# Accepted Manuscript

Mechanisms of strain accommodation in plastically-deformed zircon under simple shear deformation conditions during amphibolite-facies metamorphism

Elizaveta Kovaleva, Urs Klötzli, John Wheeler, Gerlinde Habler

PII: S0191-8141(17)30272-9

DOI: 10.1016/j.jsg.2017.11.015

Reference: SG 3562

To appear in: Journal of Structural Geology

Received Date: 17 March 2017

Revised Date: 21 November 2017

Accepted Date: 28 November 2017

Please cite this article as: Kovaleva, E., Klötzli, U., Wheeler, J., Habler, G., Mechanisms of strain accommodation in plastically-deformed zircon under simple shear deformation conditions during amphibolite-facies metamorphism, *Journal of Structural Geology* (2017), doi: 10.1016/j.jsg.2017.11.015.

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.





1	Mechanisms of strain accommodation in plastically-deformed zircon under simple shear						
2	deformation conditions during amphibolite-facies metamorphism						
3	Elizaveta Kovaleva <sup>*1,2</sup> , Urs Klötzli <sup>2</sup> , John Wheeler <sup>3</sup> , Gerlinde Habler <sup>2</sup>						
4	<sup>1</sup> Department of Geology, University of the Free State, 205 Nelson Mandela Drive, 9300						
5	Bloemfontein, Free State, South Africa						
6	<sup>2</sup> Department of Lithospheric Research, Faculty of Earth Sciences, Geography and						
7	Astronomy, University of Vienna, Althanstrasse 14, A-1090 Vienna, Austria						
8	<sup>3</sup> Department of Earth, Ocean and Ecological Sciences, School of Environmental Sciences,						
9	University of Liverpool, L69 3GP, Liverpool, United Kingdom						
10							
11	* Corresponding author.						
12	E-mail: kovalevae@ufs.ac.za; +27- (0)51 4019257						
13							
14	Abstract						
15	This study documents the strain accommodation mechanisms in zircon under amphibolite-						
16	facies metamorphic conditions in simple shear. Microstructural data from undeformed, fractured						
17	and crystal-plastically deformed zircon crystals are described in the context of the host shear						
18	zone, and evaluated in the light of zircon elastic anisotropy. Our work challenges the existing						
19	model of zircon evolution and shows previously undescribed rheological characteristics for this						
20	important accessory mineral.						

21 Crystal-plastically deformed zircon grains have <c> axis oriented parallel to the foliation 22 plane, with the majority of deformed grains having <c> axis parallel to the lineation. Zircon 23 accommodates strain by a network of stepped low-angle boundaries, formed by switching between tilt dislocations with the slip systems  $<100>\{010\}$  and  $<\overline{1}$  10> $\{110\}$  and rotation axis 24 [001], twist dislocations with the rotation axis [001], and tilt dislocations with the slip system 25  $<100>\{001\}$  and rotation axis [010]. The slip system  $<\overline{1}$  10>{110} is newly described for 26 zircon. Most misorientation axes in plastically-deformed zircon grains are parallel to the XY 27 plane of the sample and have [001] crystallographic direction. Such behaviour of strained zircon 28 29 lattice is caused by elastic anisotropy that has a direct geometric control on the rheology, 30 deformation mechanisms and dominant slip systems in zircon. Young's modulus and P wave 31 velocity have highest values parallel to zircon [001] axis, indicating that zircon is elastically 32 strong along this direction. Poisson ratio and Shear modulus demonstrate that zircon is also most 33 resistant to shearing along [001]. Thus, [001] axis is the most common rotation axis in zircon.

34 Such zircon behaviour is important to take into account in structural and geochronological 35 investigations of (poly)metamorphic terrains. Geometry of dislocations in zircon may help 36 reconstructing the geometry of the host shear zone(s), large-scale stresses in the crust, and, 37 possibly, the timing of deformation, if the isotopic systems of deformed zircon were reset.

- 38
- 39 Keywords

40 Zircon, elastic anisotropy, strain accommodation, slip, crystal-plastic deformation

41

42 **1. Introduction** 

43 Zircon is a very common accessory minerals and it is widely used in geochronology (e.g. 44 Davis et al., 2003). Consequently, the possible effects of the deformation of zircon are important 45 to consider for isotopic analyses and mineral thermometry measurements, because it can 46 dramatically affect the outcomes of these analyses. Crystal-plastic microstructures, such as 47 dislocations and low-angle boundaries, can cause re-distribution of trace elements and their isotopes, including isotopes of U, Th, Pb, Ti and Y (e.g. Reddy et al., 2006, 2009; Moser et al., 48 49 2009, 2011; Timms and Reddy, 2009; Flowers et al., 2010; Reddy and Timms, 2010; Piazolo et 50 al., 2012, 2016; MacDonald el al., 2013; Bellucci et al., 2016; Peterman et al., 2016; Kovaleva et 51 al., 2017). For example, loss of Pb radiogenic isotopes and gain of Th and U can result in much 52 younger isotopic age of deformed zircon than its formation age (e.g. Timms et al., 2006; Moser 53 et al., 2009; MacDonald et al., 2013), and gain or loss of Ti through the dislocations can cause senseless results for Ti-in-zircon thermometry (e.g. Timms et al., 2011). 54

55 Crystal-plastically deformed zircon crystals are often observed in shear zones, and thus ductile deformation of zircon is attributed to the metamorphism and deformation of the host rock 56 (e.g. Kaczmarek et al., 2011; Piazolo et al., 2012; Kovaleva et al., 2014, 2016). During these 57 58 deformation event(s), zircon grains accumulate strain through elastic deformation, fracturing or 59 formation of geometrically necessary dislocations (GNDs) and their subsequent migration (creep) into lower energy configurations (e.g. Hobbs, 1968; 1985; White, 1973; Poirier and 60 61 Nicolas, 1975; Drury and Urai, 1990; Poirier, 1985). The geometry of GNDs in zircon can be determined with three methods: 62

63 64

65

(1) Transmitted electron microscopy (TEM) of focussed ion beam (FIB) foils (e.g. Leroux et al., 1999), which is the most precise, but also time-consuming and expensive method, requiring specific equipment.

- (2) In an absence of TEM, GNDs can be assumed from the electron backscatter diffraction
  (EBSD) maps using Weighted Burgers Vector (WBV) calculations (e.g. Wheeler et al.,
  2009; 2012; MacDonald et al., 2013), which is especially useful when no low-angle
  boundaries are present. Such method, however, does not determine dislocations, which
  Burger's vector is parallel to the mapped surface. This method requires a special
  software (see Methodology section).
- (3) Where well-developed low-angle boundaries are present, GND's geometry can be
  inferred from boundary geometry relative to the crystallographic geometry of the host
  grain ("boundary trace orientation analyses", e.g. Reddy et al., 2007). This method is
  the simplest of all three, fairly reliable and requires obtaining EBSD maps only.
- 76 In our contribution we make use of methods (2) and (3).
- The resulting geometry of GNDs and their slip systems in zircon would be determined by (a) elastic anisotropy of zircon (e.g. Reddy et al., 2007) and (b) crystallographic orientation of the zircon in the shear zone (e.g. Kaczmarek et al., 2011; Kovaleva et al., 2016).
- Elastic anisotropy properties of zircon under low temperature (≤ 300 °C) are generally
  well-constrained (e.g. Özkan and Jamieson, 1978; Bass et al., 1995; den Toonder et al., 1999).
  However, analysis of zircon deformation as a function of its elastic anisotropy at greater
  temperatures is not as well developed (e.g. Reddy et al., 2007; Timms et al., 2012).
  Consequently, the temperature dependence of elastic properties for zircon is also unknown.
- Among rock-forming minerals, olivine, quartz, plagioclase and calcite deformation properties are the most studied in regard to their crystallographic orientation and elastic anisotropy (e.g. White, 1973; Hansen and Warren, 2015; Kruse et al., 2001; Bestmann and Prior,

88 2003; Menegon et al., 2011; Prior et al., 2011 and references therein). The more limited studies 89 of zircon deformation evolution in shear zones include Kaczmarek et al. (2011), which was 90 recently updated by Kovaleva et al. (2016). These studies evaluated the orientation of 91 misorientation axes in crystal-plastically deformed zircon with respect to a sample vorticity axes. 92 In particular, Kovaleva et al. (2016) demonstrated that crystal-plastically deformed zircon grains 93 have <c> axes roughly parallel to the foliation plane of the sample, whereas undeformed grains 94 have their <c> axes at a high angle to the foliation plane. The modelling (Kaczmarek et al., 95 2011) and empirical observations (Kovaleva et al., 2016) did not describe geometries of dislocations and other microstructures with regard to zircon elastic anisotropy, nor did they 96 97 discuss specific mechanisms of strain accommodation in zircon at certain conditions. To our knowledge, there are no studies that correlate grain orientation in a shear zone, dislocation 98 99 orientations in deformed zircon crystals and zircon anisotropy properties.

