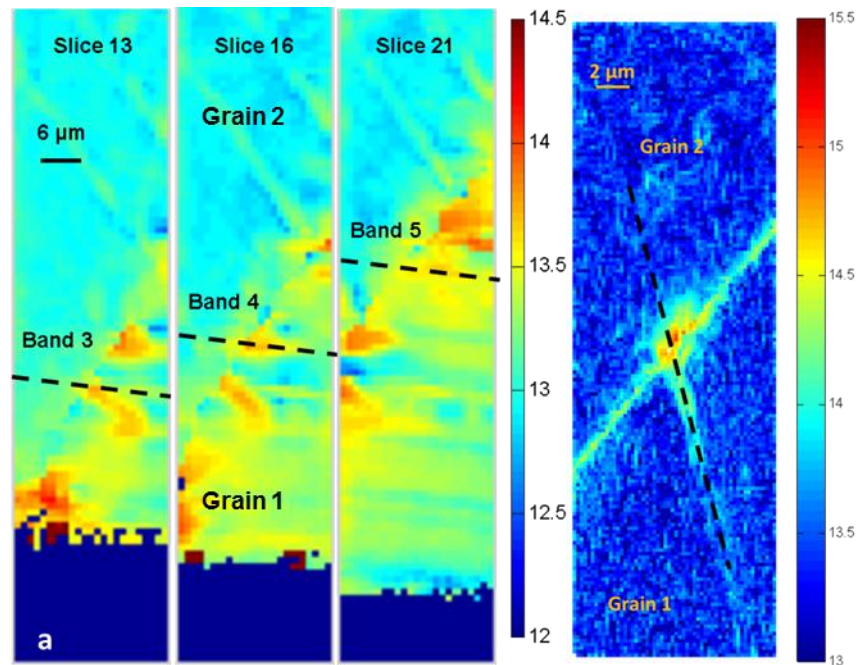


Figure 5.8: GND density versus Schmid factor for: 1-3 prismatic slip systems, 4-6 prismatic slip systems, 7-12 $\langle a \rangle$ pyramidal slip systems. And 13-24 $\langle c+a \rangle$ pyramidal slip systems. The image set (a) is from the DAXM measurement and the image set (b) is from the HR-EBSD measurement.

Figure 5.8 demonstrates no meaningful correlation between the GND density of different type of dislocation and the resolved shear stress the corresponding slip system were experiencing. The reason may be that the GND density calculated from both DAXM experiment and HR-EBSD experiment were based upon minimizing the total line energy of dislocations and the constitutive behaviour of material was not involved. Care must be taken interpreting data in Figure 5.8 as there were large uncertainties involved in distinguishing the position of the grain boundary, the position of the incoming slip band, and

therefore the position of the GND hotspot. These consequently leads to large uncertainty in determining the average GND density.

The distribution of GND density away from the grain boundary into the interior of both grains along the direction of slip bands 3, 4, and 5 (same as indicated in Figure 4.5) as indicated by the black dashed line in Figure 5.9 (a) are plotted in Figure 5.9 (b). The equivalent sample surface band and dislocation density profile measured by HR-EBSD are also shown.



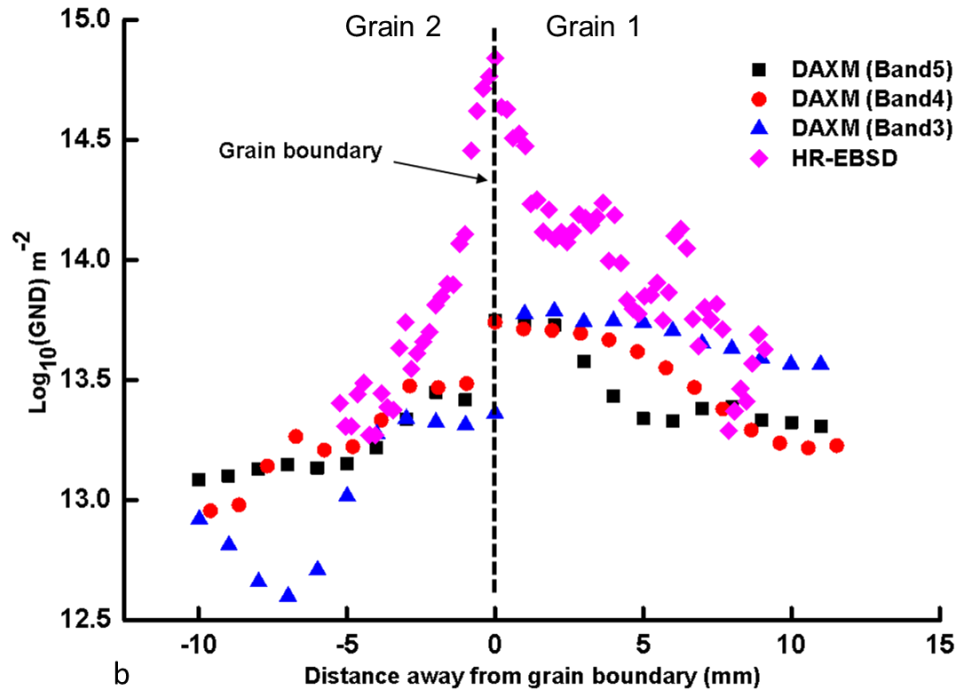


Figure 5.9: (a) Slices where the indicated slip band intersects the grain boundary in the middle of the image. Black dashed line indicates the direction of the slip band and along which the GND density profiles were extracted. The fourth image is the measurement made using HR-EBSD on sample surface. (b) The GND line profile along the dashed line indicated in (a).

Both DAXM and HR-EBSD have shown (Figure 5.9 (b)) that the higher GND density is near grain boundary and decay rapidly over 5 μm distance away from grain boundary before reaching a relatively stable level. This observation is consistent with the large population GND density study made in copper with HR-EBSD [207]. HR-EBSD result in Figure 5.9 (b) have shown a much higher (an order of magnitude) GND density in a much more local scale ($\sim 2 \mu\text{m}$) around the grain boundary compared to the DAXM measurement. For the HR-EBSD data the GND density is observably higher in grain 2 than in grain 1 in

the peak very near the boundary. However for the DAXM measurements the opposite can be observed (Figure 5.9 (a)). Care must be taken with the scale bar in interpreting the maps from the two different measurements. The highest GND densities from HR-EBSD measurement is largely confined within the first 2 μm from the grain boundary which in DAXM was effectively averaged out due to a much coarser spatial resolution. This is one reason the observed density in grain 2 is much lower than in grain 1 from DAXM measurement as also evident in Figure 5.5 (b). It is clear that the HR-EBSD technique provides more information in a much local scale. The rate of decay of the measured GND density away from the grain boundary is higher from HR-EBSD than from DAXM. This is likely to be linked with the ability of DAXM to measure lattice curvature in all three direction while the curvature associated with the direction into sample surface was not sampled by HR-EBSD. The effect of sampling lattice curvatures in all three dimensions is likely to result in a wider spread of high density GNDs and slower decay over distance away from the grain boundary as is evident from Figure 5.5. Of course HR-EBSD can also sample rotation gradient in full dimension by coupling with serial sectioning technique, but this hasn't been done yet (existing literatures calculate 3D GND by Hough based technique, for example [209]). Alignment of data slice to sufficient accuracy to make use of the HR-EBSD method to measure slice to slice variation in lattice rotation is unlikely to be achieved in the near future. It should be noted that the GND density calculated from both DAXM and HR-EBSD are naturally a lower bound estimation as only 6 lattice curvatures in HR-EBSD and 9 in DAXM were used to solve 33 types of dislocations in titanium.

5.5 Conclusion

In this Chapter, the GND density measurement capability between two high fidelity deformation gradient characterisation techniques, HR-EBSD and DAXM, were compared by applying both techniques to a deformed CP titanium sample.

