

Full Length Article

Deformation behaviour of magnesium bicrystals with symmetrical 90° $\langle 11\bar{2}0 \rangle$ tilt grain boundaries analysed by large area EBSD mapping

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Abstract

Grain boundaries play a significant role in the deformation of polycrystals. Their response to deformation is however not completely understood, particularly with respect to how they accommodate lattice rotation of adjoining crystallites by changing their structure and geometry. The current study thus investigates the deformation behaviour of Mg bicrystals with 90° $\langle 11\bar{2}0 \rangle$ symmetric tilt boundary strained in plane-strain compression up to different final strains. Due to the initial soft orientation of the two crystals, activation of basal slip in each crystal gave rise to lattice rotation around the transverse direction towards the compression direction of the channel-die. Hundreds of single EBSD maps with a small step size were obtained from the GB region and stitched together to produce large panoramic maps of a macroscopic scale. Although very time-consuming, this technique has proven useful in clarifying the origin of the non-uniform deformation zones in the vicinity of the grain boundary and explains the mechanisms, by which the grain boundary was able to cope with the imposed strain before fracture. Interestingly, several variants of extension twins were observed as an additional deformation mechanism despite having negative Schmid factors. Systematic investigation of their resulting combined shear components with respect to the sample coordinate system revealed an alignment along the longitudinal direction of the channel-die, therefore justifying their nucleation.

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1. Introduction

Magnesium, being the lightest structural metal, has a very high potential for use in automobile structural applications through reducing vehicle weight, which consequently leads to a lower carbon footprint. However, low room temperature ductility of conventional wrought Mg alloys currently impedes their wide use industrial application [1,2]. To improve the formability of Mg alloys, it is therefore crucial to fully understand their deformation behaviour in terms of strain accommodation capacities in grain interiors and in the vicinity of grain boundaries (GB). Fundamental deformation studies

on magnesium single crystals carried out in the past investigated the activation of different slip and twin systems as a function of crystal orientation and loading condition [3–12]. Generally, basal slip is the only slip system that can be easily activated at room temperature. The resulting simple shear is accompanied by a lattice rotation where the c-axis rotated towards the compression direction (CD). In other cases, where basal slip is geometrically restricted, extension and contraction twins are formed during deformation, which can occur despite a negative Schmid factor (SF). In that case, such twins are referred to as anomalous twins [8,13–15].

Unlike single crystals, research studies on Mg bicrystals have received less attention, likely because it is more difficult to fabricate bicrystals with specific grain boundary geometry. Schreiber et al. [16] analysed the deformation behaviour of a 90° $\langle 10\bar{1}0 \rangle$ symmetric tilt grain boundary in

Abbreviations: Magnesium, (Mg).

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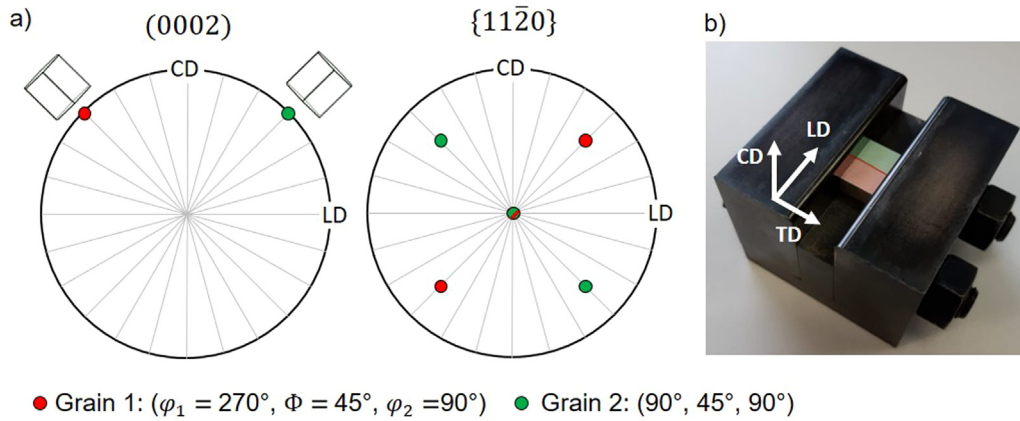


Fig. 1. (0002) and {1120} pole figures depicting the initial grain orientations of the bicrystalline sample (a) mounted in a channel-die device for plane-strain compression (b). The position of the grain boundary is indicated by the adjoining grains coloured in red (grain 1) and green (grain 2).

a pure Mg tricrystal deformed in plain strain compression (PSC) using a channel-die. The investigated grains were oriented for easy basal slip, i.e. with their c-axes at $\pm 45^\circ$ from the CD. The authors were interested in how the grain boundary would account for the lattice rotation of the adjoining grains undergoing basal slip during deformation. Similar to the case of a single crystal, the authors reported that with increasing strain the lattice in both grains rotated around the $\langle 10\bar{1}0 \rangle$ axis in opposite directions with respect to the boundary. The change of boundary misorientation during deformation was explained by interaction of gliding basal dislocations with the GB leading to gradual annihilation of the GB structural units. The authors also reported that at higher strains, the strain-induced lattice rotation becomes less homogenous. In addition, low angle boundaries were observed to form near the original boundary, which were able to accommodate additional local plasticity, where a lattice curvature of $\sim 0.005^\circ/\mu\text{m}$ was observed over a distance of few millimetres.

The appearance of a lattice curvature near the grain boundary as a result of non-uniform deformation and subsequent intergranular fracture were investigated in different bicrystalline systems in aluminium [17,18] and zinc [19]. As in magnesium and due to the presence of the GB, a non-uniform deformation zone is formed in the vicinity of the GB leading to a lattice curvature on both sides of the boundary. This demonstrates the capability of grain boundaries to accommodate lattice rotation of adjoining crystallites up to certain strains without fracture by changing their structure and geometry. This process remains elusive, as the details of how the initial grain boundary character changes during deformation are still largely unknown. Nevertheless, simulation models, such as the grain fragmentation model [20] already assume, that the slip induced lattice rotation of the grain interior leads to a lattice curvature in the vicinity of the GB. The focus of the present work is thus to shed light on the deformation behaviour of Mg bicrystals with $90^\circ \langle 11\bar{2}0 \rangle$ tilt grain boundary, explain the mechanisms, by which the grain boundary is able to account for the imposed strain before fracture and

to improve the understanding of the formation of a lattice curvature in GB region.

2. Experimental

The pure magnesium ($>99.95\%$ purity) monocrystalline seeds, used for the growth of the bicrystals investigated in this work, were aligned with their c-axes at $\pm 45^\circ$ around the $\langle 11\bar{2}0 \rangle$ axis, which was parallel to the transverse direction (TD) of the channel-die (Fig. 1). The growth process was carried in a stainless-steel mould covered with graphite coating using the vertical Bridgman technique. PSC rectangular samples with dimensions of 14 mm x 10 mm x 6 mm were cut out of the fabricated bicrystal using electrical discharge machining. The orientation of the seeds and that of the fabricated bicrystal was determined by the Laue technique. Fig. 1a shows the initial grain orientations (270° , 45° , 90°) and (90° , 45° , 90°) (Bunge convention) in the undeformed bicrystal with a $90^\circ \langle 11\bar{2}0 \rangle$ tilt boundary, whereas the position of the boundary with respect to the deformation geometry is shown in Fig. 1b.