100 The present study focuses on one sample from the dataset presented in Kovaleva et al. 101 (2016), and describes in detail microstructures in one population of zircon grains from a highly-102 strained meta-lamprophyric dyke (Kovaleva et al., 2014, 2016). Within the studied population, 103 we made EBSD maps of 16 grains, 13 of which are crystal-plastically deformed, and additional 104 orientation contrast images of 15 grains. In total, 31 zircon grains were microstructurally analysed. These analyses let us characterize crystal-plastic deformation of zircon under 105 amphibolite-facies conditions. We use these outcomes to further describe zircon deformation 106 107 behaviour, identify a new slip system, and relate intragranular deformation to the elastic 108 anisotropy of zircon in the context of a shear zone deforming in these metamorphic conditions.

109

#### 110 **2.** Geological setting of the sampling locality

111 The sample of this study is from a strike-slip shear zone in the "Zillertaler Kern" of the 112 Western Tauern Window, Eastern Alps (Zillertal, Tyrol, Austria). The Eastern and Western 113 Tauern Windows expose the footwall of the Austroalpine nappe system, composed here of 114 continental and oceanic rocks of Penninic and sub-Penninic nappe sequences. Nappe stacking 115 and regional metamorphism are related to the closure of the Alpine Neotethys and subsequent 116 continental collision during early Tertiary (Miller et al., 2007; Veselá et al., 2011).

117 The detail description of the sampling locality, documented shear zone and analysed 118 sample are presented in Kovaleva et al. (2016), their Figures 1 and 4A-B. Most importantly, the 119 shear zone experienced sinistral simple shear during an Alpine tectono-metamorphic event under 120 amphibolite-facies conditions of 0.5–0.7 GPa and 550–600 °C, and the thermal peak occurred at 121 ca. 30 Ma (Selverstone et al., 1991; Pennacchioni and Mancktelow, 2007). Subsequently, the 122 zone experienced retrograde metamorphism under greenschist-facies conditions during rapid exhumation of the unit at ~ 20 Ma (e.g. Selverstone, 1988, 1991; Pennacchioni and Mancktelow, 123 124 2007).

125

#### 126 3. Methodology and data representation

127 3.1. Sample preparation

Two thin section slabs were cut normal to the XY plane and at 45° to the XZ plane. Such cut orientations were used to enable characterization of all elements of the microstructural population in these plastically-deformed zircon crystals (Supplemental Fig. 1). For example, thin section slabs cut parallel to the lineation (classic thin section cut), are less useful because they will not reveal microstructures and subgrain walls that are normal to the vorticity axis of the

sample (e.g. Kaczmarek et al., 2011). The thin sections were mechanically polished with 0.25
µm diamond paste and chemically polished on a rotating disc with the colloidal silica solution
Köstrosol 3530.

136

#### 137 3.2. Scanning electron microscopy

Backscattered electron (BSE) images were collected with the FEI Inspect S scanning electron microscope (SEM). Electron backscatter diffraction (EBSD) maps and orientation contrast images of zircon grains were acquired with a FEI Quanta 3D field emission gun (FEG) SEM. Both instruments are located at the Faculty of Earth Sciences, Geography and Astronomy, University of Vienna, Austria. Details of analytical settings are presented in Kovaleva et al. (2014, 2016).

144

#### 145 3.3. EBSD and elastic anisotropy data representation

146 The EBSD data are presented as: (1) EBSD pattern quality maps, showing the quality of 147 the collected Kikuchi diffraction patterns, (2) colour-coded cumulative misorientation maps, showing relative misorientation of each pixel to a user-selected reference point within the crystal, 148 149 or (3) local misorientation EBSD maps, where each pixel is color-coded according to the mean 150 misorientation of each data pixel relative to its six neighbouring pixels. During processing and 151 plotting of the data, no noise reduction or smoothing was applied due to the very high quality of 152 the dataset (99.99% of pixel indexing). The pole figures with orientation of crystallographic axes 153 are plotted in the lower hemisphere equal-area projections. Cumulative EBSD maps, EBSD 154 pattern quality images and pole figures were produced with the EDAX OIM v6.2.1 Analysis

software. The foliation plane in all pole figures is oriented horizontally. The local misorientation
EBSD map and the visualization of density contours of misorientation axes orientation in the
inverse pole figures were produced using the MATLAB MTEX toolbox (Bachmann et al., 2010,
2011; Mainprice et al., 2011). Elastic properties of zircon are visualised with MATLAB MTEX
toolbox using the elastic constraints of Özkan and Jamieson (1978), Bass et al. (1995) and den
Toonder et al. (1999).

161

# 162 3.4. Weighted Burgers Vector (WBV) calculations

We gained insight into geometrically necessary dislocation densities using WBV 163 164 calculations (Wheeler et al., 2009; 2012). WBV quantifies the total Burgers vector for all the 165 dislocations passing through the user-selected rectangular region in the EBSD map (the "integral 166 form", according to Wheeler et al., 2009). This value can be expressed in terms of lattice vectors and then divided by the sample region area to measure dislocation density including Burgers 167 168 vector direction. The components of summary WBV across the user-selected rectangular areas 169 are expressed by three numbers: a, b and c, which are listed for each selected subarea and are measured in  $(\mu m)^{-2}$ . Rectangular areas with WBV components were calculated using EBSD 170 171 maps with the MATLAB toolbox CrystalScape 1.3 based on the method described in Wheeler et 172 al. (2009, 2012). For this calculation, the maps were transformed to a rectangular grid and the Euler angles were recalculated with the Channel software. For better visual representation, the 173 174 rectangular areas were superimposed on top of a hexagonal "local misorientation" EBSD map.

The WBV can be calculated for any crystal system, but the method has several limitations. WBV is calculated from orientation gradients in a map view, i.e. it only documents dislocations that intersect a 2D surface of the map. Therefore, all dislocations that are parallel to the map are

178 not analysed by the software, and no assumptions are made about gradients in the third 179 dimension, which may be non-zero (see Wheeler et al., 2009, 2012). However, our sampling 180 geometry let us document dislocations that lie in the XZ plane (see section 3.1).

Calculation of the WBV involves no assumptions about minimization of dislocation energy. The only assumption involved in the calculation is that elastic strains are small, so that the lattice distortion is entirely due to dislocations (Wheeler et al., 2012). Therefore, we assume that a non-homogeneous distribution of WBV values in analysed zircon grains are caused by stress-related dislocations.

186

# 187 **4.** Elastic anisotropy of zircon and its implications

The elastic anisotropy of zircon can be visualized by constraining Young's modulus and P-188 189 wave velocity contours (Fig. 1A-B). Large values for Young's modulus and P-wave velocity 190 parameters are parallel to zircon {100} planes with the greatest values in the basal plane {001} 191 and along the crystallographic directions <100> and [001]. High P-wave velocity values along 192 the main crystallographic planes (Fig. 1B) indicate that these planes are the most densely 193 occupied by atoms. At the same time, high Young's modulus correspond to crystallographic 194 directions where the lattice is elastically stronger. Preferred slip in crystal lattices is expected to 195 occur parallel to the crystallographic direction with the most closely spaced atoms, so that the 196 Burgers vector has the shortest possible length (e.g. Poirier, 1985). In zircon, these directions are 197 [100] and [001] (Fig. 1). Consistently, <100> was experimentally shown to be the energeticallypreferable orientation of the Burgers vector, as the shortest translation vector in the zircon 198 199 structure (Leroux et al., 1999). Therefore, slip or fracturing will more likely occur in zircon 200 along these low indices planes. Thus, elastic anisotropy of zircon suggests preferable activation

of slip systems <100>{010}, <010>{001} and <001>{010}. The densest plane is the basal plane
{001}, and, consistently, deformed grains most often have a rotation axis parallel to [001] (e.g.
Reddy et al., 2007; Kaczmarek et al., 2011; Kovaleva et al., 2016).

These properties are confirmed by the shape of Poisson ratio and Shear modulus contours (Fig. 1C-D). Poisson ratio, being greatest parallel to [001] and the smallest parallel to [100] (Fig. 1C), indicates that the strain along [001] always exceeds strain along [100]. This property defines the energetically-preferable rotation axis in zircon, which is [001]. Shear modulus is maximum parallel to [001], showing that zircon is most resistant to shearing along this axis, and thus confirming that this axis is the most preferable for a rotation axis.

This elastic anisotropy for Young's modulus and Shear modulus of zircon was demonstrated before by Timms et al. (2012). However, the Young's modulus anisotropy shape presented in Timms et al. (2012) reflect properties of metamict zircon. Their image, therefore, does not characterize the non-metamict zircon populations, such as the population presented in this study. Our population is characterized by a high crystallinity degree, reflected by Raman spectroscopy analyses (Kovaleva et al., 2014) and by high EBSD pattern quality images of all analysed grains (see microstructural data).