The following conclusion can be drawn:

1. Both techniques are able to capture the heterogeneous distribution of GND density and broadly converge well with each other.
2. HR-EBSD offers better spatial resolution and therefore provides more information at smaller scale.
3. DAXM offers less information at very local scale but captures better the range of GND density distributions.
4. The effect of step size is a more determining factor over lattice curvature in the captured magnitude of GND density.

Chapter 6 Stress and GND distribution related with twin lattice and twin grain boundary interactions

Deformation twinning is a very important and frequently occurring deformation mechanism for HCP metals. Twin formation produce large shear within the crystal lattice [128] and the interaction of deformation twinning with grain boundaries is suspected to lead to damage nucleation [135]. Although a large body of research has successfully addressed the crystallography [11, 128], morphology [131], and certain aspects of macro mechanical behaviour [106, 135, 144], the interaction between grain boundary, matrix defects and deformation twins as well as local strain accommodation between matrix and twin remain poorly understood [9, 210].

This chapter contributes to the solution of the above mentioned problems by probing the local stress concentration and GND distribution near the tip of twins in the following situations: twins fully imbedded in the interior of a grain far away from any grain boundary, twins that have propagated across the grain interior and have tips near grain boundaries, and at the place where twins intersect with grain boundaries. The characterisation technique used was HR-EBSD and these results shed light on how matrix and neighbouring grains accommodate twin shear, and how the stress field caused by twin- grain boundary interactions affect the neighbouring grains.

6.1 Materials and experimental methods

The material used here has the same chemical composition as in Chapter 3. The dimension of the tensile specimen used here was the same as discussed in Chapter 3.1, but the sample thickness was about 500 μm compared with the 1 mm thick sample used for Chapter 3. Sample preparation followed the same route as mentioned in Chapter 3.1. Tensile testing was also conducted at the same displacement rate of 1 $\mu\text{m/s}$ to the same strain level of 1%. It was found that even under the same deformation condition, in this case the sample surface was covered by large population of twins while little twinning activity had been observed in the previous case. This is most probably due to the texture of the sample as both samples were cut from different positions of a rolled bar, the texture of which varies from centre to edge. It is also possibly due to the thickness of the sample as thinner samples have less constraints for accommodating twin shear.

Various regions of interest featuring twin tip in the interior of a grain, twin tip near grain boundary, and twin tip intersecting with grain boundary were scanned using a Zeiss Merlin FEG-SEM system and Bruker e-flash high resolution (1600 x 1200 pixels) EBSD detector coupled with fore-scatter detector below the phosphor screen. Working distance was set to 18 mm and scanning was performed using 14 nA beam current under 20 KV excitation voltage.

6.2 Twin trace analysis and finding a Cartesian reference frame on twin system

The active twin variant that coincides with the one seen on a SEM image can be found in several ways. One of the methods is to study the trace of all twin variant of an identified twin type as was used in reference [210]. This procedure is demonstrated below:

A compression twin was found in SEM as shown in Figure 6.1. All variants associated with this twin type can be identified and transformed into Cartesian reference frame following the method demonstrated in Chapter 3.2. These twin variants are then rotated into the global coordinate system of the matrix using the Euler angles obtained from EBSD measurement. The trace of these twin variants can then be plotted and the one that converges with the experimentally observed twin trace can be identified as shown in Figure 6.1 (a).

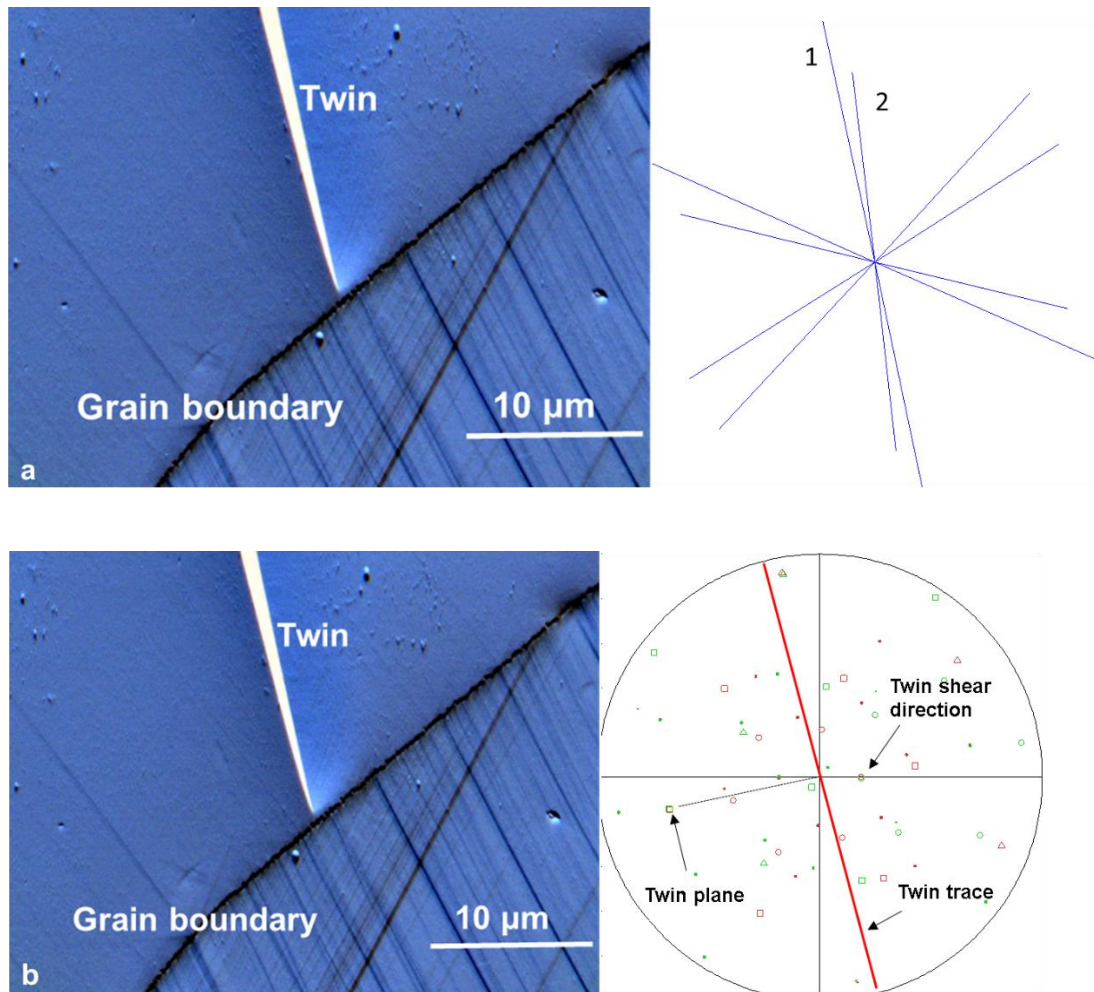


Figure 6.1 (a): Twin trace analysis by plotting traces of all possible twin variants. (b): Twin trace analysis by plotting pole figure.

As can be seen in Figure 6.1 (a), sometimes this method does not work unambiguously as multiple twin variant may have similar surface traces as identified by number 1 and 2 in the trace map.

Another method is to plot pole figures for all variants of the identified twin system in both twin orientation and matrix orientation, the overlapping poles should be the active twin variant. As the twin trace in real space should be perpendicular to both sample plane normal and twin plane normal, therefore the

trace on pole figure can be revealed by the line perpendicular to the line linking the overlapped twin plane normal and the pole for sample normal (central point of the pole figure), this is shown in Figure 6.1 (b).