Multiple bicrystalline specimens were deformed in PSC at ambient temperature and a constant strain rate of 10^{-3} s^{-1} up to selected strains of 5%, 15%, 30% and 40%. Deformation in the channel-die was confined to the CD and LD directions, whereas TD was the constraint direction. Frictional resistance imposed by the die walls was minimized by boron nitride serving as a lubricant. Automated data acquisition software collected the force and displacement data during the deformation tests. For microstructure investigations, the CD-LD mid-plane of the deformed specimens was subjected to metallographic preparation consisting of mechanical grinding and polishing with diamond suspensions along with final electropolishing in a 5:3 ethanol and phosphoric acid electrolyte at 2 V. Microstructure characterization was done by optical microscopy and electron backscatter diffraction (EBSD). To obtain a macroscopic view of the deformed microstructure at different strains, panoramic EBSD mapping over a large representative area was employed by measuring and stitching of

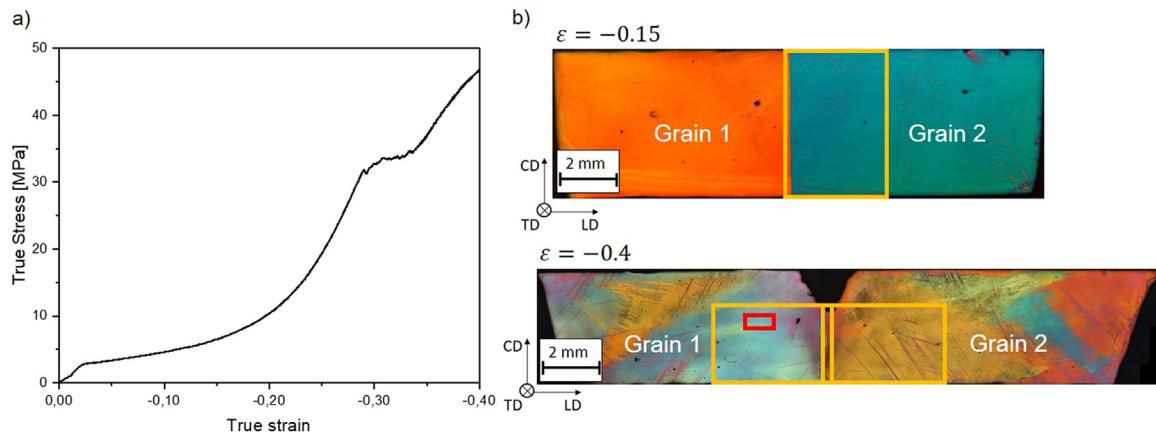


Fig. 2. (a) Flow curve of the 40% deformed bicrystalline sample at room temperature and $\dot{\varepsilon} = 10^{-3} \text{ s}^{-1}$. (b) Optical microstructures of the entire CD-LD mid-plane of the 15% (top) and 40% (bottom) deformed samples. Note the drastic shape change of the sample at -0.4 strain due to shear of both crystallites by basal slip and corresponding rigid body rotation due to the imposed plane-strain fixture.

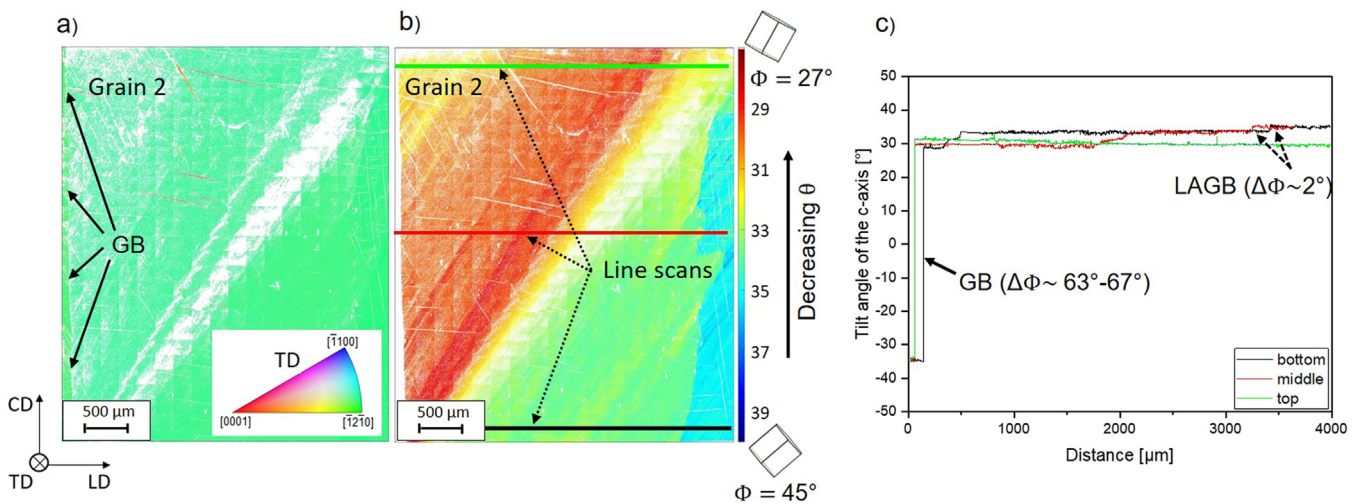


Fig. 3. Panorama EBSD map of the outlined area of the 15% strained sample shown in Fig. 2b represented in terms of inverse pole figure (IPF) colouring (a) and basal pole tilt distribution with respect to CD (b). In the latter map the initial orientation of $\Phi = 45^\circ$ was taken as a reference. The orientation profiles of the measured line scans in (b) at different positions of the sample (GB → grain 2 interior) are plotted in (c).

300 individual maps on average using step sizes of 3–4 μm . Analysis of the EBSD data was carried out using the MATLAB toolbox MTEX [21].

3. Results

3.1. Mechanical behaviour

Fig. 2a shows the flow curve of the sample deformed at the highest strain $\varepsilon_t = -0.4$. The initial portion of the stress-strain curve up to a true strain of -0.02 is likely attributed to surface asperities and imperfect lateral constraint due to minor misalignment of the specimen with respect to the loading direction. The flow curve after that depicted strain hardening of basal slip systems that rose more steeply at $\varepsilon_t \sim -0.15$. At a true strain of approximately -0.28 the stress-strain curve showed a small region of decreased work hardening resulting from the cracking of the sample, followed by a rapid increase

of stress up to 47 MPa just before the test was terminated at $\varepsilon_t = -0.4$.

The deformed microstructures of the 15% and 40% strained specimens are shown in Fig. 2b in terms of optical micrographs of the whole CD-LD mid-plane. The outlined areas (yellow rectangle) indicate the mapped regions by means of panoramic EBSD covering several square millimetres of the deformed bicrystalline samples (cf. Figs. 3–5). The smaller outlined area (red rectangle) in the higher deformed sample indicates another region of interest shown in Fig. 6. As shown by the optical micrographs, the GB at 15% strain remained intact, whereas at 40% it revealed a cavity of $\sim 1.4 \text{ mm}$ along the CD, evident in the upper region of the deformed sample (Fig. 2b). Noteworthy at the higher strain is the drastic macroscopic shape change of the PSC sample at both ends, which can be expected from shear due to basal slip in conjunction with rigid body rotation imposed by the PSC constraint [15].