217

#### 218 **5.** Sample description

The studied sample is a metamorphosed, strained and isoclinally-folded lamprophyre (see Kovaleva et al., 2016). Sample is composed of amphibolite-facies mineral assemblage, including plagioclase, biotite, titanite and quartz (Fig. 2A-B). Biotite and titanite form layers 1–6 mm thick, which alternate with mylonitized plagioclase–quartz layers with similar thickness (Figs.

223 2A-B, Supplemental Fig. 1). Biotite is chloritized as a result of retrograde greenschist-facies 224 metamorphism, which occurred during exhumation of the unit (Fig. 2C). The accessory minerals 225 are apatite, zircon, calcite, pyrite, and rutile (Fig. 2C-D). The foliation plane of the sample is 226 formed by biotite-rich layers and is used here as the kinematic reference plane (Bestmann and 227 Prior, 2003).

228 Zircon in the sample is present as small (10-30 µm) mostly euhedral grains, slightly 229 elongate to the aspect ratio of up to 1:3 (Figs. 2C-D, 3-6). Zircon grains are enclosed in 230 chloritized biotite (Fig. 2C-D), plagioclase, titanite or quartz (Table 1). The observed zircon 231 population was initially magmatic in origin, but in some crystals, we observe metamorphic rims 232 that grew on magmatic oscillatory-zoned cores (for CL images see Supplemental Fig. 2), and formed coevally with the host biotite grains, presumably, during peak amphibolite-facies 233 234 metamorphism. Some zircon grains were subsequently fractured (Fig. 4) or crystal-plastically 235 deformed (Figs. 5-6). Crystal-plastic deformation in zircon presumably occurred during 236 amphibolite-facies Alpine metamorphism (30 Ma), however, the overprint of the greenschistfacies deformation (30-20 Ma) might have also taken place and influenced zircon deformation 237 238 patterns. This overprint is a potential complication in our study. However, zircon deformation 239 patterns are similar for amphibolite and upper-greenschist-facies conditions and are difficult to 240 distinguish. Moreover, in our case, the transition between two regimes was gradual, thus, we 241 assume that the potential greenschist-facies metamorphic overprint, if it happened, did not change the overall mode of zircon deformation. The population of zircons displays little radiation 242 243 damage and is characterized by a high crystallinity degree, documented by high-quality EBSD 244 patterns (Figs. 3; 5; 6C).

245

246

#### 6. Microstructural and crystallographic zircon data

247 For this study, we have microstructurally investigated total of 31 zircon crystals (see Table 248 1). Zircon grains were decoupled from the host matrix (Kovaleva et al., 2014). Absence of 249 coating, dragging of the surrounding biotite, open interface voids and pressure shadows provide 250 evidence of zircon-matrix decoupling (e.g. Figs. 3A-B; 4A; 6C), which enables inhomogeneous 251 stress distribution within zircon grains, especially when hosted by the rheologically softer 252 material (e.g. see numerical modelling by Schmid and Podladchikov, 2005) and may lead to their 253 crystal-plastic deformation (Kenkmann, 2000). The decoupling also indicates that post-254 deformational rigid-body rotation was absent, preserving the syn-deformation orientation of the 255 zircon grains in the shear zone (see Kovaleva et al., 2016). Zircon grains from the sampled meta-256 lamprophyre are unstrained (e.g. Fig. 3), fractured (e.g. Fig. 4), or crystal-plastically deformed (e.g. Figs. 5-6), where about 45% of the zircon grains are crystal-plastically deformed (Table 1). 257 258 The studied zircon population does not display crystallographic preferred orientation (e.g. Fig. 259 7A, Table 1).

260 Undeformed (e.g. Fig. 3) and fractured (e.g. Fig. 4) zircon crystals have <c> axes that are oriented at a high angle ( $\geq 15^{\circ}$ ) to the foliation plane XY, otherwise stretching in all other 261 262 directions (e.g. Fig. 7A, open circles; Table 1). Within the fractured grains, fractures are 263 generally subparallel to {100} crystallographic planes (e.g. Fig. 4A-B). Long axis <c> of all 264 plastically-deformed crystals are parallel or subparallel to the foliation plane (e.g. Figs. 5; 6B; 7A, solid circles; Table 1). Moreover, orientations of <c> axis of ~ 55% of analysed crystal-265 266 plastically deformed grains are parallel or subparallel to the stretching lineation with their <c> 267 (Figs. 5A; 6B, grains 03 and 04; Table 1). The rest of the plastically-deformed grains are

oriented with  $\langle c \rangle$  axis either at ~ 45-55° to the lineation (e.g. Fig. 5B, grain 15; Table 1), or at ~ 90° to the lineation (e.g. Fig. 5C, grain 24; Table 1).

270 The finite deformation pattern in plastically-deformed zircon grains is characterized by the 271 presence of the strain-free subgrains that do not share lattice geometry orientations (e.g. Fig. 6A). 272 This type of zircon deformation corresponds to distortion pattern type III (Piazolo et al., 2012). 273 Low-angle boundary traces, which separate subgrains in the sampled zircon grains, reveal a 274 continuous network of stepped lines, and, as a result, subgrains form irregular-shaped domains 275 from 1 to 10 µm in diameter (Figs. 5; 6A). In rare cases, the low-angle boundaries are rounded or 276 curved, forming oval-shaped subgrains (e.g. Table 1).

277 Misorientation axes of plastically-deformed grains are mostly parallel to the XY (foliation) 278 plane, and sometimes are parallel to the YZ plane (Fig. 7B) for grains with comparatively low 279 degree of strain (Table 1). In cases, where misorientation axes are parallel or subparallel to Y 280 direction, they lie simultaneously in XY and YZ planes. Most intensely-strained grains have 281 misorientation axes distributed around X, the direction of stretching lineation.

282 Most crystal-plastically deformed zircon grains (~77%) have misorientation axes clustering 283 around the [001] crystallographic direction (Figs. 5; 6D; Table 1). Other misorietnation axes 284 directions are <212>, <311> or close to these (such as <310>, <201> and <321>, see Table 1), 285 but these directions are usually less significant and form minor clusters. For example, the 286 misorientation axes of grain 24 are dispersed in the inversed pole figure, forming one strong 287 cluster parallel to [001] and a few smaller maxima about <212> <321> and <110>. In grain 15, 288 the misorientation axes cluster around [001] and [100] (Fig. 5B). Consistently, crystallographic axes in the pole figures for grains 15 and 24 are dispersed around multiple axes (Fig. 5A, C), 289 290 which points to strain acting in localized parts of the grain with different slip systems, so-called

"asterism" of crystallographic axes (e.g. Moser et al., 2009). In contrast, grains 03 and 04 reveal rotation of the crystallographic axes around the [001] direction in the pole figures and only one cluster of misorientation axes (Figs. 5A; 6B, arrows). This suggests that plastically-deformed grains with the orientation of <c> close to the stretching lineation, develop only one misorientation axis, which is usually parallel to [001]. The larger is the angle between <c> axis and the lineation, the more clusters of misorientation axes are observed (see Table 1).

If the main host phase for zircon is biotite, or chlorite as a result of biotite alteration, then a high probability of activation of misorientation axis [001] is observed (Table 1). Therefore, there also seem to be a strong control of the host phase's rheology on zircon deformation (e.g. Kenkmann et al. 2000; Kovaleva et al., 2014; 2016). Rheologically softer host phases allow for the activation of the preferable rotation axis.

302

# 303 6.1. Analyses of the slip systems geometry

304 Geometrically necessary dislocations (GNDs) form in the crystal lattice to accommodate 305 lattice strain. During strain recovery, GNDs may be clustered by dislocation creep, forming 306 dislocation walls or low-angle boundaries. Therefore, the presence of low-angle boundaries in 307 plastically-deformed grains provides an opportunity to identify dislocation slip system(s) that 308 contributed to boundary formation. The geometry of low-angle boundaries reflects the slip-309 system geometry that can be reconstructed (e.g. Reddy et al., 2007; Kaczmarek et al., 2011; 310 Timms et al., 2012). However, we should emphasize that the two-dimensional boundary traces 311 may not always unequivocally identify the slip system(s) related to boundary formation.

312 In this contribution, we focus on detailed analyses of four crystal-plastically deformed 313 grains, representative for our sample, as they reveal the most typical microstructures for this 314 population. The amount of strain within these grains is comparatively high for the population. 315 Therefore, it is easier to provide reliable geometry analyses of low-angle boundaries and GNDs. 316 Additionally, we present microstructural orientation data on nine more crystal-plastically 317 deformed grains from this sample (Fig. 7; Table 1), which show similar behavior. We provide boundary orientation trace analyses for grains 15, 24 (Fig. 5B-C) and 04 (Fig. 6B) and WBV 318 319 analyses for grain 04 (Fig. 6A), in order to demonstrate that these two methods are in good 320 agreement with each other.