The pole figure plotting was done using in-house developed Matlab code (copyright to Dr. Ben Britton, Imperial College London). As can be seen both twin plane and twin direction can be identified as well as the twin trace (red line) that converges well with experimental observations. The problem with the current analysis is that the overlapping twin plane on the pole figure are from two different variants of the same twin family. This problem is demonstrated below:

The active twin type can be identified using c axis angle between the parent and the twin [136], after which all six variants can be identified using crystallography. For example, Figure 6.2 demonstrates a compression twin:

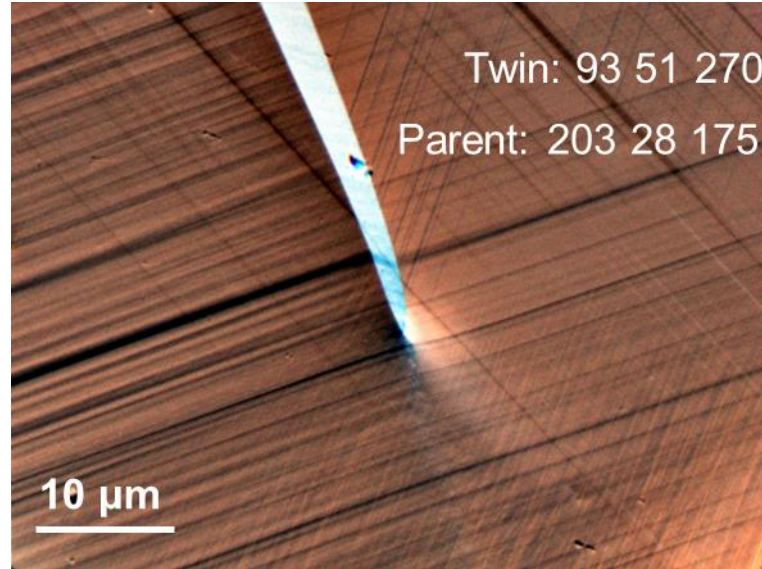


Figure 6.2: A compression twin within a grain of cp-titanium deformed to 1% tensile strain. Bunge convention Euler angles as measured from the experiment (Φ_1 Φ_2 Φ_3 , degree) for the twin and the parent are also labelled.

A rotation matrix can be constructed from the Euler angles measured from EBSD using the following equation:

$$g = \begin{pmatrix} \cos\phi_1\cos\phi_2 - \cos\phi\sin\phi_1\sin\phi_2 & \sin\phi_1\cos\phi_2 + \cos\phi\cos\phi_1\sin\phi_2 & \sin\phi\sin\phi_2 \\ -\cos\phi_1\sin\phi_2 - \cos\phi\sin\phi_1\cos\phi_2 & \cos\phi_1\cos\phi\cos\phi_2 - \sin\phi_1\sin\phi_2 & \sin\phi\cos\phi_2 \\ \cos\phi_1\sin\phi & -\cos\phi_1\cos\phi & \cos\phi \end{pmatrix} \quad (6.1)$$

The six twin variants can then be rotated into parent and twin orientation using equation 6.1 and the Euler angles, the results of which are shown in Table 6.1.

		crystal reference	rotate to parent orientation	rotate to twin orientation
Variant 1	Shear plane	(1 1 -2 2)	(0.47 0.83 0.30)	(0.85 0.48 -0.22)
	Shear direction	[1 1 -2 -3]	[0.52 0.01 -0.85]	[-0.37 0.24 -0.90]
Variant 2	Shear plane	(2 -1 -1 2)	(0.70 0.13 0.70)	(0.90 -0.37 -0.22)
	Shear direction	[2 -1 -1 -3]	[0.67 -0.44 -0.60]	[-0.34 -0.29 -0.89]
Variant 3	Shear plane	(1 -2 1 2)	(0.13 -0.47 0.87)	(0.46 -0.82 0.34)
	Shear direction	[1 -2 1 -3]	[0.31 -0.81 -0.49]	[-0.61 -0.58 -0.54]
Variant 4	Shear plane	(-1 -1 2 2)	(-0.67 -0.37 0.64)	(-0.03 -0.42 0.91)
	Shear direction	[-1 -1 2 -3]	[-0.20 -0.75 -0.63]	[-0.93 -0.33 -0.18]
Variant 5	Shear plane	(-2 1 1 2)	(-0.91 0.34 0.24)	(-0.09 0.42 0.90)
	Shear direction	[-2 1 1 -3]	[-0.35 -0.30 -0.89]	[-0.96 0.20 -0.19]
Variant 6	Shear plane	(-1 2 -1 2)	(-0.34 0.94 0.07)	(0.35 0.87 0.34)
	Shear direction	[-1 2 -1 -3]	[0.01 0.08 -0.99]	[-0.68 0.49 -0.54]

Table 6.1: Six variant of a compression twin type with twin shear plane and shear direction expressed in crystal reference, parent orientation and twin orientation.

Theoretically, the twin variant that demonstrates the same indices after it has been rotated to both parent and twin orientation is the active twin variant. However, in current situation, parent and twin orientation seem to indicate two different variants. In another word, the overlapping twin shear directions and planes on the pole figure (Figure 6.1 (b)), which should come from one variant, came from two different variants. This problem persists to all such measurements conducted in the Bruker EBSD analysis system and is likely to be wrong indexing of the diffraction pattern from the twin¹.

In this Chapter, as the analysis was based on stress state in the parent grain and its effect on the matrix surrounding the twin lath, the variant identified by the parent was taken as the activated variant. After variant identification, a

¹ Discussions with Bruker to understand the exact meaning of the Euler angles their software provides is still on-going.

coordinate system could be constructed on twin system following the convention that the X direction is pointing to the twin shear direction, the Z direction is the twin plane normal, and the Y direction is the cross product between the two vectors ($Z \times X$). Stress tensors mapped on sample surface were rotated onto this local twin coordinate system to reveal the stress caused by twin shear.

To reveal the effect of local stress environment on the identified twin variant, local Schmid factors (LSF) were calculated using equation:

$$LSF = \frac{\sigma:S}{\|\sigma\|} \quad (6.2)$$

Where σ is the stress tensor mapped on global coordinates (sample surface) measured by HR-EBSD, S is the tensor product of shear direction, d , and shear plane normal, n , which takes the following form:

$$S = \begin{pmatrix} n_1 d_1 & n_1 d_2 & n_1 d_3 \\ n_2 d_1 & n_2 d_2 & n_2 d_3 \\ n_3 d_1 & n_3 d_2 & n_3 d_3 \end{pmatrix} \quad (6.3)$$

The tensor S was populated for each interrogation point of the EBSD map using the local Euler angles and the identified twin shear direction and twin plane normal. The $\|\sigma\|$ term is the highest normal stress component of the “diagonalised” stress tensor. For a symmetrical matrix such as stress tensor, it is also equal to the square root of the maximum Eigen value of the product between the stress tensor and its conjugate transpose. The calculated LSF takes into account both local stress tensor variations and orientation variations.

As a comparison, the global Schmid factor (GSF) was also calculated using:

$$\text{GSF} = \frac{\text{Loading direction} \cdot \text{shear direction}}{\text{norm}(\text{Loading direction} \cdot \text{shear direction})} * \frac{\text{Loading direction} \cdot \text{shear plane normal}}{\text{norm}(\text{Loading direction} \cdot \text{shear plane normal})}$$

(6.4)