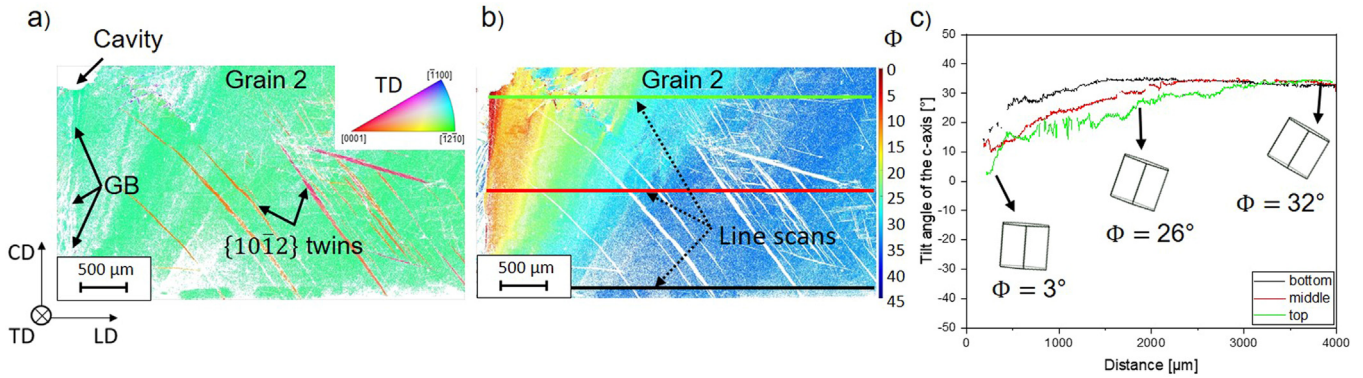


Fig. 4. Panorama EBSD map of the outlined area in grain 2 of the 40% strained sample shown in Fig. 2b represented in terms of IPF colouring with respect to TD (a) and basal pole tilt distribution with respect to CD (b). In the latter map the initial orientation of $\Phi = 45^\circ$ was taken as a reference. The orientation profiles of the measured line scans in (b) at different positions of the sample (GB $\rightarrow$ grain 2 interior) are plotted in (c). The unit cells illustrate the position of the basal plane with respect to the loading axis at different LD locations from the boundary.

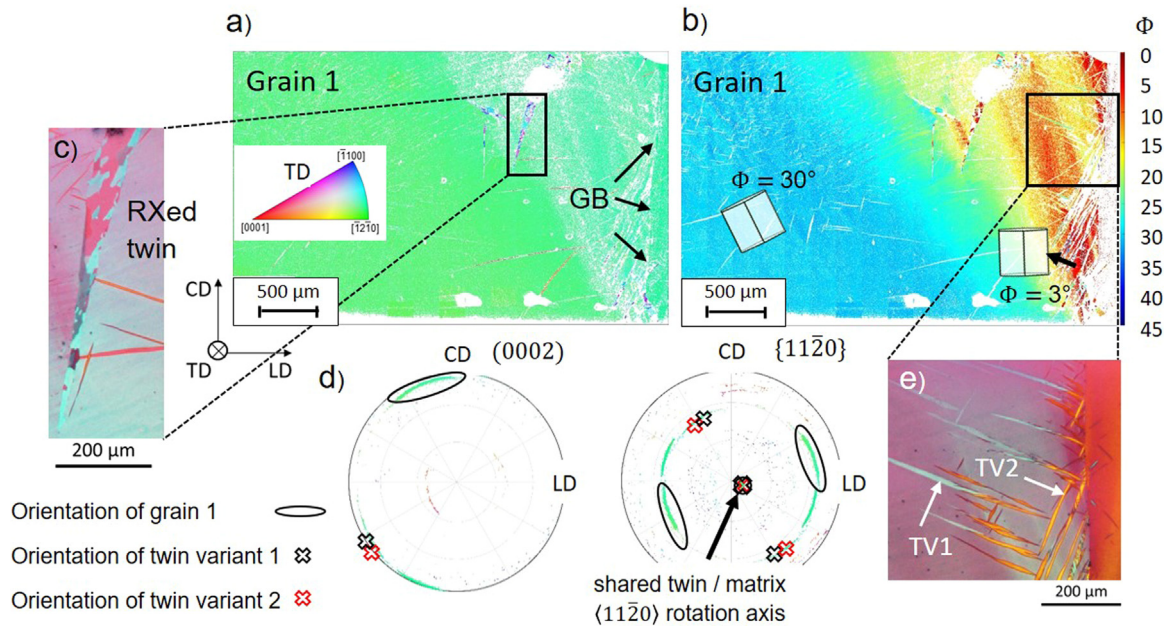


Fig. 5. Panorama EBSD map of the outlined yellow area in grain 1 of the 40% strained sample shown in Fig. 2b represented in terms of IPF colouring with respect to TD (a) and basal pole tilt distribution with respect to CD (b). (c) Optical micrograph of the outlined area of interest in (a) depicting a dynamically recrystallized twin band at ambient temperature. (d) Basal and prismatic scatter pole figures of the IPF map in (a) revealing the positions of basal poles after twinning and the shared $\langle 11\bar{2}0 \rangle$ rotation axis. (e) Optical micrograph of the outlined area of interest in (b) depicting intersecting extension twins of different variants TV1 and TV2 near the boundary.

3.2. Microstructure evolution at early stages of deformation ($\varepsilon = -0.15$)

The macroscopic nature of the investigated bicrystalline samples necessitates orientation analyses of large representative areas that can reveal information on how the grain boundary and the adjacent bulk are evolving with deformation. Fig. 3 shows a panoramic EBSD map of a total sampled area of $\sim 4 \text{ mm} \times 5 \text{ mm}$ that corresponds to the outlined region in Fig. 2b of the 15% strained sample. As evident from the IPF map in Fig. 3a, the $\langle 11\bar{2}0 \rangle$ axis of grain 2 remained closely aligned with the TD of the channel die. This indicates that the orientation gradients shown in Fig. 3b arise from dif-

ferent lattice rotations around the $\langle 11\bar{2}0 \rangle$ axis caused by basal slip. Taking the initial orientation of the undeformed sample as a reference ($\Phi = 45^\circ$), the tilt angle of the basal plane Φ revealed considerable changes ranging from 5° (dark blue) to 20° (dark red). In the vicinity of the boundary the tilt angle between the c-axis and the compression axis was $\sim 30^\circ$ on average, as compared to $\sim 35^\circ$ at a distance of 4 mm away from the GB. As a result of lattice rotation in both grains, the boundary misorientation after straining of 15% dropped to $\sim 65^\circ$ (Fig. 3c). It is noted that the amount of lattice rotation in the sampled CD-LD region was not homogeneous. As seen by the orientation profiles, the top part of the deformed sample depicted more or less a steady profile over