321 Grain 03 is highly strained, compared to the other zircon grains in the population, and has a dense network of low-angle boundaries (Fig. 5A, left panel, pointed by arrows) with an average 322 323 of four to five along any normal sampling line of about five microns length. The traces of low-324 angle boundaries form irregular, stepped lines with a complicated geometry. Obviously, several 325 interchanging slip systems operated in this grain, switching along low-angle boundaries where 326 they change their direction. All of these slip systems have rotation axes around [001], as 327 indicated by the systematic rotation of the all crystallographic axes about zircon [001] (Fig. 5A, 328 pole figure in the middle panel, arrows). Moreover, an inversed pole figure for this grain shows 329 clustering of the misoritention axes close to [001] (Fig. 5A, right panel). As we have discussed 330 earlier, [001] is the most commonly documented rotation direction (e.g. Leroux et al., 1999; 331 Reddy et al., 2007; Kovaleva et al., 2016) and energetically-preferable rotation axis in zircon, 332 due to elastic and crystallographic anisotropy of non-metamict zircon (Fig. 1; Reddy et al., 333 2007). Usually, subgrain wall, consistent with one dislocation geometry would also have a 334 consistent direction. In this grain, subgrain boundaries reveal complicated geometry: stepped

335	configurations and change of direction over small distances. Therefore, we suggest that subgrain						
336	boundaries in grain 03 (Fig. 5A) represent both tilt and twist ("mixed") walls (e.g. see boundary						
337	trace analyses in Reddy et al., 2007).						
338	In grain 15, low-angle boundary traces extend in two general directions (NNW-SSE and						
339	WSW-ENE in the Fig. 5B) and apparently occupy planes (100) and (010) (Fig. 5B, "LAB-1" and						
340	"LAB-2" accordingly). Such geometry of low-angle boundaries, together with the directions of						
341	misorientation axes determined for this grain ([001] and [100]), implies several possibilities for						
342	present slip systems in this grain:						
343	a. If the rotation axis for low-angle boundary 1 is [001], then it is a tilt						
344	boundary with the slip system [100](010).						
345	b. If the rotation axis for low-angle boundary 1 is [100], it is a twist						
346	boundary.						
347	c. If the rotation axis for low-angle boundary 1 is [010], it is a tilt boundary						
348	with the slip system [100](001).						
349	d. If the rotation axis for the low-angle boundary 2 is [001], it is a tilt						
350	boundary with the slip system [010](100).						
351	e. If the rotation axis for the low-angle boundary 2 is [010], it is a twist						
352	boundary.						
353	f. If the rotation axis for the low-angle boundary 2 is [100], it is a tilt						
354	boundary with the slip system [010](001).						
355	Low-angle boundary traces in grain 15 form stepped rather than straight lines, slightly						
356	changing directions over short distances. This low-angle boundary network most likely						
	12						

represents a system of interconnected tilt walls with the slip geometries <100>{010} and <100>{001} and twist walls and may include all possible slip systems a-f, valid for different intervals of a given boundary. Asterism of crystallographic axes is also evidence that there are several slip systems operating at the same time in different grain domains (Moser et al., 2009), indicating spatial heterogeneity of misorientation axes.

362 In grain 24 (Fig. 5C), one of the low-angle boundaries ("LAB-1") is straight and parallel to 363 the (100) crystallographic plane. This geometry for low-angle boundary, together with the 364 misorientation axis parallel to [001], implies the slip system [100]{010} for this boundary, 365 because for the tilt boundary slip direction in normal to it and lies in the normal slip plane, 366 whereas rotation axis is parallel to the boundary and normal to the slip direction. The other lowangle boundary in the lower part of the grain extending from left to right ("LAB-2"), has a 367 368 complicated geometry and most likely represents a result of combination of several slip systems. 369 Minor clusters of misorientation axes around high index directions (Fig. 5C) are either a result of 370 analytical error due to small misorientation angles (e.g. Prior, 1999; Reddy and Buchan, 2005) in 371 this grain, reaching only 3° (see Kovaleva et al., 2016), or evidence of minor slips activated to 372 accommodate strain, connecting major tilt and twist walls that have dispersion around [001]. The 373 second suggestion is also supported by a minor asterism of crystallographic axes in the pole 374 figure (Fig. 5C), which is the evidence of multiple slip systems operating in different small grain 375 domains.

In grain 04, trace of low-angle boundary 1 ("LAB-1", Fig. 6A) is parallel to the (100) plane (Fig. 6B). Misorientation axes in this grain coincide with the dispersion axis and are parallel to [001] (Fig. 6D). Such geometry implies a tilt boundary correlated with the [100]{010} slip system (Fig. 6B, black lines). Low-angle boundary 2 ("LAB-2", Fig. 6A) is apparently parallel to

the (110) plane, which implies a tilt boundary with the slip system  $<\overline{1}$  10>{110} and rotation axis [001] (Fig. 6B, grey lines).

382 In the lower left portion of the crystal 03, the WBV values are small (Fig. 6A, grey solid 383 rectangles), indicating low dislocation density and, therefore, a strain-free domain. In the lower 384 right portion, WBV is dominated by the *a* component (dotted rectangles), which, taking into account rotation axis [001], implies domination of dislocations with the slip system <100>{010}. 385 386 WBV analyses across the low-angle boundary 1 ("LAB-1") are dominated by the b component (dashed rectangles) that, taking into account rotation axis [001], points to the domination of 387 dislocations with slip along the [010] direction (i.e. slip system <010>{100}, geometrically 388 389 equivalent to slip system <100>{010}). The WBV analyses across low-angle boundary 2 ("LAB-2") show high values of the *a* and *b* components, implying the above mentioned slip system < 1390  $10>\{110\}$  with slip along the [1, 10] plane (Fig. 6B), if the rotation axis geometry is [001]. A 391 highly non-homogeneous distribution of WBV values in grain 03 (Fig. 6A) points to 392 393 inhomogeneous distribution of post-growth strain, and indicates that the plastic deformation in 394 this zircon is a post-growth process, caused by a directed external differential stress (e.g. MacDonald et al., 2013). 395

In naturally-deformed crystals, it is difficult to observe pure edge or screw dislocations. Almost all dislocations have both screw and edge components so that they are mixed dislocations (Poirier, 1985). Consistently, summary of our data demonstrate that subgrain walls mostly appear as stepped lines. These walls apparently reflect an interplay between tilt dislocations with the slip systems  $<100>{010}$  and  $<\overline{1}$  10>{110} and rotation axis [001] and twist dislocations with rotation axis [001], and between tilt dislocations with the slip system  $<100>{001}$  and twist

dislocations with rotation axis [010]. Thus, strain under amphibolite-facies conditions in
plastically-deformed zircon grains is accommodated by formation of a continuous network of
low-angle boundaries, which formed by a combination of tilt and twist dislocations with above
mentioned geometries.

406 This slip is easily activated when zircon <c> axis has a specific orientation normal or 407 parallel to the vorticity axis of the shear zone (Figs. 7A; 8i and iii). Selective crystal-plastic 408 deformation of zircon grains that are aligned within the XY plane (Fig. 7A) could be explained 409 by the critically resolved shear stress (CRSS) that is more easily reached along specific planes, 410 when the grain orientation is favourable with respect to a local stress field (Hobbs, 1985). Along 411 the <c> axis, the zircon atomic structure consists of chains of alternating edge-sharing SiO<sub>4</sub> 412 tetrahedra and ZrO<sub>8</sub> dodecahedra that are joined laterally by edge-sharing dodecahedra 413 (Robinson et al., 1971; Finch and Hanchar, 2003). To develop a slip in the zircon crystal lattice, 414 it is energetically preferable to break the bonds between SiO<sub>4</sub> tetrahedra and ZrO<sub>8</sub> dodecahedra 415 and displace them along [100], rather than between the strongly bonded  $ZrO_8$  dodecahedra along [001]. Consistently, the geometry of slip systems  $<100>\{010\}$  with the rotation axis [001] are 416 417 the most frequently observed in zircon (e.g. Leroux, 1999; Reddy et al., 2007; Kovaleva et al., 418 2014), whereas rotation axis [010] and slip systems  $<100>\{001\}$  are less common (e.g. 419 Kaczmarek et al., 2011). This study agrees with previous investigations (Leroux, 1999; Reddy et 420 al., 2007; Timms et al., 2012; Kovaleva et al., 2014; etc.), and shows that slip along  $<100>\{010\}$ 421 with rotation around [001] is the most energetically preferable geometry. This interpretation is 422 also consistent with the elastic anisotropy of zircon, suggesting rotation around [001] as the least elastic direction, which is most resistant to shearing (Fig. 1). Slip system  $\langle \overline{1} | 10 \rangle \{110\}$ , 423

424 observations of which are reported above, is a newly described (Fig. 6B, "LAB-2"), as such slip
425 system geometry was not reported before.