6.3 Stress field associated with twin imbedded within a grain and far from grain boundary

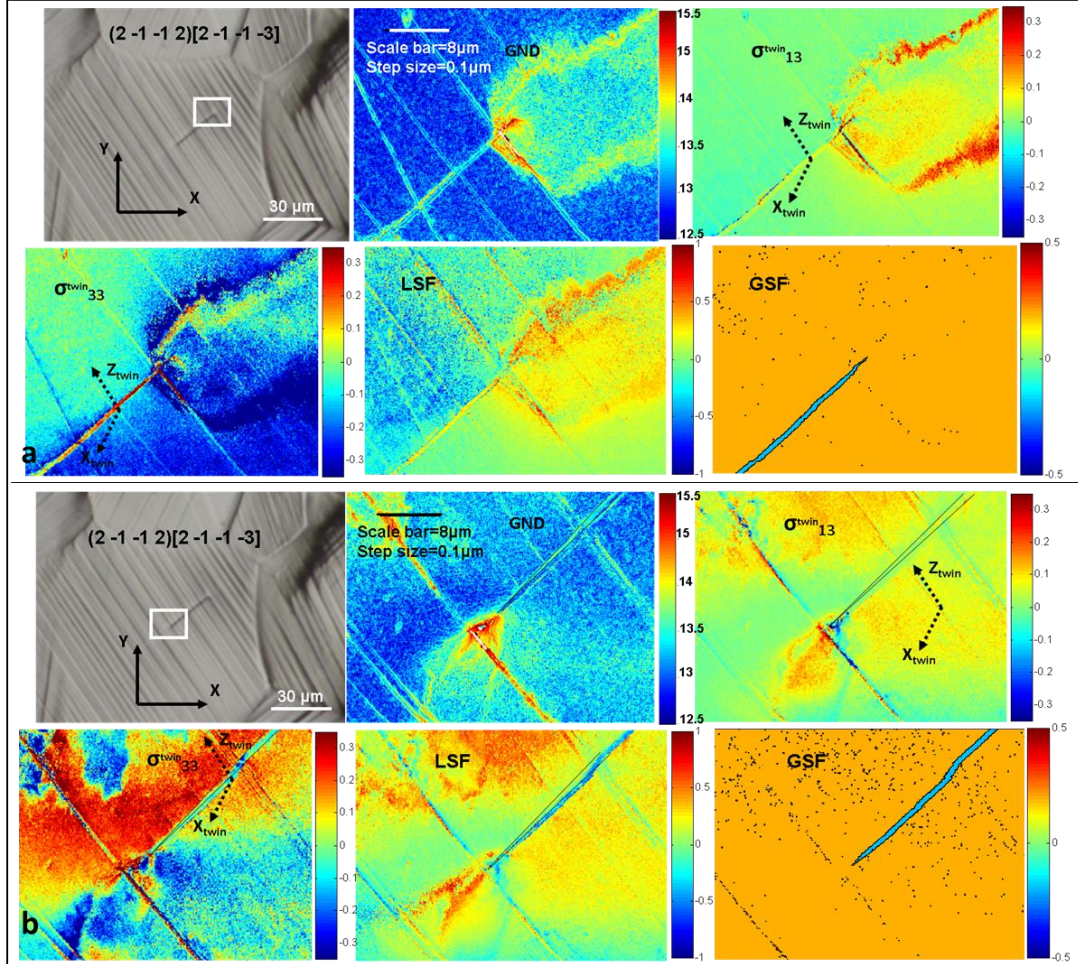


Figure 6.3: Twin imbedded in the interior of a large grain. Images in (a) and (b), from top left to bottom right, are optical image, GND density map, σ_{13}^{twin} stress map, σ_{33}^{twin} stress map, local Schmid factor map (LSF, calculated using stress tensor obtained from HR-EBSD analysis as shown in eq.6.2), and global Schmid factor (GSF, calculated using external loading direction and twin plane orientation as shown in eq.6.4) map. Stress tensor components are those resolved onto twinning plane. 2D projection of the local coordinate system on the twin variant is indicated by X_{twin} (twin shear direction) and Z_{twin} (twin plane normal). The X and Y on first image indicate the global coordinate

system. GND density scale bar is in $\log_{10}$ scale with unit m^{-2} . Stress map scale bar unit is GPa.

Figure 6.3 demonstrates that from the surface observation, the twin is about 30 μm long and 2 μm wide and is fully imbedded within a grain and far away from the grain boundary. The slip bands continue on either side of the twin suggesting they were present before the existence of the twin. Twin trace analysis found that this twin is of compression type with twinning plane $(\bar{2} 1 1 2)$ and shear direction $[\bar{2} 1 1 \bar{3}]$. The trace analysis also revealed that while the twin plane normal is always perpendicular to the twin lath long axis, the projection of the shear direction in the sample surface plane is generally not parallel to the long axis direction unless in some special cases (this can also be seen for other figures regarding twinning in this chapter). The high GND density is mainly concentrated around the tip of the twin and tends to radiate out along the twin lath direction. Raised GND density can also be observed along the slip band intersecting the twin tip, revealing possible local interactions. The 2D projection of twin plane normal (Z_{twin}) and twin shear direction (X_{twin}) are shown by the black arrows on the $\sigma_{13}^{\text{twin}}$ and $\sigma_{33}^{\text{twin}}$ stress maps, with dashed arrow indicating the direction with a component into the paper surface (the global coordinate is shown in the first image). The $\sigma_{13}^{\text{twin}}$ and $\sigma_{33}^{\text{twin}}$ stress tensor component represent the stress corresponding to twin shear and normal to twin plane (rotated from sample coordinates to twin coordinates). It can be seen that at both ends of the twin lath there are sharp changes of stress states, the distribution of the shear stress ($\sigma_{13}^{\text{twin}}$) states are highly polarized, as at one end of the twin lath the stress state change from negative to positive from one

side to the other and the same transition is observed at the other end of the twin. This polarity of stress state is consistent with the LSF maps, which show that the locations at twin tip that have negative shear stress generally have a lower LSF state and the places with positive shear stress have higher LSF. While the LSF surrounding the twin tip provides a favourable condition for twin propagation, the GSF doesn't seem to be in favour of this, as the average GSF surrounding twin lath is only about 0.25.

Similar effects around a propagating twin (approximately 3 μm wide and 100 μm long with the top end 20 μm from the grain boundary) tip can be observed for a different variant as shown in Figure 6.4. This twin is also a $\{1\ 1\ \bar{2}\ 2\} \langle \bar{2}\ 11\bar{3} \rangle$ compression twin type.