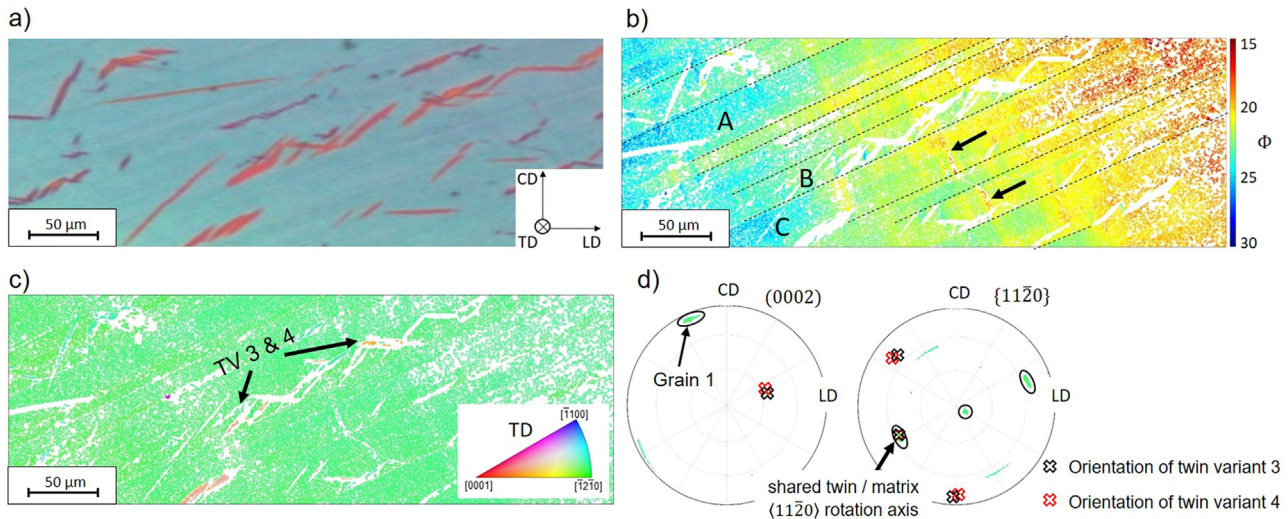


Fig. 6. Optical micrograph (a) and local EBSD Φ and IPF maps (b, c) of the outlined red area in grain 1 of the 40% strained sample shown in Fig. 2b. (d) Basal and prismatic scatter pole figures of the IPF map in (c) revealing the positions of basal poles of the twin pairs 3 & 4 and the shared $\langle 11\bar{2}0 \rangle$ rotation axis. The dotted lines in the Φ -map mark bands with local gradients in the lattice orientation, in which twin pairs of variants 3 & 4 were seen to form.

the measured length of 4 mm along the longitudinal direction LD. By contrast, the middle and bottom parts of the sample revealed noticeable transitions in their orientation profiles amounting to $\sim 5^\circ$ located at 1.8 mm μm and 0.15 mm from the GB, respectively. Additionally, a low angle boundary was also detected, which was indicated by abrupt jumps of $\sim 2^\circ$ in the orientation profile in the bottom and middle parts of the map.

3.3. Microstructure evolution at advanced stages of deformation ($\varepsilon = -0.4$)

A comparable matrix deformation behaviour regarding the formation of a heterogeneous deformation zone in the vicinity of the GB and associated lattice rotation was observed in the higher deformed sample at 40%. However, at this stage of deformation, the sample failed from its top part at the GB resulting in a cavity (cf. Figs. 2b and 4a). Additionally, the matrix revealed several extension twin bands of different variants that were not yet visible at low strains. Some of these twin bands (particularly the ones closer to the GB) were bent following the pattern of lattice curvature generated in the deformation zone close to the boundary. In grain 2 (Fig. 4), the matrix far away from the GB exhibited a similar degree of rotation to the one seen at 15% deformation ($\Phi \sim 32^\circ$), suggesting that the bulk behaviour is not strongly impacted by the increase of deformation. By contrast, the material within 500 μm from the boundary demonstrated substantial rotations of the basal plane from an initial $\Phi = 45^\circ$ to $\Phi < 10^\circ$ seen mostly in the top and middle part of the sample (Fig. 4b and c).

The matrix in grain 1 of the 40% deformed sample showed similar microstructural features to the ones seen in the neighbouring grain 2, particularly with respect to the characteristic conical shape of the deformation zone along the loading axis. Near the GB, the basal plane was almost perpendicular to the

loading axis, which can be seen in Fig. 5b by a tilt angle distribution below 5° (red regions) and in Fig. 5d by the proportion of basal poles populating the (0002) pole figure near CD. The $\{11\bar{2}0\}$ pole figure showed that the $\langle 11\bar{2}0 \rangle$ rotation axis of the lattice rotation was still very close to the TD. In addition to the orientation gradients in the matrix, the EBSD pole figures in Fig. 5d also show new orientations resulting from $\{10\bar{1}2\}$ extension twinning. At a distant of $\sim 1500 \mu\text{m}$ from the boundary one dynamically recrystallized twin was evident (Fig. 5c). Fig. 5e shows an example of these twins (twin variants 1 and 2) forming at / near the grain boundary. With respect to the matrix, twin variant 1 and 2 have $\pm 86^\circ$ misorientation angles to the parent grain matrix and share a common $\langle 11\bar{2}0 \rangle$ rotation axis (cf. Fig. 5d).

In addition to the aforementioned twins, multiple other extension twins of much smaller sizes were observed in the red outlined area within grain 1 (cf. Fig. 2b). The formation of these twins and the local environment were investigated on a micrometre scale by means of EBSD maps shown in Fig. 6. The optical micrograph in Fig. 6a shows that the twins formed in pairs of two variants (3 & 4). The Φ -map in Fig. 6b (limited to $15^\circ < \Phi < 30^\circ$) clearly demonstrates that the twin pairs were formed within bands (or stripes) of local orientation gradients (e.g. B). These bands were a clear indication of the existence of additional microstructural heterogeneity at smaller scales. The tilt angle of the c -axis in these bands revealed orientation gradients in the range of 5° – 10° , with some bands exhibiting smaller lattice rotations (e.g. A) than others (e.g. C). Interestingly, the amount of lattice rotation / strain accommodation in the band comprising the twins seemed to decrease / relax in comparison to direct neighbouring areas of adjacent bands with no twins. In addition, the tips of some twins (black arrows in Fig. 6b) were associated with narrow segments perpendicular to the band alignment, characterized by a larger degree of lattice rotation. The pole figures in Fig. 6d reveal that the twin variants 3 and 4 had a differ-