426

427 **7. Discussion** 

428

429 7.1. Influence of lattice orientation and elastic anisotropy on deformation behaviour

During simple shear, the vorticity axis is perpendicular to the XZ plane, parallel to the Y 430 431 axis on the foliation plane and normal to the lineation direction (e.g. schematically shown in Fig. 432 8, after Reddy and Buchan, 2005). The crystallographic orientations of misorientation axes in plastically-deformed zircon grains, depending on lattice orientation in the simple shear 433 434 deformation, were observed in Kovaleva et al. (2016) on a large dataset using 6 different samples 435 from a variety of lithologies and shear zones. Our detailed analyses of zircon from one of these 436 samples confirm the earlier results and provides more detailed and accurate data on zircon deformation behaviour. Zircon behaviour in the shear zone is summarized in Fig. 8. Zircon 437 438 crystals with c-axis oblique to the XY (foliation) plane (>15°) are either undeformed (Fig. 3) or 439 fractured (Figs. 4; 8iv). Grains with  $\langle c \rangle$  oriented at  $\sim 15^{\circ}$  to the foliation may experience a minor 440 amount of crystal-plastic strain, but, at the same time, can also be fractured (Table 1). Zircon crystals with <c> aligned to the XY plane with <c> parallel or normal to the X direction 441 442 (stretching lineation) develop misorientation and dispersion axes parallel to [001] (Figs. 7, grains 443 03, 04, 24, and closely oriented; 8i and iii; Table 1). Grains oriented at 90° usually have 444 additional clusters of misorientation axes. Finally, zircon crystals with <c> aligned in the XY 445 plane at 45° to the X direction reveal two or three misorientation axes, usually orthogonal to each

446 other (e.g. [100] and [001]) in order to accommodate the applied strain (Figs. 5B; 8ii). 447 Misorientation axes in plastically-deformed zircon grains are parallel to the XY plane of a shear 448 zone, where the most strained zircon grains have their misorientation axes parallel to the X 449 direction (Fig. 7B). Less frequently, misorientation axes lie in the YZ plane of a shear zone (Fig. 450 7B). The observed zircon properties are not in agreement with the model of zircon evolution, 451 suggested by Kaczmarek et al. (2011). These authors implied that that in plastically-deformed 452 grains, where the <c> axis is parallel or normal to the vorticity axis, only one slip system is 453 activated. In contrast, we observed that zircon lattice does not accommodate strain by a single 454 slip system. As a three-dimensional object, zircon should accommodate strain by several slip 455 systems, except for some extreme deformation conditions, such as formation of planar low-angle 456 boundaries (i.e. planar deformation bands) during seismic events (e.g. Kovaleva and Klötzli, 2017). Moreover, Kaczmarek et al. (2011) concluded that in all cases, where the zircon <c> axis 457 458 is not parallel or normal to the vorticity axis, zircon would necessarily plastically deform and 459 reveal activation along multiple (two or more) glide systems. In our sample, crystal-plastic deformation in grains with  $\langle c \rangle$  orientated at a high angle (>15°) to the foliation plane is not 460 observed (Figs. 3; 4; 7A; Table 1). This discrepancy in the results might be caused by different 461 462 deformation conditions of the sampled zircon. Finally, Kaczmarek et al. (2011) suggested that 463 "The exact selection of glide-system could be dependant of deformation conditions such as 464 pressure, temperature, and strain rate" [sic], and our observations show that the glide systems 465 depend on the orientation of the zircon crystal in the framework of a host shear zone. Pressure, 466 temperature and strain rate affect the lattice distortion pattern of deformed zircon grains (Piazolo 467 et al., 2012; Kovaleva et al., 2014), but not the glide systems.

468 The observed properties of misorientation axes are important to document in order to 469 understand zircon rheological behaviour and its evolution in the shear zone. Our observation 470 suggest that the orientation and distribution of misorientation axes in deformed zircon is 471 controlled by the host shear zone, and thus can be used to reconstruct the conditions of 472 deformation, such as tectonic framework and directions of the stress field with respect to 473 deformed zircon (Fig. 8). Because we attribute crystal-plastic deformation of zircon to a specific 474 metamorphic/deformation event, we infer the timing and P-T conditions of the event even for 475 detrital and inherited grains. Given that different lattice distortion patterns in zircon are typically restricted to specific stress-strain and metamorphic conditions (e.g. Piazolo et al., 2012; 476 477 Kovaleva et al., 2014), these patterns may represent a snapshot during potentially complex deformation and metamorphic histories for the shear zone development. Our result demonstrate 478 479 that analysing a micro-scale microstructures in zircon and its orientation data, analyses of large-480 scale systems and regional deformation events can be provided. It was recently demonstrated that 481 zircon can act as a mineral chronometer (e.g. Moser et al., 2011; MacDonald et al., 2013; 482 Cavosie et al., 2015; Peterman et al., 2016) and even as mineral thermometer (Timms et al., 483 2017) for large-scale deformation events, provided careful sample selection for analyses. We 484 suggest that zircon can also act as a structural indicator of tectonic conditions that cause zircon 485 deformation. Positions and distribution of misorientation axes and type of lattice distortion in 486 zircon carry important information about the large-scale processes such as formation of host 487 shear zone, conditions of regional metamorphism, and even timing of metamorphism.

488

489 7.2. Implications for zircon geochronology and recommendations

490 Our work is thus important for the zircon microchemistry and geochronology, as it 491 describes crystal-plastic deformation mechanisms, which should be considered making isotopic 492 and geochemical analyses of zircon from the shear zones. A number of authors demonstrated that 493 crystal-plastic deformation can cause zircon isotopic system resetting (e.g. Reddy et al., 2006, 494 2007, 2009; Moser et al., 2009, 2011; Timms and Reddy, 2009; Flowers et al., 2010; Reddy and 495 Timms, 2010; Timms et al., 2011, 2012; Piazolo et al., 2012, 2016; MacDonald el al., 2013; 496 Bellucci et al., 2016; Peterman et al., 2016; Kovaleva and Klötzli, 2017; Kovaleva et al., 2017). 497 For example, Peterman et al. (2016) and Piazolo et al. (2016) found toroid-shaped clusters of 498 radiogenic Pb that are associated with dislocations and mark the timing of 499 metamorphism/deformation. It was suggested that radiogenic Pb can be trapped and moved/re-500 equilibrated by dislocation loops due to increased temperatures associated with metamorphism 501 (Petermant et al., 2016).

502 There were several attempts to date deformation and metamorphic events in the Earth's 503 crust using plastically-deformed zircon. For example, significant (≥650 Ma) rejuvenation of U-504 Pb isotopic ages together with CL distortion was documented by Flowers et al. (2010) in zircon 505 grains deformed under granulite-facies conditions. Piazolo et al. (2012) demonstrated that under 506 amphibolite to granulite-facies metamorphic conditions, zircon grains with low-angle boundaries 507 show significant U, Th, and Th/U ratio increase. Consistently, authors indicated younger zircon 508 ages and disturbed CL signal in grains with low-angle boundaries. Piazolo et al. (2012) related 509 this Pb-loss to the presence of interconnected low-angle boundary network, which was formed 510 by imperfectly arranged screw and edge dislocations and resulted in increased pipe diffusion and 511 partial resetting of the isotopic system. Because the conditions of deformation in our sample are 512 similar, and the distortion pattern is the same as in Piazolo et al. (2012), i.e. an interconnected

513 network of low-angle boundaries, formed by mixed (tilt and screw) dislocations, we may expect 514 similar rejuvenation of plastically-deformed zircon in our sample. Moreover, MacDonald et al. 515 (2013) have documented plastically-deformed zircon grains with Pb-Pb distorted isotopic ages 516 that correspond, within an error, to the regional amphibolite-facies metamorphic event. 517 In that case, crystal-plastic deformation of zircon occurred, as in our sample, under amphibolite-518 facies conditions, and lead to a complete resetting of isotopic ages. With the help of our finding 519 one cannot only attempt dating the deformation event, using certain grains in a shear zone, but 520 also attribute different microstructures to distinct stress-strain fields, which were active at 521 different times during the evolution of the geological units. Mineral dating and interpretations 522 can be done in situ based on the orientation of the zircon grains and their misorientation axes and glide systems within the host shear zone. 523

524

#### 525 Conclusions

526 In this work, we demonstrate mechanisms of strain accommodation in zircon under the 527 amphibolite-facies conditions during the simple shear deformation. We demonstrate that the 528 resulting geometry of dislocations and slip systems in zircon are mainly determined by zircon 529 elastic anisotropy and, at the same time, by crystallographic orientation of the zircon in the shear 530 zone. In particular, zircon deforms plastically if it <c> axis lies parallel to the foliation plane, 531 with the majority of deformed grains having their <c> axes aligned with the lineation. To 532 accommodate strain, zircon develops an interconnected network of stepped low-angle boundaries, formed by tilt dislocations with the slip systems  $<100>\{010\}$  and  $<\overline{1}$  10>{110} 533 534 and rotation axis [001], twist dislocations with rotation axis [001], and tilt dislocations with the slip system  $<100>\{001\}$  and rotation axis [010]. The slip system  $<\overline{1}$  10>{110} with rotation axis [001] has not been previously reported in zircon. Observed slip planes are conditioned by the shape of zircon Young's modulus, which has high values along the low indices planes and maximum value in zircon basal plane. Poisson ratio and Shear modulus have the maximum values parallel to [001], defining the most energetically favourable and thus the most common rotation axis in zircon crystal lattice. Zircon crystals, hosted by rheologically weak phases, also tend to develop [001] rotation axes.