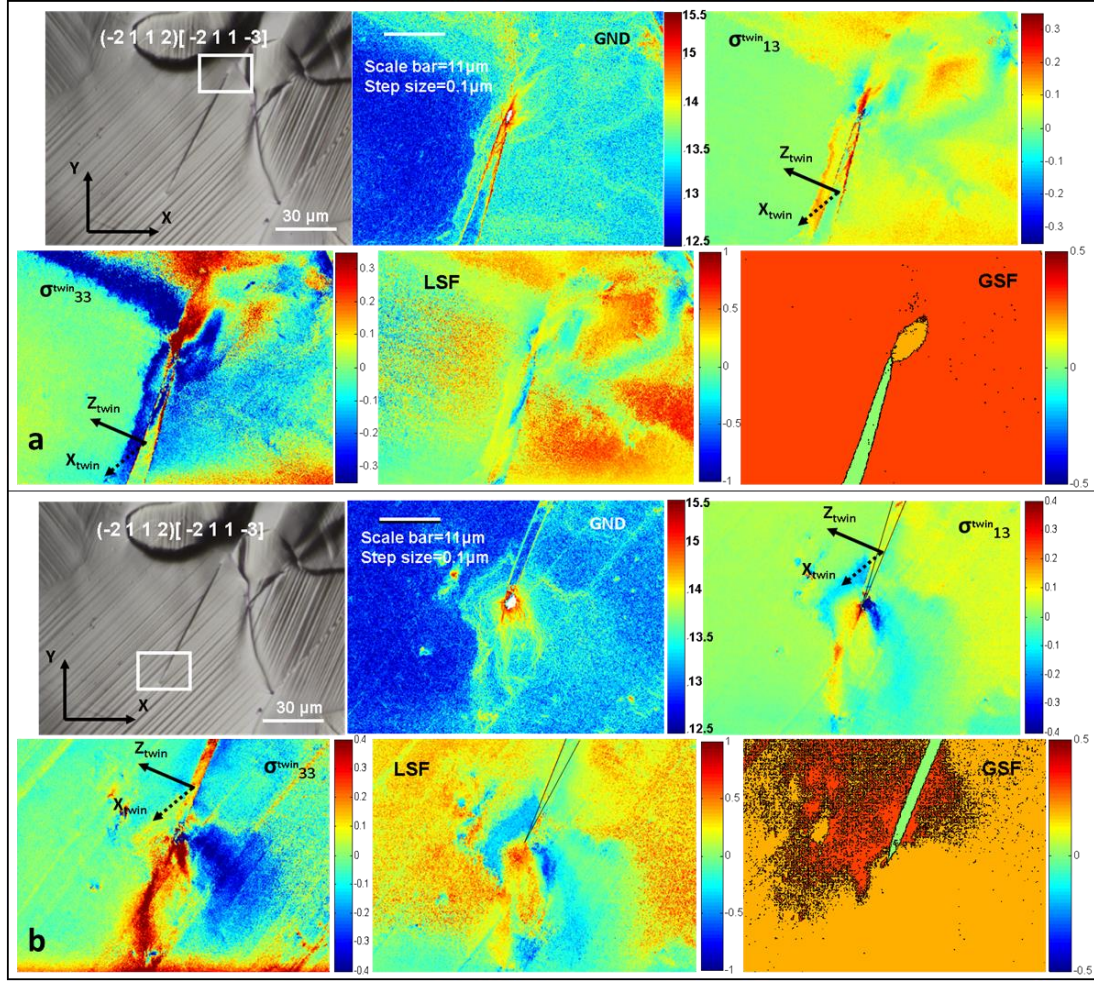


Figure 6.4: Twin imbedded in the interior of a large grain. Images in (a) and (b), from top left to bottom right, are optical image, GND density map, σ_{13}^{twin} stress map, σ_{33}^{twin} stress map, local Schmid factor map (LSF, calculated using stress tensor obtained from HR-EBSD analysis as shown in eq.6.2), and global Schmid factor (GSF, calculated using external loading direction and twin plane orientation as shown in eq.6.4) map.. Stress tensor components are those resolved onto twinning plane. 2D projection of the local coordinate system on the twin variant is indicated by X_{twin} (twin shear direction) and Z_{twin} (twin plane normal). The X and Y on first image indicate the global coordinate system. GND density scale bar is in $\log_{10}$ scale with unit m^{-2} . Stress map scale bar unit is GPa.

Again, the GND density is mainly concentrated around the twin tips and tends to radiate out along twin lath direction. The stress states change sharply across twin lath around the twin tips, as revealed from both the $\sigma_{13}^{\text{twin}}$ and $\sigma_{33}^{\text{twin}}$ stress maps. The positive shear at both ends of the twin lath tend to provide a favourable condition for twin propagation as revealed by the consistency between the position of highly positive LSF and $\sigma_{13}^{\text{twin}}$ shear stress around twin tips. It can also be seen from the GSF map from figure 6.4 (a) that while the parent grain generally provides a favourable resolved shear stress (RSS) environment for this twin variant, the local deformation at one side of the tip tends to lower the RSS on the twin plane. The shape of the region of lowered RSS broadly converges with the shape of negative stress along both shear direction ($\sigma_{13}^{\text{twin}}$) and along twin plane normal ($\sigma_{33}^{\text{twin}}$).

6.4 Stress field at twin tip approaching grain boundary

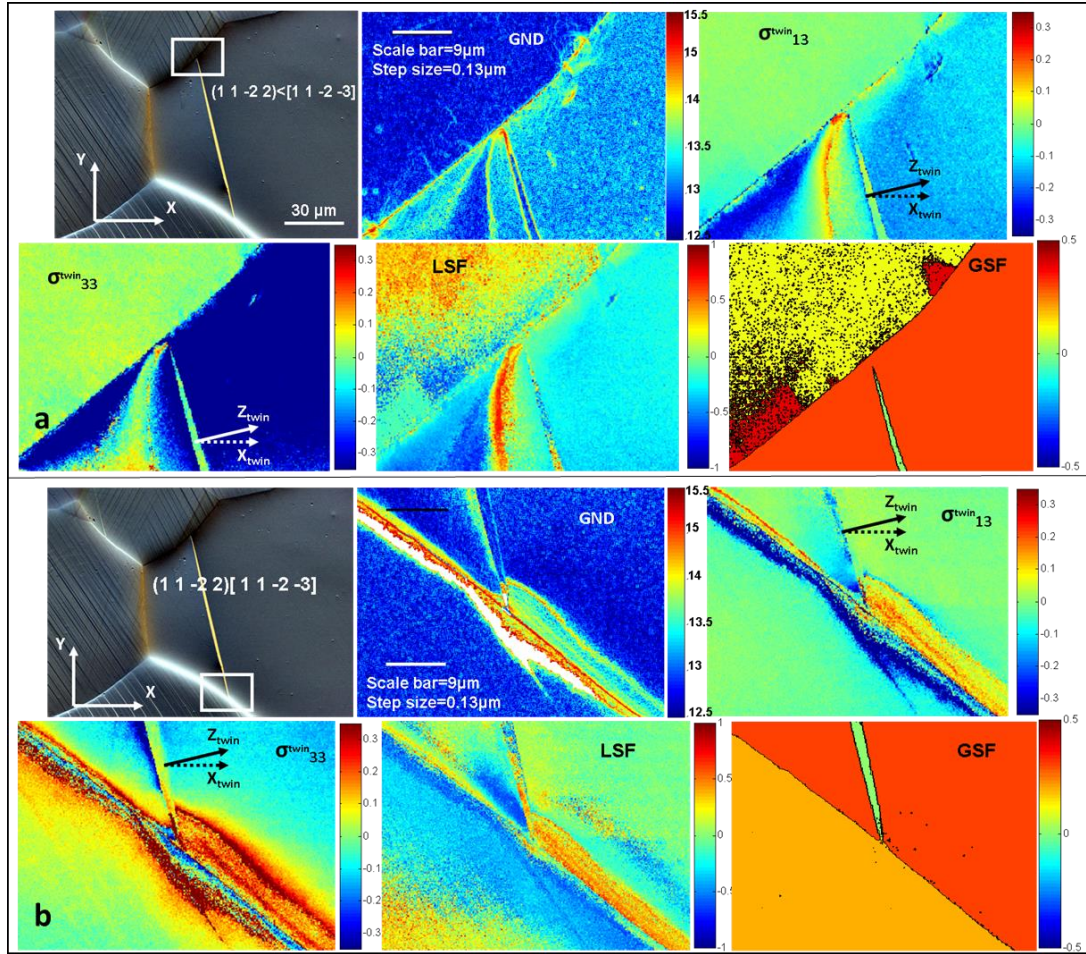


Figure 6.5: Local deformation field at both ends of a twin across a whole grain. Images in (a) and (b), from top left to bottom right, are fore scattered electron image, GND density map, σ_{13}^{twin} stress map, σ_{33}^{twin} stress map, local Schmid factor map (LSF, calculated via eq.6.2), and global Schmid factor (GSF, calculated via eq.6.4) map. Stress tensor components are those resolved onto twinning plane. 2D projection of the local coordinate system on the twin variant is indicated by X_{twin} (twin shear direction) and Z_{twin} (twin plane normal). The X and Y on first image indicate the global coordinate system. GND density scale bar is in $\log_{10}$ scale with unit m^{-2} . Stress map scale bar unit is GPa.

The twin variant shown in Figure 6.4 is also of compression type on the $(1\ 1\ \bar{2}\ 2)$ plane and along $[1\ 1\ \bar{2}\ \bar{3}]$ direction. It is about 200 μm long and 5 μm wide on the sample surface and spans across the grain with tips located close to grain boundaries. This twin is suspected to be reaching the end of the propagating phase and may have just started the thickening stage, judged from the lenticular shape of the tips (most obvious on the GSF maps) and the thickness of the twin lath. GND density and stress maps have shown that the majority of the deformation gradients are confined within the twining grain while no obvious stress and GND density are passed on to the top grain, the bottom grain also demonstrated limited amount of deformation transfer largely confined within the vicinity of the grain boundary. Both the stress along twin shear direction and twin plane normal show a more spread distribution along both grain boundary and twin lath rather than being concentrated mainly on twin tips as was seen in Figure 6.3 and 6.4. The negative-positive polarity of twinning shear is still visible at both ends of the twin lath. This fundamental change of the distribution of local positive shear stress field as well as the LSF condition (i.e. from mainly at tip to mainly at the twin sides) as compared with Figure 6.3 and 6.4 seems to be effectively indicating the change from twin propagation phase towards twin widening phase.