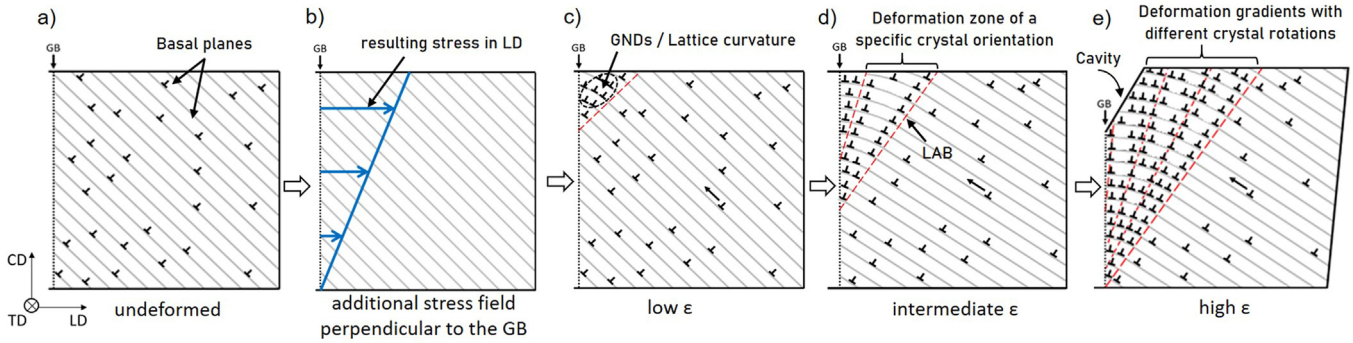


Fig. 7. Schematic illustration of the interaction of basal slip with the GB of the bicrystal. (a) initial state, (b) resulting stress field perpendicular to the GB, (c-e) at different stages of deformation corresponding to strain between 5% and 40% marking the formation of a lattice curvature, plastic zone and orientation gradients as a result of multiple non-uniform deformation zones that result in a crack in the upper part of the specimen as was observed by the experimental data.

ent orientation and another $\langle 11\bar{2}0 \rangle$ rotation axis compared to variants 1 and 2.

4. Discussion

4.1. Lattice rotations and the non-uniform deformation near the grain boundary

Since the deformation experiments were carried out at room temperature, it can be expected that basal slip is the only activated slip system [6]. Due to the deformation geometry and the symmetrical orientation of the bicrystals, two basal slip systems in each grain had a relatively high Schmid factor (SF) of 0.43, while the third system with a slip direction in TD had a SF of zero. In [15,16] activation of single basal slip results in shear in conjunction with a lattice rotation around the $\langle 10\bar{1}0 \rangle$ axis perpendicular to the $\langle 11\bar{2}0 \rangle$ slip direction and in the basal plane. In the present case, where two basal slip systems are equally favoured, the two crystals deformed in PSC by co-planar double basal slip, and have therefore experienced a lattice rotation around a $\langle 11\bar{2}0 \rangle$ axis. This gave rise to a rigid body rotation around TD accompanied by a rotation of the c -axis towards the CD rendering basal slip geometrically less favourable.

In contrast to the specimen regions far away from the GB, at the proximate location of the boundary, due to the shear associated with the basal slip in the two adjacent grains and mainly owing to the presence of the GB connecting the two parts of the bicrystal, additional tensile stresses were generated normal to the boundary in opposite directions in both grains (see Fig. 7b), leading to additional strain and, consequently, to additional rotation of the lattice/basal planes in the course of further compression (for illustration, see Fig. 8 in [16]). These stresses are minimal at the bottom surface of the specimen and increase with CD. The gradually changing, i.e. increasing (when approaching the boundary along LD) rotation of the basal planes resulted in a lattice curvature, as schematically shown in Fig. 7c–e. Thus, and with increasing strain, a conical-shaped deformation zone close to the GB is formed on top, which separates the deformation

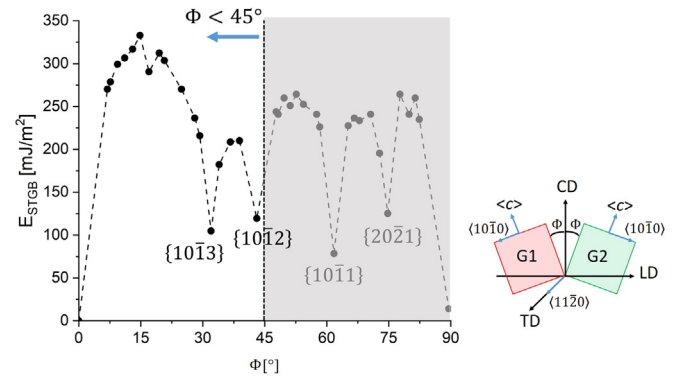


Fig. 8. Excess potential energies of STGB as a function of rotation angle Φ around a $\langle 11\bar{2}0 \rangle$ tilt axis, reproduced from [27]. The schematic illustrates the geometrical relationship between the STGB in this work and the deformation axes. The GB plane lies in the CD-TD plane (cf. Fig. 1b). The initial GB structure is indicated by a dashed line at 45° . The arrow denotes the range of Φ , relevant to the current work.

in the vicinity of GB from the one occurring in the bulk far away from the boundary (Fig. 7c). The interaction of multiple gliding basal dislocations facilitates the formation of low angle boundaries (LABs) from arrays of geometrically necessary dislocations (GND) associated with a lattice curvature, as illustrated in Fig. 7d, and reported in a previous study dealing with a $90^\circ(10\bar{1}0)$ symmetrical tilt GB [16]. At this point, it is unclear how strong is the interaction of gliding dislocations with these generated LABs. However, an obvious decrease in the EBSD pattern quality near such regions (e.g. white regions in Fig. 3) reasonably suggests that dislocations are hampered and entangled in front of these boundaries despite their very low misorientations. At higher strains and with an expanding conic stress field perpendicular to the GB due to the attempt of both grains to shear along LD, the border of the deformation zone moved deeper into the bulk to accommodate larger gradients of plastic deformation and maintain strain compatibility with the deforming neighbouring grain at the boundary. In that event, the crystal near the GB is split into deformation zones of slightly differing orientations (Fig. 7e). Noteworthy is that with increasing deformation in

conjunction with the imposed stress along CD, the distance between the GND arrays is expected to decrease, as was calculated by Eshelby et al. [22] and experimentally observed by Britton and Wilkinson via high resolution-EBSD [23]. Hence, the large reorientation in the lattice near the boundary ($\Phi \sim 0^\circ$) is explained by the large extent of dislocation accumulation. This condition renders any further basal slip unlikely due to the attained hard basal orientation ($SF=0$). As a result, the strain compatibility at the boundary can no longer be satisfied, which causes a cracking of the sample in the respective region at the GB (Fig. 7e). Additionally, the interaction of the GB with the dislocations changed the grain boundary character, which will be discussed in detail in the following section.

4.2. Change of the grain boundary character during deformation

As observed in previous bicrystal deformation experiments employing different crystal orientations and TEM analysis [16,24–26], gliding basal lattice dislocations were found to interact with the GB and cause annihilation of its structural units, thereby changing its original character. A similar process was observed in the investigated bicrystals of this work based on the orientation measurements carried out at different strains. Given the macroscopic stress state and the geometrical constraints of the channel-die, it was revealed that the produced shear in both grains by basal slip was accompanied by rigid body rotation around TD in opposite directions relative to the boundary. This resulted in a decrease of the angle Φ between the respective c-axes and CD on both sides of the boundary, which means that the GB character, and hence, the GB energy changed continuously during the deformation due to the additional stress field in LD (see Fig. 4). Additionally, one would expect a continuous decrease of Φ , and thus of the GB misorientation, with increasing strain (due to the increasing stress along LD) but also a homogeneously increase along CD (due to the larger stress field in the top part of the bicrystal). This behaviour, i.e. the change (increase) in misorientation across the boundary along the CD from top to the bottom of the sample, is indeed very well seen in Fig. 4b. In contrast, the specimen deformed to 15%, where the misorientation decreased along the CD (see Fig. 3), showed a completely different behaviour. To understand this anomaly, an energetic consideration is required, which is outlined in the following.