542 Misorientation axes of plastically-deformed zircon grains are, in most cases, oriented 543 parallel to the XY plane of the sample and are often parallel to zircon <c> axis. In a few cases 544 misorientation axes are parallel to YZ plane of a sample, where the strain degree is 545 comparatively low.

546 Our finding is significant for zircon geochronology and can be useful when reconstructing 547 the history and structural elements of polymetamorphic terrains. We demonstrate how micro-548 scale structures can be relevant for studying large-scale and even regional events.

549

#### 550 Acknowledgements

551 This study was funded by the University of Vienna (doctoral school "DOGMA", project 552 IK 052) and the Austrian Science Foundation Fund (FWF): I471-N19, which is part of the DFG-553 FWF funded international research group FOR741-DACH. The authors acknowledge access to 554 the Laboratory for scanning electron microscopy and focused ion beam applications, Faculty of 555 Earth Sciences, Geography and Astronomy at the University of Vienna (Austria). Authors are 556 grateful to Claudia Beybel, Franz Biedermann, Bernhard Grasemann, Sigrid Hrabe, Hugh Rice,

557 Markus Palzer, Nicholas Timms, Claudia Trepmann, and all colleagues of the FOR741 research 558 group for fruitful discussions and comments. Thanks goes to Matthew Huber for his support, and 559 for checking the text for spelling and grammar. Insightful comments and suggestions of the 560 editor William Dunne, reviewer Elena Druguet and anonymous reviewers helped to improve this 561 manuscript greatly.

#### 563 **References**

- Bachmann, F., Hielscher, R., Schaeben, H., 2010, Texture Analysis with MTEX Free and
  Open Source Software Toolbox. Solid State Phenomena 160, 63-68.
- Bachmann, F., Hielscher, R., Schaeben, H., 2011, Grain detection from 2d and 3d EBSD
  data-specification of the MTEX algorithm. Ultramicroscopy 111, 1720-1733.
- Bass, J. D., 1995. Elasticity of minerals, glasses and melts. In: Ahrens, T.J. (Ed.), Mineral
  Physics and Crystallography: a Handbook of Physical Constants. American Geophysical Union,
  Washington DC, 45–63.
- Bellucci, J.J, Whitehouse, M.J., Nemchin, A.A., Snape, J.F., Pidgeon, R.T., Grange, M.,
  Reddy, S.M., Timms, N.E., 2016. A scanning ion imaging investigation into the micron-scale UPb systematics in a complex lunar zircon. Chemical Geology 438, 112-122.
  Bestmann, M., Prior, D.J., 2003. Intragranular dynamic recrystallization in naturally
- 575 deformed calcite marble: diffusion accommodated grain boundary sliding as a result of subgrain
- 576 rotation recrystallization. Journal of Structural Geology 25, 1597–1613.

<sup>562</sup> 

577 Cavosie, A.J., Erickson, T.M., Timms, N.E., Reddy, S.M., Talavera, C., Montalvo, S.D.,
578 Pincus, M.R., Gibbon, R.G., Moser, D., 2015. A terrestrial perspective on using ex situ shocked
579 zircons to date lunar impacts. Geology 43, 999–1002.

Davis, D.W., Williams, I.S., Krogh, T.E., 2003. Historical development of zircon
geochronology. In: Hanchar, J.M., Hoskin, P.W.O. (Eds.), Zircon. Mineralogical Society of
America and Geochemical Society, Reviews in Mineralogy and Geochemistry, 53, Washington
D.C., 145–181.

584 Den Toonder, J.M.J, van Dommelen, J.A.W., Baaijens, F.P.T., 1999. The relation between 585 single crystal elasticity and the effective elastic behaviour of polycrystalline materials: theory, 586 measurement and computation. Modelling and Simulation in Materials Science and Engineering 587 9, 909–928.

588 Drury, M.R., Urai, J.L., 1990. Deformation-related recrystallization processes. 589 Tectonophysics 172, 235-253.

Finch, R.J., Hanchar, J.M., 2003. Structure and Chemistry of Zircon and Zircon-Group
Minerals. In: Hanchar, J. M., Hoskin, P. W. O., eds. Zircon. Mineralogical Society of America
and Geochemical Society, Reviews in Mineralogy and Geochemistry 53, 1-26.

593 Flowers, R.M., Schmitt, A.K., Grove, M., 2010. Decoupling of U–Pb dates from chemical 594 and crystallographic domains in granulite facies zircon. Chemical Geology 270, 20–30.

Hansen, L.N., Warren, J.M., 2015. Quantifying the effect of pyroxene on deformation of
peridotite in a natural shear zone. , Journal of Geophysical Research: Solid Earth 120, 2717–
2738.

- Hobbs, B.E., 1985. The geological significance of microfabric analysis. In: Wenk, H.R.
  (Ed.), Preferred orientation in deformed metal and rocks. An introduction to modern texture
  analysis, Academic Press, Inc., Orlando, Florida, 463–484.
- Hobbs, B.E., 1968. Recrystallization of single crystals of quartz. Tectonophysics 6, 353-401.
- Kaczmarek, M.A., Reddy, S.M., Timms, N.E., 2011. Evolution of zircon deformation
  mechanisms in a shear zone (Lanzo massif, Western-Alps). Lithos 127, 414-426.
- Kenkmann, T., 2000. Processes controlling the shrinkage of porphyroclasts in gabbroic
  shear zones. Journal of Structural Geology 22, 471-487.
- Kovaleva, E., Klötzli, U., 2017. NanoSIMS study of seismically deformed zircon:
  Evidence of Y, Yb, Ce and P re-distribution and resetting of radiogenic Pb. American
  Mineralogist, in press.
- Kovaleva, E., Klötzli, U., Habler, G., Libowitzky, E., 2014. Finite lattice distortion patterns
  in plastically deformed zircon grains. Solid Earth 6, 1799-1861.
- Kovaleva, E., Klötzli, U., Habler, G., 2016. On the geometric relationship between
  deformation microstructures in zircon and the kinematic framework of the shear zone. Lithos
  262, 192-212.
- Kovaleva, E., Klötzli, U., Habler, G., Huet, B., Guan, Y., Rhede, D., 2017. The effect of
  crystal-plastic deformation on isotope and trace element distribution in zircon: Combined BSE,
  CL, EBSD, FEG-EMPA and NanoSIMS study. Chemical Geology 450, 183-198.
- Kruse, R., Stuenitz, H., Kunze, K., 2001. Dynamic recrystallization processes in
  plagioclase porphyroclasts. Journal of Structural Geology, 23, 1781–1802.

620	Leroux, H., Reimold, W.U., Koeberl, C., Hornemann, U., Doukhan, J.C., 1999.						
621	Experimental shock deformation in zircon: a transmission electron microscopic study. Earth and						
622	Planetary Science Letters 169, 291–301.						
623	MacDonald, J.M., Wheeler, J., Harley, S.L., Mariani, E., Goodenough, K.M., Crowley, Q.,						
624	Tatham, D., 2013. Lattice distortion in a zircon population and its effects on trace element						
625	mobility and U-Th-Pb isotope systematics: examples from the Lewisian Gneiss Complex,						
626	northwest Scotland. Contributions to Mineralogy and Petrology 166, 21–41.						
627	Mainprice, D., Hielscher, R., Schaeben, H., 2011, Calculating anisotropic physical						
628	properties from texture data using the MTEX open source package. In: Prior, D.J., Rutter, E.H.,						
629	Tatham, D.J. (Eds.), Deformation Mechanisms, Rheology and Tectonics: Microstructures,						
630	Mechanics and Anisotropy. Geological Society, London, Special Publications 360, 175-192.						
631	Menegon, L., Nasipuri, P., Stünitz, H., Behrens, H., Ravna, E., 2011. Dry and strong quartz						
632	during deformation of the lower crust in the presence of melt. Solid Earth 116, B10,						
633	doi:10.1029/2011JB008371						
634	Miller, C., Konzett, J., Tiepolo, M., Armstrong, R.A., Thöni, M., 2007. Jadeite-gneiss from						
635	the eclogite zone, Tauern Window, Eastern Alps, Austria: metamorphic, geochemical and zircon						
636	record of a sedimentary protholith. Lithos 93, 68-88.						
637	Moser, D.E., Davis, W.J., Reddy, S.M., Flemming, R.L., Hart, R.J., 2009. Zircon U-Pb						
638	strain chronometry reveals deep impact-triggered flow. Earth and Planetary Science Letters 277,						
639	73–79.						