6.5 Stress field at twin tip interacting with grain boundary

The twin types shown in Figure 6.6 are also compression types. The difference with the cases shown in section 6.3-6.5 is that in this case the twin tips are no longer sharp, they are blunted due to interaction with grain boundary and seem to conform to the trace of grain boundary as is evident from the fore scattered electron images (first image) in Figure 6.6. The two twin and grain boundary interaction cases shown in Figure 6.6 are from two different grains with Figure 6.6 (a) demonstrating a twin tip propagation stopped by a grain boundary and just started to widen, and Figure 6.6 (b) showing a twin tip that has widened significantly as can be judged from the relative length between the line of intersection between twin tip and grain boundary. A limited amount of plasticity have been induced in the neighbouring grain as can be seen from both the slip feature distribution in the fore scattered electron images and the GND density maps. The amount of plasticity transfer seems to increase with the thickness of twin lath as the thicker twin (Figure 6.6 (b)) has induced GNDs in larger population and in wider spatial range compared with the thinner twin (Figure 6.6 (a)).

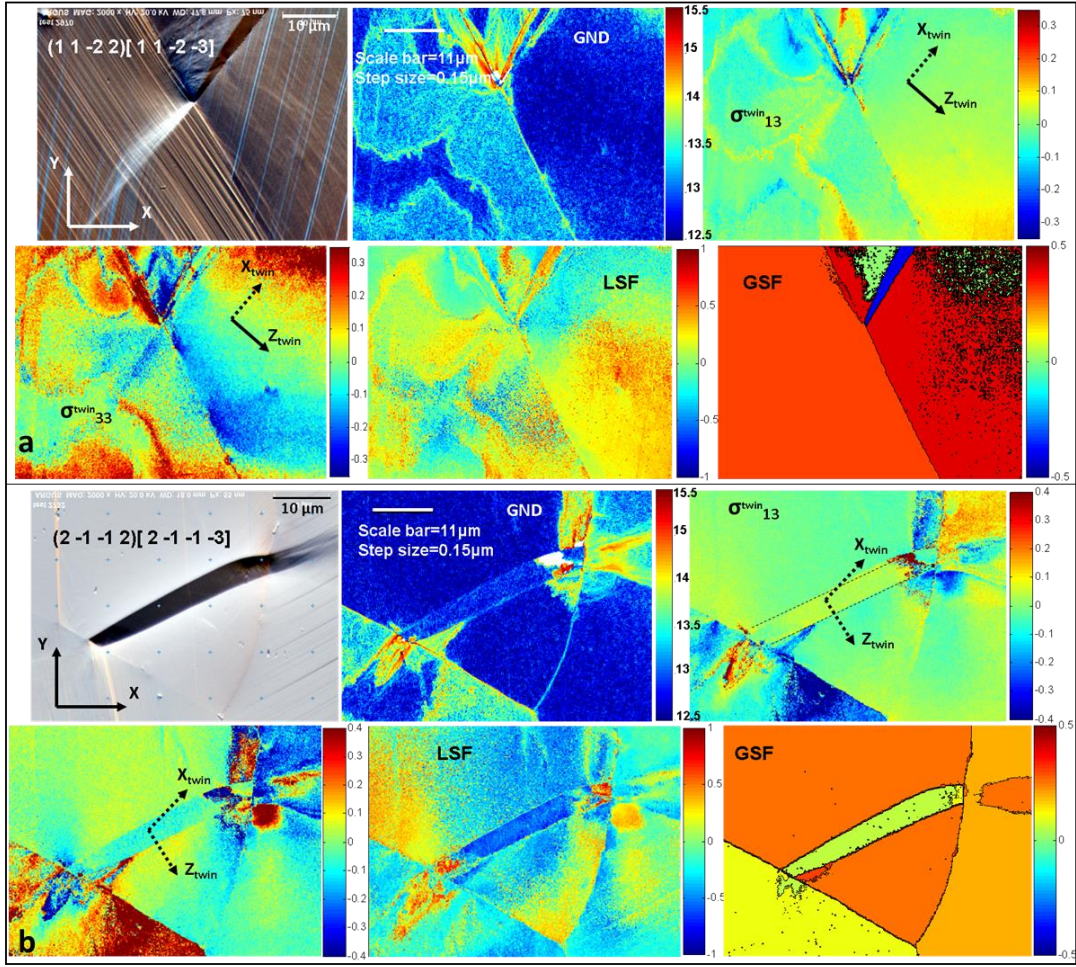


Figure 6.6: Twin tip interactions with grain boundaries. Images in (a) and (b), from top left to bottom right, are fore scattered electron image, GND density map, σ_{13}^{twin} stress map, σ_{33}^{twin} stress map, local Schmid factor map (LSF, calculated using stress tensor obtained from HR-EBSD analysis as shown in eq.6.2), and global Schmid factor (GSF, calculated using external loading direction and twin plane orientation as shown in eq.6.4) map.. Stress tensor components are those resolved onto twinning plane. 2D projection of the local coordinate system on the twin variant is indicated by X_{twin} (twin shear direction) and Z_{twin} (twin plane normal). The X and Y on first image indicate the global coordinate system. GND density scale bar is in $\log_{10}$ scale with unit m^{-2} . Stress map scale bar unit is GPa.

The shear stress ($\sigma_{13}^{\text{twin}}$) map in Figure 6.6 (a) shows a distribution of positive component radiating out from twin tip and grain boundary intersection similar to the case shown in Figure 6.5, indicating a strong driving force for twin to widen. This observation is consistent with the case reported in Section 6.4, where twin is just about to intersect with grain boundary and differs from Section 6.3, where twin is far away from grain boundary. This difference again seems to indicate that the difference in stress and LSF distribution at the surrounding matrix near twin tip is an indicator of different phases of twin growth. The GSF in general have a large value in the parent, providing favourable condition for twin thickening, but the local deformation fields lead to such a condition that the LSF surrounding large portion of the twin lath in the parent grain is negative apart from the very local area at twin tip. This seems to indicate that twin propagation is a very local phenomenon, it starts from the tip region, grow towards the region of highly positive LSF, and this growth spread from twin tip to the middle with continued application of external strain. The directionality (towards high magnitude and positive LSF) of twin thickening correlates well with the upward bulging of the twin lath as can be seen from the SEM image in Figure 6.6 (a).

The twin grain boundary interaction cases shown in this chapter differs from the slip band grain boundary interactions in that the former have imparted both positive and negative shear into the neighbouring grain while the latter only induce shear stress which is either positive or negative.

6.6 Discussion

Twinning activated by a tensile loading has been characterised using HR-EBSD at different phases of twin generation, i.e. propagation, near grain boundary, and intersection with grain boundary. The convenience of accessing local stress tensors at submicron scale makes HR-EBSD a powerful tool in studying deformation heterogeneity in crystalline materials.

In this Chapter, the stress tensor mapped on sample surface in global coordinate system at twin tips were captured by HR-EBSD. After identifying the active twin variant, this stress tensor was then rotated onto the local coordinate system defined by the twin system in such a way that the twin shear direction is defined as the X_{twin} direction and the twin plane normal is defined as the Z_{twin} direction. Under such convention, the σ_{13} component of the rotated stress tensor is aligned with the twinning shear and the σ_{33} component is the stress perpendicular to the twin plane. In addition, the local stress tensor at each interrogation point was also combined with the corresponding orientation matrix to generate a local Schmid factor which should be able to provide information on the resolved shear stress on the activated twin variant in the parent matrix.