Fig. 8 depicts an energetic plot from MD simulations that was reproduced from [25] and shows the excess potential energies of symmetrical tilt grain boundaries (STGBs) as a function of the rotation angle Φ around $a \langle 11\bar{2}0 \rangle$ tilt axis that coincides with TD. Starting at Φ of 45° , which represents the initial state of the bicrystal, the character of the boundary will change with increasing strain as a result of decreasing tilt angle between the c-axis and CD. The corresponding Φ region in the energetic plot in Fig. 8 suggests that in the course of deformation, the initial GB can adopt two potential low energy structures, corresponding to coherent twin boundaries

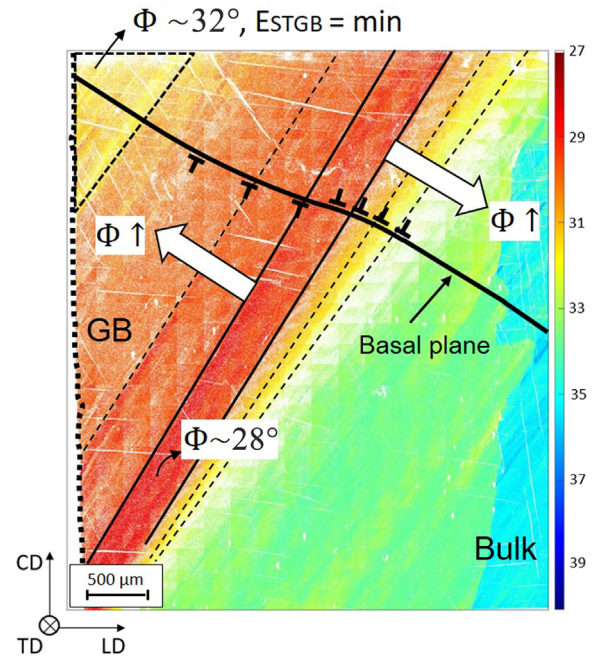


Fig. 9. Panorama EBSD Φ -map (grain 2, 15% deformation) that was shown in Fig. 3b featuring a non-uniform spread of lattice rotation within the CD-LD plane near the GB. The highest lattice rotation relative to the initial grain orientation was detected inside the band with $\Phi \sim 28^\circ$ bounded by two solid lines. Adjacent regions on both sides of the band are associated with lower rotations. The upper segment of the GB is hypothesized to have a low energy structure based on the data from Fig. 8. More explanation in the text.

$\{10\bar{1}2\}$ at ($\sim 43^\circ$) and $\{10\bar{1}3\}$ at ($\sim 32^\circ$). All other configurations were shown in [25] to contain grain boundary dislocations (or disconnections) with possible coherent boundary segments.

From all the different formulations in the literature regarding the interaction of gliding lattice dislocations with grain boundaries, one common outcome is the trapping of dislocations at the boundary. The magnitude of such interaction is influenced amongst other things by the GB misorientation. It is thus anticipated that with the change of the GB character during PSC deformation, the capacity of the boundary to trap dislocations can vary or even become exhausted when the boundary assumes a low energy structure as shown in Fig. 8. In such cases, it is likely that a repulsion between the lattice dislocations and the intrinsic GB dislocations occurs [25]. This might be linked to the inhomogeneous lattice rotation along CD shown in Fig. 3b and again in Fig. 9 (for a clearer view) for the bicrystalline sample deformed at 15% strain. The upper segment of the GB clearly depicts a reduced lattice rotation ($\Phi \sim 32^\circ$) compared to the middle and bottom parts ($\Phi \sim 30^\circ$), which according to the data from [27] in Fig. 8 can be associated with low ($\sim 100 \text{ mJ/m}^2$) and high ($\sim 188 \text{ mJ/m}^2$) energy structures, respectively. This emphasizes that even the slightest changes of the misorientation angle can result in significant alterations to the GB character / energy.

Fig. 9 also shows that the conical-shaped deformation zone residing between the GB and the bulk region of the sample consists of bands of equal Φ outlined by dashed / solid lines. These orientation bands were aligned perpendicular to the basal planes as was explained earlier on the basis of GND arrays. The orientation band with the largest rotation relative to the initial crystal orientation is outlined by two solid lines ($\Phi \sim 28^\circ$). Moving away from that band in the direction of the sample bulk results in a bulk deformation behaviour characterized by lower lattice rotations (i.e. larger Φ) and a more homogenous orientation spread. On the other hand, moving in the opposite direction, i.e. towards the GB should have resulted in higher lattice rotations in conjunction with the constrained GB deformation and the formation of the heterogeneous deformation zone. However, as revealed in Fig. 9, by moving closer to the boundary (e.g. along the marked basal slip plane) a lower amount of lattice rotations ($\Phi \sim 30^\circ$ – 32°) was evident.

Following the arguments presented above regarding the existence of minimum energy boundaries near $\Phi \sim 32^\circ$, and the low capacity of these boundaries to absorb gliding dislocations, it is hypothesized that at $\sim 15\%$ deformation, the investigated GB seeks to preserve its favourable atomic configuration by exerting a repelling force towards approaching dislocations. This force can be in the order of the yield stress [28]. This is seen in Fig. 9 in the upper GB segment, where the adjoining crystallite (yellow region) preserved its $\Phi \sim 32^\circ$ orientation as compared to the middle and bottom segments of the GB. This process is sketched in Fig. 9 in terms of GNDs of opposite signs associated with a positive and a negative lattice curvature on both sides of the orientation band with the maximum lattice rotation. Note that when the stress concentration induced by the increasing GND density reaches a critical value, the repelling force would fail to stop the movement of dislocations, which results in a change of the GB character out of the energetic minimum (e.g. Fig. 9 middle and bottom GB segments). At advanced strains when the lattice rotation in both adjoining grains approaches $\Phi \sim 0^\circ$, respective segments of the GB would technically disappear.

4.3. The role of anomalous twinning as an additional deformation mechanism

Microstructure characterization by means of optical microscopy and EBSD orientation mapping revealed activation of twinning in different parts of the bicrystal at strains higher than 15%. The very large extension twins near the edges are believed to form due to friction between the bicrystal and the channel-die. Hence, these twins will not be discussed any further. The other extension twins shown in Fig. 5 (variants of type 1 and 2) and Fig. 6 (type 3 and 4) at 40% strain appeared to be activated in pairs. This is usually the case where the formation of only a single twin variant would be associated with a negative Schmid factor and a strain opposite to the imposed deformation, as was explained in [15] for the case of deformed Mg single crystals oriented for

easy basal slip. For the SF to become slightly positive, the reactive stresses in TD due to the lateral constraints need to exceed the imposed active stress in CD, which is not realistic.