Moser, D. E., Cupelli, C.L., Barker, I.R., Flowers, R.M., Bowman, J.R., Wooden, J., Hart,
J.R., 2011. New zircon shock phenomena and their use for dating and reconstruction of large

642	impact structures revealed by electron nanobeam (EBSD, CL, EDS) and isotopic U-Pb and (U-
643	Th)/He analysis of the Vredefort dome. Canadian Journal of Earth Sciences 48, 117–139.
644	Özkan, H., Jamieson, J.C., 1978. Pressure dependence of the elastic constants of
645	nonmetamict zircon. Physics and Chemistry of Minerals 2, 215-224.
646	Pennacchioni, G., Mancktelow, N.S., 2007. Nucleation and initial growth of a shear zone
647	network within compositionally and structurally heterogeneous granitoids under amphibolite
648	facies conditions. Journal of Structural Geology 29, 1757-1780.
649	Peterman, E.M., Reddy, S.M., Saxey, D.W., Snoeyenbos, D.R., Rickard, W.D.A.,
650	Fougerouse, D., Kylander-Clark, R.A.C., 2016. Nanogeochronology of discordant zircon
651	measured by atom probe microscopy of Pb-enriched dislocation loops. Science Advances 2,

652 e1601318.

653 Piazolo, S., Austrheim, H., Whitehouse, M., 2012. Brittle-ductile microfabrics in naturally 654 deformed zircon: Deformation mechanisms and consequences for U-Pb dating. American Mineralogist 97, 1544–156. 655

656 Piazolo, S., La Fontaine, A., Trimby, P., Harley, S., Yang, L., Armstrong, R., Cairney, 657 J.M., 2016. Deformation-induced trace element redistribution in zircon revealed using atom probe tomography. Nature Communications 7, 10490. 658

659 Poirier, J. P., 1985. Creep of Crystals: High-Temperature Deformation Processes in Metals, 660 Ceramics and Minerals. Cambridge University Press, New York.

661 Poirier, J.P., Nicolas, A., 1975. Deformation-induced recrystallization by progressive 662 misorientation of subgrain-boundaries, with special reference to mantle peridotites. Journal of Geology 83, 707-720. 663

30

Prior, D.J., 1999. Problems in determining the orientation of crystal misorientation axes for
small angular misorientations, using electron backscatter diffraction in the SEM, Journal of
Microscopy 195, 217-225.

Prior, D.J., Rutter, E.H., Tatham, D.J., 2011. Deformation mechanisms, rheology and
tectonics: microstructures, mechanics and anisotropy: introduction. In: Prior, D.J., Rutter, E.H.,
Tatham, D.J. (Eds.), Deformation mechanisms, rheology and tectonics: Microstructures,
mechanics and anisotropy. Geological Society Special Publications 360, London, 1-5.

Reddy, S.M., Buchan, C., 2005. Constraining kinematic rotation axes in high-strain zones:
a potential microstructural method? In: Gapais, D., Brun, J.P., Cobbold, P.R. (Eds.), Deformation
mechanisms, rheology and tectonics: from mineral to the lithosphere, 243. Special Publications,
Geological Society, London, 1–10.

Reddy, S.M., Timms, N.E., 2010. Deformation of zircon and implications for geochemistry
and geochronology. Source Abstracts with Programs - Geological Society of America 42, 634.

Reddy, S.M., Timms, N.E., Trimby, P., Kinny, P.D., Buchan C., Blake K., 2006. Crystalplastic deformation of zircon: a defect in the assumption of chemical robustness. Geology 34,
257-260.

Reddy, S.M., Timms, N.E., Pantleon, W., Trimby, P., 2007. Quantitative characterization
of plastic deformation of zircon and geological implications. Contributions to Mineralogy and
Petrology 153, 625–645.

Reddy, S.M., Timms, N.E., Hamilton, P.J., Smyth, H.R., 2009. Deformation-related
microstructures in magmatic zircon and implications for diffusion. Contributions to Mineralogy
and Petrology 157, 231–244.

686	Robinson, K., Gibbs, G. V., Ribbe, P. H., 1971. The structure of zircon: a comparison with
687	garnet. American Mineralogist 56, 782-790.
688	Schmid, D.W., Podladchikov, Y.Yu., 2005. Mantled porphyroclast gauges. Journal of
689	Structural Geology 27, 571–585.
690	Selverstone, J., Morteani, G., Staude, JM., 1991. Fluid channelling during ductile
691	shearing: transformation of granodiorite into aluminous schist in the Tauern Window, Eastern
692	Alps. Journal of Metamorphic Geology 9, 419–431.
693	Timms, N.E., Reddy, S.M., 2009. Response of cathodoluminescence to crystal-plastic
694	deformation in zircon. Chemical Geology 261, 11–23.
695	Timms, N.E., Kinny, P., Reddy, S.M., Evans K., Clark C., Healy D., 2011. Relationship
696	among titanium, rare earth elements, U-Pb ages and deformation microstructures in zircon:
697	Implications for Ti-in-zircon thermometry. Chemical Geology 280, 33–46.
698	Timms, N.E., Reddy, S.M., Healy, D., Nemchin, A.A., Grange, M.L., Pidgeon, R.T., Hart,
699	R., 2012. Resolution of impact-related microstructures in lunar zircon: a shock-deformation
700	mechanism map. Meteoritics & Planetary Science 47, 120–141.
701	Timms, N.E., Erickson, T.M., Zanetti, M.R., Pearce, M.A., Cayron, C., Cavosie, A.J.,
702	Reddy, S.M., Wittmann, A., Carpenter, P.K., 2017. Cubic zirconia in >2370° C impact melt
703	records Earth's hottest crust. Earth and Planetary Science Letters 477, 52–58.
704	Veselá, P., Söllner, F., Finger, F., Gerdes, A., 2011. Magmato-sedimentary Carboniferous
705	to Jurassic evolution of the western Tauern window, Eastern Alps (constraints from U-Pb zircon
706	dating and geochemistry). International Journal of Earth Sciences 100, 993–1027.

Wheeler, J., Mariani, E., Piazolo, S., Prior, D. J., Trimby, P., Drury, M. R., 2009. The
Weighted Burgers Vector: a new quantity for constraining dislocation densities and types using
Electron Backscatter Diffraction on 2D sections through crystalline materials. Journal of
Microscopy 233, 482-494.

Wheeler, J., Mariani, E., Piazolo, S., Prior, D. J., Trimby, P., Drury, M. R., 2012. The
Weighted Burgers Vector: a quantity for constraining dislocation densities and types using
Electron Backscatter Diffraction on 2D sections through crystalline materials. Materials Science
Forum 715-716, 732-736.

White, S., 1973. Syntectonic recrystallization and texture development in quartz. Nature244, 276-278.

717

#### 718 **Figure captions**

Figure 1. Visualization of zircon elastic properties as a function of crystallographic
geometry. A. Young's modulus. B. P waves velocity. C. Poisson ratio. D. Shear modulus. Elastic
constants after Özkan and Jamieson (1978), Bass et al. (1995) and den Toonder et al. (1999).
Solid black circles with labels indicate main crystallographic directions in zircon.

Figure 2. A. and B. Optical microscope images (cross-polarized light) of the thin sections. Foliation and compositional layering are outlined (dotted white line) and labelled. Numbers indicate positions of studied crystal-plastically deformed zircon grains (for more positions refer to Supplemental Fig. 1). C. and D. Petrographic context of zircon grains within the analysed sample (BSE images). Bt = biotite, Chl = chlorite, Kfs = K-feldspar, Pl = plagioclase, Qtz = quartz, Ttn = titanite, Zrn = zircon.

Figure 3. Microstructural data of unstrained zircon from the sampled meta-lamprophyre. Cumulative EBSD maps (left column), user-selected reference point marked with a white star. EBSD pattern quality maps (middle column), patchy inhomogeneities in C indicate surface contamination of the sample by polishing material. Lower hemisphere equal-area projections (right column) show the position of <c> of the corresponding grain (empty circles). The foliation plane in the figures is subhorizontal. Dotted grey circles indicate the direction of stretching lineation.

Figure 4. Microstructural data for fractured zircon. Orientation contrast images of grains with brittle deformation (left column). Zrn = zircon. Lower hemisphere equal-area projections (right column) with inferred position of long axes of corresponding grains (empty circles). The foliation plane in the figures is subhorizontal. Dotted grey circles indicate the direction of stretching lineation.