Due to strong texture of the test specimen, all twins captured for this investigation were $\{1\ 1\ \bar{2}\ 2\} \langle 1\ 1\ \bar{2}\ \bar{3} \rangle$ compression twins. The twinning process was categorised into 3 phases: (1) twin propagation phase, when the twin lath is relatively short and fully imbedded in the interior of the grain and far away from the grain boundary, (2) end of twin propagation phase, when the twin tip is touching but not fully intersecting grain boundary (i.e. the twin tip maintains a

sharp lenticular shape at the grain boundary), and (3) twin thickening phase, when the twin tips intersect with grain boundary and have become blunted.

At the twin propagation phase, illustrated by figure 6.3 and figure 6.4, the twin stress concentration is significantly focused at the twin tip and radiates out along the twin shear direction. The stress concentration is found to change from positive to negative across the twin lath at the twin tips, and this polarity is consistent with the distribution of LSF. It was found that the local positive shear creates a positive LSF environment while the place of negative shear corresponds to the place of negative LSF. It has been found that twin variant activation is highly correlated with RSS [11], and unlike dislocation slip that can happen in both positive and negative directions, twinning only happens in the positive direction [128]. Therefore, the local positive shear provides a favourable LSF environment right in front of the twin tips to support continued twin propagation across the grain even when the global Schmid factor is not in favour of twin propagation in some cases. The local negative shear, located just away from directly in front of the twin, creates a negative LSF condition that might serve to prevent twin from thickening. This observation is broadly consistent with the recent modelling work [211] but the magnitude of twin shear in this research is much higher.

When approaching a grain boundary, the stress field at the twin tip changes dramatically as the compatibility of the neighbouring grain also starts to take significant effect in addition to the compatibility with the parent grain. Both high shear stress and high normal stress were found to distribute at twin tip and along twin lath and grain boundary. The local positive shear and positive LSF at

the twin tip were no longer found to distribute to directly in front of the twin tip, rather, they radiate out from the twin front sideways into the surrounding matrix, which is possibly due to effect of neighbouring grain and subsurface grain boundary orientation. This change of shear stress distribution and LSF environment seems to indicate the change of twining phase from propagating to thickening.

In both propagating and thickening phases, the LSF doesn't seem to provide a long ranging field that affects large portions of the twin lath, rather, there exists only a small window of favourable LSF field around the twin tip. This indicates that twin propagation and thicken is a highly localized event. A 'tip-control' mechanism seems to take effect based on the very concentrated LSF field around twin tip at every case inspected in this study. The twin is able to grow longer because of the local stress state providing large RSS on the twin plane and when the twin is approaching grain boundary, the twin tip experiences a stress state that facilitates it to grow sideways, i.e. become thicker, and this thickening process starts from twin tip and progress into the middle of the twin lath under a continued application of far field loading. The commonly held impression that twins are lenticular in shape seems to act against this hypothesis, but the fact that a twin experiences less constraint in the interior of grain compared with grain boundary should also be taken into account. In fact as twins grow thicker the lenticular shape seems to diminish as can be seen from the case reported in figure 6.6 (b) as well as other twin investigation literatures [210, 212].

When a twin is approaching a grain boundary stress may or may not be induced into the neighbouring grain as can be seen in Figure 6.5. This depends on several factors, the most obvious and phenomenological one being that if the twin tip is actually ‘touching’ the grain boundary then significant stress in the neighbouring grain is likely. This is most obvious from the GSF map in Figure 6.5 (a), in which the twin tip is about 1 μm from the grain boundary, while the other end of tip is actually intersecting with the grain boundary (Figure 6.5 (a)) and has induced deformation in the neighbouring grain. Another factor is that the twin shear direction (as indicated by the dashed arrow X_{twin} in figure 6.5) in this case does not have significant resolved component perpendicular to the grain boundary plane. This effect also influences the bottom twin tip in Figure 6.5 (b) which is actually interacting with the grain boundary and due to large shear component along the twin plane rather than perpendicular to twin plane the stress field in this case is smeared along the grain boundary plane rather than extended into the interior of the neighbour grain.

The capability of the neighbouring grain in accommodating twinning shear was assessed by Qin et al. [212]. In principle, this method works by first rotating the

twin shear displacement gradient tensor $\begin{pmatrix} 0 & 0 & S \\ 0 & 0 & 0 \\ 0 & 0 & 0 \end{pmatrix}$ (S is twinning shear and

$S = 0.218$ for $\{1\ 1\ \bar{2}\ 2\}$ compression twins in titanium), as defined in twin local coordinate system consistent with directions defined consistent with this study, into the orientation of the neighbouring grain. Once this is done for the case reported in Figure 6.4, the shear displacement in the top and bottom grains takes the form of

$$E_{\text{top}} = \begin{pmatrix} -0.0563 & 0.0587 & 0.0783 \\ 0.0503 & -0.0525 & -0.0699 \\ -0.0782 & 0.0816 & 0.1088 \end{pmatrix}$$

And

$$E_{\text{bottom}} = \begin{pmatrix} -0.0375 & 0.0155 & -0.0161 \\ -0.1678 & 0.0694 & -0.0723 \\ -0.0740 & 0.0306 & -0.0319 \end{pmatrix}.$$

Assume a crystal Cartesian reference frame on a crystal orientation is defined such that X direction is consistent with the $\langle 1\ 1\ \bar{2}\ 0 \rangle$ Burger's vector direction and Z direction is the basal plane normal direction, the rotated displacement gradient tensor should have $|E_{12}| + |E_{21}|$ representing the tendency for prismatic slip, $|E_{13}| + |E_{23}|$ the tendency for basal slip, and $|E_{31}| + |E_{32}|$ the tendency for twinning or $\langle c+a \rangle$ pyramidal slip. The $|E_{12}| + |E_{21}|$ was found to be 0.183 for the bottom grain and 0.109 for the top grain, and therefore the bottom grain is 'softer' and the top grain is 'harder' in terms of accommodating the twinning shear. This might help explain the less well-developed deformation field in the top grain compared with the bottom grain in Figure 6.4. While neat in categorizing grains into soft and hard based on the ability to accommodate twinning shear, this method is perhaps most useful in performing a statistical analysis of twin variant selections.

6.7 Conclusion

Different stages of a twin lath development were characterised using HR-EBSD. This study focus on the $\{1\ 1\ \bar{2}\ 2\}$ compression twin variants nucleated in the interior of the grain and progressing to interact with the grain boundary. Based on the HR-EBSD stress maps and LSF field calculation, the following conclusions may be drawn:

1. During the propagation stage, both positive and negative shear stress concentration can be found at twin tips and the positive stress field creates a favourable LSF condition for twin extension while the negative stress field off the tip front and at twin lath boundary tend to suppress twin from growing thicker.
2. When a twin is approaching grain boundary, the local stress state changes dramatically and the positive shear field is no longer found in front of the twin tip, rather, a small window of positive shear stress and favourable LSF condition affecting only small portion of the twin tip can be found at the edge of twin lath and radiate out sideways into the matrix.
3. The change of LSF condition indicates the change of twinning phases, i.e. twin stopped to propagate and started to grow thick.
4. Resolved shear stress as computed from far-field external loading is not reliable in analysing twin variant selection. The resolved shear stress based on local stress tensor and lattice orientation at immediate twin environment is needed for better prediction of active twin variant.
5. A 'tip-control' mechanism is proposed for twin propagation and thickening processes. Local stress state from twin tip/parent grain

interactions play a major role in controlling twin extension, and twin thickening starts from the tip and progress into the bulk at continued application of external loading.

Chapter 7 Summary

This thesis has presented the local deformation gradient characterisation of the interactions between slip bands, the dominant deformation mechanism for all metals, and grain boundaries. The difference of crystal orientations across the grain boundary lead to different scenarios of interactions. When the incoming slip system is better aligned with hard slip systems (i.e. high CRSS) in the neighbouring grain, it is likely to be stopped and leads to stress concentration in the neighbour grain. When the incoming slip system is better aligned with easy slip systems (i.e. low CRSS) in the neighbour grain it can either transfer or dissociate into grain boundary plane and no localized high intensity stress concentration is caused. These scenarios are consistent with other major investigations in the field using TEM [117, 120, 123, 208, 213, 214] and could be linked to different theories and hypothesis of slip band and grain boundary interaction in the history of this field of research [87, 88, 135, 136, 215, 216].