In the current work, the stress state correlated with the observed twin variant selection was very complicated due to the constraint stresses and the non-uniform lattice rotation around TD, resulting in internal stresses with tensile and compressive stress components. Fig. 10 is an attempt at estimating the variation of the SF for all the observed twin variants taking into account the change of the rotation angle Φ between the c-axis and CD, and the normal GB stress. The calculation was done following [15] assuming a stress tensor with compressive stresses σ_{CD} and $\sigma_{TD} = \sigma_{CD}/\beta$, with β being the ratio between the applied stress and the constraint stress. The value of $\beta = 3.47$ was determined for the initial bicrystal orientations on the basis of a zero strain in TD. In order to account for the constraining effects of the adjoining grains across the GB, the stress tensor included a tensile stress component in LD acting on the grain boundary σ_{LD} . The normal GB stress σ_{LD} changes along the GB, and was hence approximated to range between 0 and 0.5 of σ_{CD} .

The trend in Fig. 10 reveals that the SF for all considered twin variants becomes even more negative with increasing lattice rotation from the initial $\Phi = 45^\circ$ up to the ultimate case of $\Phi \sim 0^\circ$ of c-axis compression. The variation of the tensile stress normal to the GB does not affect this trend. It is therefore concluded, in line with the literature [29], that the activation of the observed twins was not to comply with the Schmid law but rather to accommodate local stresses associated with the non-uniform lattice rotation.

The resulting twinning shear from each of the four twin variants observed in this work is plotted in Fig. 11 with respect to the sample coordinate system. The initial orientation of the c-axis is marked by a thick arrow, and the shear directions of the respective twins by thin arrows. Due to the lattice rotation of the parent crystal towards $\Phi \sim 0^\circ$, the resultant shear direction of each twin variant is liable to a rotation around the TD up to a maximum of 45° over the course of deformation. This is also displayed in Fig. 11 in terms of normalized resolved shear components of the rotated vectors in the LD-TD and CD-TD planes. For simplicity, the calculations assumed equal twin thickness among all twin variants. Given the deformation geometry of the channel-die, sample extension is only allowed in LD. Hence, favourable twin variants are ones with a resolved shear component oriented in LD, without any shear component in TD and CD. From Fig. 11, the twinning direction of variant 1 depicted the best alignment with LD in the beginning of deformation with almost zero magnitude in the CD-TD plane. With the decrease of Φ due to the slip-induced lattice rotation, the corresponding SF becomes more negative due to increasing magnitude along the CD, thereby adding a deformation component of c-axis compression.

The fact that the formation of any single twin variant is not favourable in the current case supports the idea that the pairwise twinning events succeed in producing a favourable

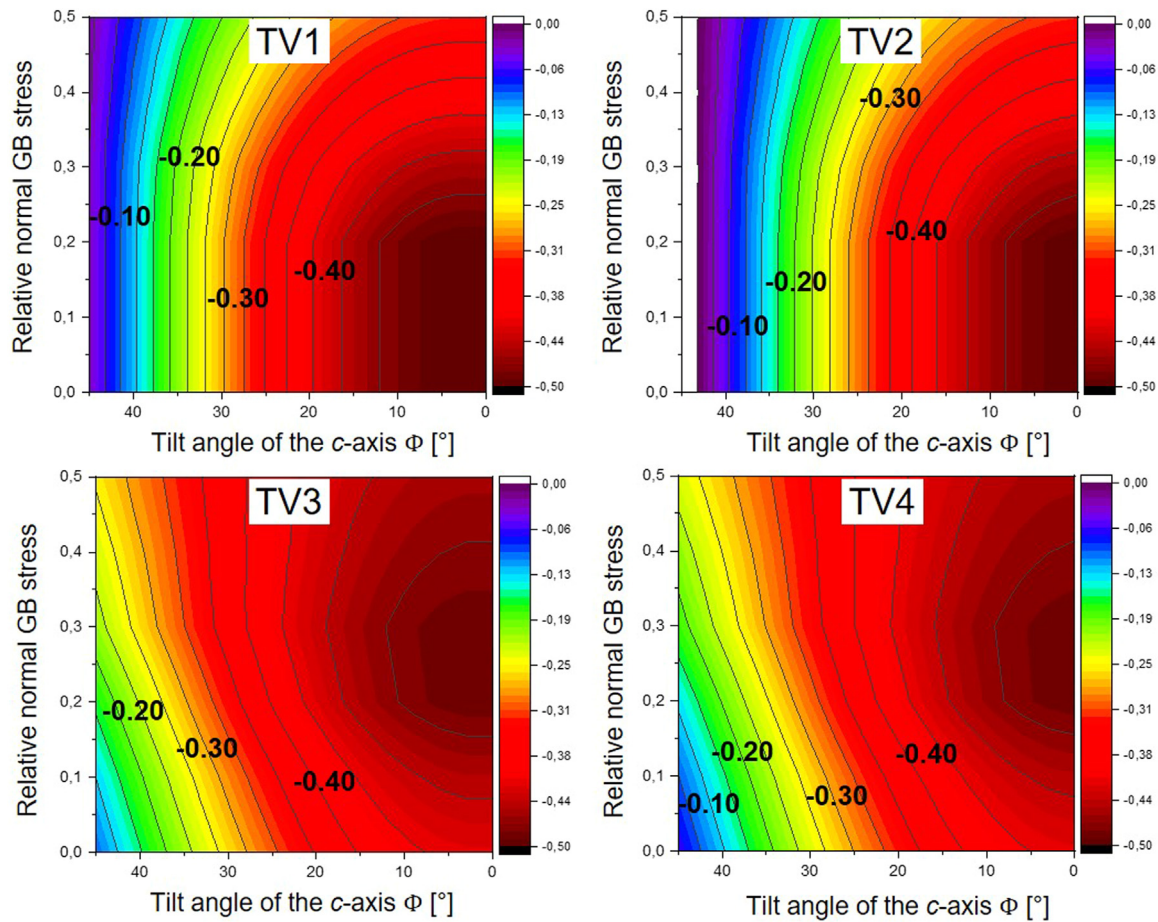


Fig. 10. Estimation of the SF variation for the observed twin variants as a function of the tilt angle Φ and normal GB stress relative to the imposed compressive stress in CD.

net twinning shear with respect to the geometrical constraints. This is explained in Fig. 12 by considering the additive and subtractive shear components of different twin pairs in order to identify the ones that can best comply with the imposed deformation. It is noted that the twinning shear around a shared twin/matrix rotation axis can be of a positive (p) or a negative (n) sign depending on the definition of the coordinate system. Hence, the combined shear of pairwise twinning can have 4 different scenarios, i.e. p - p, n - n, p - n, n - p. From symmetrical perspectives, it is unnecessary to plot all four scenarios, which is why Fig. 12 only considers the additive (p - p or n - n) and subtractive (p - n or n - p) combinations.

As evident in Fig. 12a, the subtractive combination of twin pairs 1 and 2 was favourably positioned in the CD-LD plane, whereby the magnitude of the shear component in CD decreased gradually with increasing lattice rotation indicating a very high chance for this twin pair to form at later stages of deformation. An ideal LD alignment is obtained at $\Phi \sim 1^\circ$ that was observed experimentally at large strains in the vicinity of the GB, where twin variants 1 and 2 formed in pairs (cf. Fig. 5b, c). Other twin pair combinations in Fig. 12a possessed unfavourable resolved shear components in TD or CD

in the initial state and over the course of deformation, which rendered their formation unlikely.