741 Figure 5. Crystal-plastically deformed zircon crystals: microstructural data, modified from 742 Kovaleva et al. (2016). Left column: EBSD pattern quality images. Arrows point to low-angle 743 boundary network (dark-grey stepped lines). Light-grey areas, separated by low-angle boundaries are subgrains. Middle column: pole figures with lower hemisphere projections of 744 745 zircon crystallographic directions. Labels in square braces ([]) indicate main crystallographic 746 axes, black lines are reconstruction of the subgrain boundary planes. The foliation plane in the 747 figures is subhorizontal. The direction of the lineation is highlighted by dotted grey circles. 748 Arrows in (A) indicate the rotation of all crystallographic directions about [001], which is the 749 least dispersed. Right column: inversed pole figures with misorientation axes density contours 750 with scale bars, numbers are crystallographic directions of zircon.

751 Figure 6. A. Local misorientation map of deformed grain 04. Weighted Burgers Vector 752 (WBV) components are indicated by rectangular subareas. Grey rectangles show the areas with 753 WBV that are comparatively small, indicating no strain. Dotted and dashed rectangles show 754 areas with WBV dominated by the *a* or *b* components, respectively. Black rectangles show areas 755 with WBV with mixed components. B. Reconstruction of the low-angle boundaries and slip 756 systems of grain 04 plotted in the pole figure with lower hemisphere projections of zircon 757 crystallographic directions. Thick lines outside the circle indicate the direction of the low-angle 758 boundary traces. Solid lines inside the circle indicate the reconstruction of low-angle boundary 759 planes. Dashed lines indicate the reconstruction of slip planes for the low-angle boundaries 760 highlighted in A. Elements that correspond to low-angle boundary 1 ("LAB-1") are indicated in 761 black. Elements that correspond to low-angle boundary 2 ("LAB-2") are indicated in grey. The 762 small circle highlights the dispersion and misorientation axis. C. EBSD pattern quality image of 763 grain 04. Horizontal dark line in the upper part is a scratch. D. Inversed pole figure with a scale 764 bar, showing misorientation axes density contours, numbers are crystallographic directions of 765 zircon.

Figure 7. A. Pole figure with <c> positions of analysed zircon grains, adopted from 766 767 Kovaleva et al. 2016. Labels in angle brackets (<>) indicate misorientation axes for the 768 corresponding grains. The grey dashed line shows the direction of the foliation. The grey dotted 769 circle shows the direction of lineation. Solid black circles correspond to <c> directions of grains 770 that are plastically-deformed. Empty circles correspond to unstrained or fractured zircon grains, 771 grey circle shows the orientation of a grain with very minor degree of strain. B. Pole figure with 772 directions of the misorientation axes (indicated by circles) within the sampled shear zone. 773 Misorientation axes of deformed grains analysed in this study are labelled with the

corresponding numbers. Circles are color-coded according to the total amount of strain
corresponding to the cumulative misorientation angle and the number of low-angle boundaries,
using a 0 to 10 scale, where 10 (black circles) are the most strained grains and 0 (white) are the
least strained in the population.

Figure 8. Schematic sketch showing how zircon deformation evolution depends on its orientation in the macroscopic kinematic frame. Cases (i), (ii) and (iii) indicate plastic deformation, (iv) indicates unstrained and fractured grains with c-axes at a high angle to the foliation. (i) <c> is parallel to the vorticity axis, the misorientation axis is parallel to [001]. (ii) Grain with <c> at an angle 45° to the kinematic rotation axis develops [001] and [100] rotation axes. (iii) <c> is normal to the kinematic rotation axis, and the rotation axis is parallel to [001]. Misorientation axes in cases i-iii are parallel to the XY plane of the shear zone.

	Grain #	Types of analyses	Host phase(s)	Type of deformation	Degree of crystal- plastic strain (0-10)	Orientation of <c> axis in the shear zone</c>	Orientation of misorientation axes	Orientation of misorientation axes in the shear zone
Thin section A	01 (Fig. 3A)	EBSD maps	Biotite	None	0	High angle to the foliation	N/A	N/A
	03 (Fig. 5A)		Chlorite and K- feldspar interface	Cr-pl. type III, stepped LABs	10	Parallel to lineation	[001]	Parallel to lineation
	03b		Biotite	Cr-pl. type III, one semi-circular LAB	2	<u>A</u>	<212>	Subparallel to foliation at 55° to lineation
	04 (Fig. 6)		Biotite with titanite pressure shadow	Cr-pl. type III, stepped LABs	4	Subparallel to lineation	[001], minor <212>	Parallel to lineation; ~15° to foliation at 45° to lineation
	09		Quartz, plagioclase and biotite interface	Brittle and minor cr- pl. type II (semi- circular bending at the grain margin), stepped fracture	3	About 15° to foliation and 55° to lineation	[001], <311>	Same as <c> axis</c>
	10 (Fig. 3B)		Plagioclase and quartz interface	None	0	>20° to foliation and 50° to lineation	N/A	N/A
	16 (Fig. 3C)		Plagioclase	None	0	More than 50° from foliation and lineation		
	20		Biotite with titanite pressure shadow	Cr-pl. type III, stepped LAB	1	Parallel to foliation and at 45° to lineation	<101>, <310>, minor <511>	Parallel to foliation at 90° to lineation; normal to foliation and to lineation
section B	15 (Fig. 5B)	OCI and EBSD map	Biotite	Cr-pl. type III, stepped LABs	5		[001], <100>	Parallel to foliation at 45° to lineation; 15° to foliation at 50° to lineation
Thii	15b 16	OCI		None	0	Unknown	N/A	N/A
	19a		Biotite, K-feldspar					
	19b		and epidote			Long axis is at a high angle to foliation		

19c		K-feldspar			Unknown		
20	OCI and EBSD map	Plagioclase	Brittle and cr-pl. type III, stepped LABs and curved fracture	4	15° to foliation 45° to lineation	<201>, <321>	Subparallel to YZ plane at 55° to foliation; subparallel to foliation at 30° to lineation
23	OCI	Plagioclase and K- feldspar	Cr-pl. type III, stepped LABs	1	Unknown	N/A	N/A
24 (Fig. 5C)	OCI and EBSD map	Biotite and plagioclase	Cr-pl. type III, stepped and curved LABs	3	Subparallel to foliation at 90° to lineation	[001], minor <212>, <321>, <110>	Same as <c> axis</c>
24a 24c	OCI	Plagioclase and K- feldspar	None	0	Unknown	N/A	N/A
24d	OCI and EBSD maps	Biotite and plagioclase	Very minor cr-pl. type III, jigsaw- shaped LABs	0.5	High angle to foliation and to lineation	[001], <112>, <312>	Subparallel to YZ plane ~70° to foliation at 85° to lineation
25		K-feldspar, titanite, rutile	Cr-pl. type III, one rounded LAB	2	Subparallel to foliation and at 10° to lineation	<210>	15° to foliation at 55° to lineation
26		Biotite	Cr-pl. type III, stepped LABs	9	5° to foliation and 10° to lineation	[001]	Same as <c> axis</c>
27	OCI	Chlorite and K- feldspar	None	0	Unknown	N/A	N/A
28	OCI and EBSD map	Biotite	Cr-pl. type III, stepped LABs	6	Parallel to lineation	[001]	Parallel to lineation
31a (Fig. 4B)	OCI	Biotite and K- feldspar	Brittle, stepped fracture	0	Long axis is at high angle to foliation	N/A	N/A
31b	OCI and EBSD map	Biotite	Cr-pl. type III, stepped LABs	6	Parallel to lineation	[001], minor <212>, <311>	Parallel to lineation
32a	OCI	K-feldspar, titanite and plagioclase	Cr-pl. type III (very minor), stepped LAB	0.5	Unknown	N/A	N/A
32b			Brittle, curved fracture	0			
33 (Fig. 4A)		Plagioclase and biotite	Brittle, subparallel fractures		Long axis is at high angle to foliation		
36 37a	-	Biotite	None		Unknown		

Table 1. Orientation and microstructural data for the zircon grains, analysed in this study. OCI = orientation contrast imaging, Cr-pl. = crystal-plastic, LAB = low-angle boundary. In italic – minor misorientation axes directions that are not plotted in Fig. 7B. Degree of strain is empirically evaluated, based on (1) amount and density of low-angle boundaries, and, where applicable, (2) cumulative misorientation within the grain.

CHRITIAN MANUS



# Figure 2



# Figure 3







© 2017. This manuscript version is made available under the CC-BY-NC-ND 4.0 license http://creativecommons.org/licenses/by-nc-nd/4.0/

# Figure 5





103

101

201

501

Grain 24

# Figure 6









- Under amphibolite-facies zircon forms a network of low-angle boundaries by switching between tilt and twist dislocations with [010] and [001] rotation axes
- Documented tilt dislocations have <100>{010}, <100>{001} and <-110>{110} geometry
- Elastic anisotropy properties of zircon influence its rheological behavior
- The rotation axis [001] is conditioned by high Young's modulus values along this direction
- The slip system <-110>{110} is newly described for zircon