The stress field as a result of slip band and grain boundary interactions can be quantified using the EFN model [110]. And the stress intensity factor K as calculated from the EFN model correlates well with the number of dislocations in the pile up plane. No correlation between stress intensity factor K and the geometric alignment between slip systems across the grain boundary can be found and this is possibly due to stress fluctuation along the line of intersection between slip plane and grain boundary as revealed by the DAXM experiment. It is equally possible that the angle between lines of intersection between neighbouring slip systems with the grain boundary should be used instead of the angle between slip plane normal approximation to assess the geometrical

alignments between slip systems [201]. Both the HR-EBSD and DAXM stress intensity calculations agree with each other and pave the way for all such measurements. The way to quantify stress intensity used in this research could serve as a standard method to compare between experiments and modelling as well as comparison across different experimental techniques.

Deformation twinning is another very important deformation mechanism especially for HCP metals [128, 135]. Three stages of a compression twin development were captured using HR-EBSD. During the twin propagation stage (i.e. twin growing longer) the local stress states surrounding the twin tip tend to promote its elongation while suppressing it from growing wider. When the twin reaches a grain boundary, the local stress states no longer support its propagation but instead tend to promote its widening. At this stage, whether the twin imparts deformation in the neighbour grain depends on whether the twin tip is actually intersecting the grain boundary as well as the angle between the twin shear direction and the grain boundary. When a twin intersects with grain boundary, it induces significant deformation in the neighbouring grain and starts to grow wider. In all three stages of development mentioned above the twin tip seems to play a controlling role.

The stress, strain, and GND density distribution for slip band and grain boundary interactions were characterized using both electron based technique (HR-EBSD) [165, 173] and X-ray based technique (DAXM) [191]. Both yield very similar stress magnitude and the distribution of both stress and GND relative to microstructure features converge very well between the two techniques. DAXM has the advantage that it provides information in 3D but

suffer from significantly limited beam time and availability of facilities. Although HR-EBSD only provides information in a 2D plane, but it provides the same parameter characterisation capability as DAXM and with much better spatial resolution. It could even go to nano scale with transmission Kikuchi diffraction (TKD) [166]. 3D stress characterisation with HR-EBSD is also possible with FIB serial sectioning (however, this is destructive) and would require further improvement of computer reconstruction technique.

Chapter 8 Possible Future Research Directions

The HR-EBSD is a viable and highly convenient tool that characterizes point to point strain variation in a crystalline material. Compared to Synchrotron X-ray diffraction, the HR-EBSD method is faster and more accessible. Compared to TEM, it doesn't suffer from tedious sample preparation and probes much bigger area, and therefore offers more statistical sampling that is representative of the bulk. However, HR-EBSD in the conventional experimental set up suffers from limited spatial resolution and therefore not able to characterize microstructures in the lower end of nano meter scale. Recent development of the EBSD technique in transmission mode (known as TKD or T-EBSD) [166] is able to reduce the step size to 5-10 nm range and therefore enabling strain investigation in a series of scenarios where the feature of interest is not easily accessible by conventional EBSD, for example, intragranular stress corrosion cracking [217], metal-oxide interface in nuclear fuel cladding zrcaloy [218], nanoscale multilayer metal composite [219], Martensite [220], as well as thermal barrier coatings [221]. Applying HR-EBSD to these investigations has potential problems such as focused ion beam damage and thin film stress relaxation (as standard TEM samples are used for TKD) which might need to be quantified for better data interpretation.

Another limitation of the HR-EBSD is that at current stage a reference pattern needs to be chosen within the range of similarly orientated patterns. As the stress state of the reference pattern is unknown the calculated distribution and magnitude of the stress field varies with the reference patterns chosen. This has minimal effect in situations where deformation is small and the stress state

of interest is only within one grain or in a single crystal and is heterogeneous across the whole region (as is the situation reported in this thesis). However, the reference pattern issue does prevent the application of HR-EBSD to the situation where deformation is big and uniform across multiple grains. One obvious example is shot peening, where whole grains in the region near shot peened surface could demonstrate compressive strain [222]. In this situation, depending on the place of reference chosen, the final calculated stress state within the grain could either be in compression (with lower magnitude) or in tension, or even zero in places. The solution to the 'reference problem' of HR-EBSD has been attempted using CPFE modelling [223], but the certainty of the final corrected 'absolute' stress state relies on the certainty of the simulation, and is difficult to calibrate without a third-party technique. Another way to tackle the 'reference problem' is to simulate a stress free reference pattern [224]. Recent advancement on this topic [225] have demonstrated a critical issue regarding the knowledge of EBSD pattern centre position with subpixel certainty, as an error in pattern centre position of 20 μm , for example, produced strain noise of up to 0.06% [225]. Therefore, to improve the HR-EBSD characterisation technique two problems need to be solved: 1) to develop a consistent and reliable method to determine pattern centre position, 2) to simulate stress free EBSD pattern as reference pattern for cross correlation.

This thesis has presented results where stress concentration is of interest within single grain and the stress distribution is highly heterogeneous (mainly at slip band/grain boundary intersection and twin tip), therefore the reference points chosen far away from the stress concentration can be considered stress

free with relatively high certainty, as has been confirmed by the DAXM experiment. The results presented represent the status of the microstructure after loading. This doesn't give a full picture of the microstructure evolution process during the whole loading and unloading circle. For example, the blocked slip bands that induce stress concentration in the neighbouring grain could either lead to crack formation [84] or twin nucleation [86]. But to differentiate the conditions for these two processes rely on in-situ deformation investigation.

This can be done via two different routes: the first route is to do EBSD on a polycrystalline tensile testing specimen that fits into a small in situ tensile stage. Once the EBSD map of the sample surface is obtained one can calculate possible deformation systems to be activated in each grain during a uniaxial tensile testing, using the EBSD results and the deformation trace analysis techniques presented in this thesis. The established grain or grains of interest can then be monitored during the process of tensile testing with intermittent HR-EBSD scans at different strain levels (due to prolonged data acquisition time for HR-EBSD scan, mapping data in larger area is not realistic for in-situ testing). The second route is to do a uniaxial deformation first, and then various slip-band and grain boundary interactions can be determined using EBSD in a similar way as shown in this thesis. The particular cases of slip band and grain boundary interactions that are suspect of damage nucleation site can be milled into micro-pillars, with both the slip band and grain boundary extending throughout the whole depth of the pillar. Pillar compression or tension (requires specific pillar design, for example [226]) testing can then be conducted in SEM

with the assumption that the shear state in the active slip system could be 'resumed'.

The first route has the advantage that it is relatively quick and simple and the local strain/stress information can be available from HR-EBSD scan, but it suffers from the fact that the area of interest scanned using HR-EBSD may not deform the same way as predicted due to the microstructure related strain heterogeneity. The second route has the advantage that the deformation structure of interest could be accurately pin pointed, but it suffers from longer sample preparation time as large pillars are needed (in order to be sure that the grain boundary and slip plane are included through the whole length of the pillar). The advantage of doing these experiments inside SEM is that it is relatively certain that which microstructure is activated at which strain level and how does this affect the stress strain curve.

This thesis did not involve the situations of twin-grain boundary interactions. But this kind of interactions can be and should be investigated and preferably with deformation process monitored inside SEM, during a whole loading-unloading process, so that possible twin related slip activity, twin nucleation, crack nucleation, and de-twinning can be directly observed and rationalized with local crystallographic and mechanical features (from HR-EBSD scans). It is recommended that for twin/grain boundary interaction investigations the deformation rate should be higher ($>1\mu\text{m}/\text{sec}$) so that larger populations of twins could be activated.

Knowledge of the grain boundary and microstructure dependent microstructure evolution is essential to establish a reliable model for damage nucleation and texture evolution predictions.

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