The additive combination of twin pair 3 and 4 in Fig. 12b suggested a higher probability for nucleation in the beginning of deformation because with progressing lattice rotation their additive component rotates towards the CD. Unlike the twin pairs 1 and 2, these twins were much smaller in size and their existence seemed to be strictly localized, i.e. within bands of local orientation gradients (cf. Fig. 6). As illustrated in Fig. 6b, these bands were bound by LABs with misorientation angles smaller than 4° , which were generated in connection with dislocation pile-ups on the basal planes. Atomistic simulations [30] and statistical EBSD investigations [5] on pure Mg suggested easier twin nucleation at low angle GBs compared to high angle GBs. With the inhomogeneous basal slip activity evidenced by the large spread of lattice rotations inside the bands, nucleation of twin pairs 3 and 4 seemed to be a consequence of local strain accommodation. As a result, the growth of these twins was confined to the area within the band (cf. area B in Fig. 6c), and accommodated nearly the same amount of shear as the neighbouring bands, which only underwent basal slip. The parent crystallite preserved their initial orientation be-

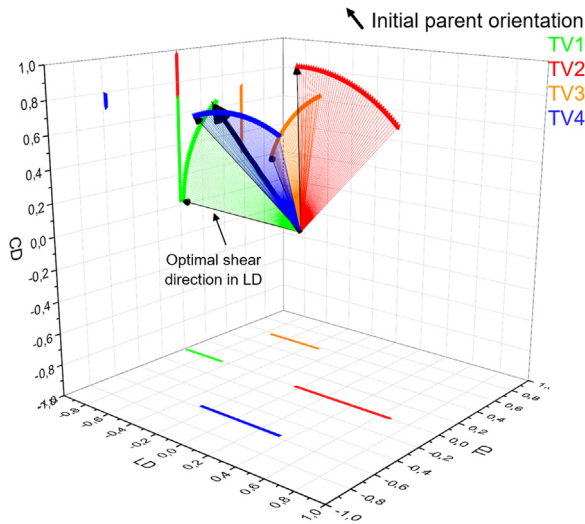


Fig. 11. Resulting shear directions of all four twin variants with respect to the sample coordinate system. The displayed magnitudes on the LD-TD and CD-TD planes represent the range of the respective shear components resulting from the reduction of Φ by the slip-induced lattice rotation up to a maximum of 45° around TD.

fore the twinning incident, which is why such areas depicted lower lattice rotations than surrounding areas sheared by basal slip.

5. Summary

Several bicrystalline Mg samples with symmetric $90^\circ \langle 11\bar{2}0 \rangle$ tilt grain boundaries were subjected to plain strain compression in a channel-die at room temperature up to specific strain values. From the systematic analyses in terms of optical microscopy and panoramic orientation imaging maps the following main results can be summarized:

1. Due to the initial orientation of the grains and the consequent activation of basal slip, the c-axes rotated around

the transverse direction towards the compression direction, similar to what is known from plasticity studies on corresponding single crystals deformed in a channel-die.

2. The presence of the GB led to an additional stress component in the longitudinal direction and therefore to additional strain, which caused an enhanced lattice rotation in its vicinity compared to the grain interior. This lattice rotation took place in a non-uniform deformation zone and expanded from the upper part of the grain boundary in the CD-LD plane with increasing strain. In the immediate vicinity of the grain boundary, the tilt angle of the c-axis decreased from an initial 45° to $\sim 0^\circ$, where the Schmid factor for basal slip approaches zero. As a result, sample cracking was initiated at the GB.
3. At certain strains (e.g. $\varepsilon_t = \sim -15\%$), the lattice rotation in the GB region starts to show a non-uniform distribution along CD. The upper part of the GB exhibited an unexpected decrease of the lattice rotation, which stands in contrast to the process described in the previous conclusion. It is hypothesized that the structure of that GB segment corresponds to a minimum energy compression twin boundary ($64^\circ \langle 1\bar{2}10 \rangle$) in accordance with results of MD simulations from the literature. As a consequence, the repulsive force of that GB segment towards gliding dislocations is comparatively higher than the repulsive force in the bottom GB part. This leads to a lower dislocation density in front of the upper GB region (i.e. lower lattice rotation) compared to the other GB regions.
4. Extension twinning was observed as an additional deformation mechanism. Four different variants of extension twins were observed despite having negative Schmid factors, which questions their nucleation. The simultaneous and pairwise formation of two variants in the vicinity of each other led to an investigation of their resulting combined shear components. The alignment of the resulting shear along the longitudinal direction justified their formation.

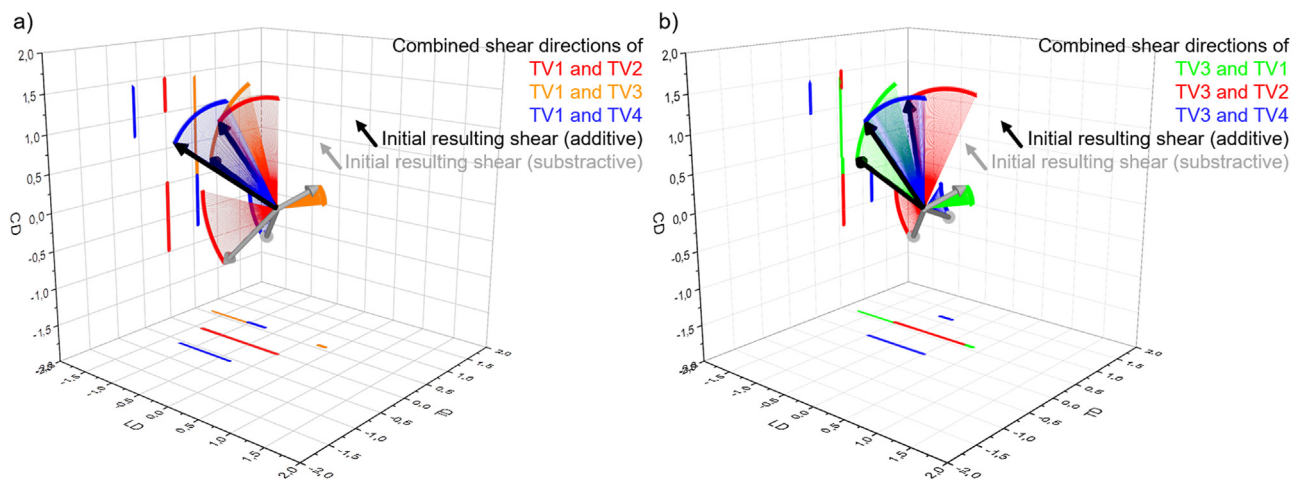


Fig. 12. Resulting shear directions with respect to the sample coordinate system as a result of combined twinning shear of different twin pairs involving twin variant 1 (a) and twin variant 3 (b). The displayed magnitudes on the LD-TD and CD-TD planes represent the range of the respective combined shear components resulting from the reduction of Φ similar to Fig. 11.

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