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- 1 Microstructurally controlled trace element (Zr, U–Pb)
- 2 concentrations in metamorphic rutile: An example from the

# 3 amphibolites of the Bergen Arcs

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- 17

#### 18 Abstract

19 As a common constituent of metamorphic assemblages, rutile provides constraints on the 20 timing and conditions of rock transformation at high resolution. However, very little is 21 known about the links between trace element mobility and rutile microstructures that result 22 from syn-metamorphic deformation. To address this issue, here we combine in situ LA-ICP-23 MS and SHRIMP trace element data with EBSD microstructural analyses to investigate the 24 links between rutile lattice distortions and Zr and U-Pb systematics. Furthermore, we apply 25 this integrated approach to constrain further the temperature and timing of amphibolite-facies 26 metamorphism and deformation in the Bergen Arcs of southwestern Norway. In outcrop, the 27 formation of porphyroblastic rutile in dynamically hydrated leucocratic domains of otherwise 28 rutile-poor statically-hydrated amphibolite provides key contextual information on both the 29 ambient conditions of hydration and deformation and the composition of the reactive fluid. 30 Rutile in amphibolite recorded ambient metamorphic temperatures of ~590-730°C during 31 static hydration of the granulitic precursor. In contrast, rutile from leucocratic domains in the 32 directly adjacent shear zone indicates that deformation was accompanied by a localized 33 increase in temperature. These higher temperatures are recorded in strain-free rutile ( $\sim 600$ – 34 860°C) and by Zr concentration measurements on low-angle boundaries and shear bands 35 (620-820°C). In addition, we also observe slight depletions of Zr and U along rutile low-36 angle boundaries relative to strain-free areas in deformed grains from the shear zone. This 37 indicates that crystal-plastic deformation facilitated the compositional re-equilibration of 38 rutile upon cooling to slightly below the peak temperature of deformation. Cessation of 39 deformation at mid-crustal conditions near ~600°C is recorded by late stage growth of small 40 (< 150 µm) rutile in the high strain zones. U–Pb age data obtained from the strain-free and 41 distorted rutile grains cluster in distinct populations of  $437.4 \pm 2.7$  Ma and c. 405-410 Ma, 42 respectively. These different ages are interpreted to reflect the difference in closure for

thermally-induced Pb diffusion between undeformed and deformed rutile during postdeformation exhumation and cooling. Thus, our results provide a reconstruction of the thermochronological history of the amphibolite-facies rocks of the Lindås Nappe and highlight the importance of integration of microstructural data during application of thermometers and geochronometers.

### 48 KEYWORDS

49 Zr-in-rutile thermometry, U–Pb geochronology, deformation microstructures, amphibolite,
50 high diffusivity pathways

## 51 **1. INTRODUCTION**

52 Accessory minerals are useful tracers of the timing and the conditions at which rocks 53 undergo physical and chemical changes. Recognition of the strong temperature dependence 54 of Zr-content in rutile (Tomkins, Powell, & Ellis, 2007; Watson, Wark, & Thomas, 2006; 55 Zack, Moraes, & Kronz, 2004) has caused a growing interest in rutile due to its applicability 56 as a metamorphic thermometer. Combined application of Zr-in-rutile thermometry with rutile 57 U–Pb geochronology (e.g. Kylander-Clark, Hacker, & Mattinson, 2008; Zack et al., 2011; 58 Zeh, Cabral, Koglin, & Decker, 2018) can therefore provide invaluable constraints on a 59 rock's metamorphic history. While providing an abundance of important geochemical data, the incomplete understanding of the susceptibility of rutile composition to deformation and 60 61 fluid alteration during metamorphism complicates its application as a thermochronometer in strained and hydrated rocks. 62

Most studies of rutile Zr-thermometry and U–Pb geochronology have either focused
on high-temperature granulites that have undergone slow cooling (e.g. Kooijman, Mezger, &
Berndt, 2010; Vry & Baker, 2006) or have been performed under anhydrous experimental

66 conditions (Cherniak, Manchester, & Watson, 2007). Thus, compositional re-equilibration of 67 rutile is commonly attributed to volume diffusion, whereas the effects of deformation and 68 fluid alteration have received relatively little attention. Accordingly, rutile ages are 69 interpreted as the time at which the grain cooled below the closure temperature for volume 70 diffusion (e.g. Dodson, 1973; Flowers, Bowring, Tulloch, & Klepeis, 2005; Ganguly & 71 Tirone, 1999; Hirdes & Davis, 2002; Mezger, Hanson, & Bohlen, 1989). For grains larger 72 than 200 µm in diameter the U-Pb system closure is estimated to be at 600°C (2-3°C/Ma 73 cooling rate; Cherniak, 2000; Vry & Baker, 2006), and that of Zr above ~640°C (2°C/Ma 74 cooling rate; Cherniak et al., 2007). This implies that rutile in retrogressed metamorphic 75 rocks should generally display correlating age and temperature data. However, analyses of 76 natural rutile suggest that the concept of closure temperature mostly applies to the U-Pb 77 system, whereas the frequently observed lack of correlation between grain size and calculated 78 temperatures implies that Zr concentrations in rutile are relatively insensitive to volume 79 diffusion (Ewing, Hermann, & Rubatto, 2013; Pape, Mezger, & Robyr, 2016).

80 Therefore, investigation into post-crystallisation modification of Zr contents in rutile 81 is of importance to applications of the thermometer. Recent studies have demonstrated that 82 high-pressure fluid alteration may not only mobilize Zr from the rutile but also provide the 83 required Si for zircon formation, thus controlling Zr partitioning with rutile (Harley, 2008; 84 Luvizotto & Zack, 2009; Meyer, John, Brandt, & Klemd, 2011; Mitchell & Harley, 2017; Pape et al., 2016). In addition, the variability of Zr concentration within and among grains 85 86 has been attributed to the proximity of nearby Zr-bearing phases and rutile grain morphology, 87 further indicating a textural control on the Zr concentration in rutile (Ewing et al., 2013; 88 Kooijman, Smit, Mezger, & Berndt, 2012; Mitchell & Harley, 2017; Pape et al., 2016). Thus, 89 despite the apparent validity of closure temperatures for the U-Pb system, mechanisms other 90 than volume diffusion need to be considered when interpreting Zr concentrations in rutile. In

addition to the mechanisms outlined above, it is reasonable to assume that rutile Zr and U–Pb
contents may be further modified by deformation. Particularly, low-angle boundaries have
been shown to act as fast diffusion pathways for trace elements in both pyroxene and zircon
(Piazolo, Austrheim, & Whitehouse, 2012; Piazolo et al., 2016; Timms et al., 2011),
enhancing the resetting of both temperature and age data. If the effect of deformation on
composition is similarly significant in rutile, then previous observations of the inconsistent
nature of temperatures recorded by rutile within samples may be partially explained.

98 In the Lindås Nappe of the Bergen Arcs in southwestern Norway the complete 99 dehydration of rocks at granulite-facies conditions prior to the fluid-induced Caledonian 100 tectonometamorphic amphibolite-facies overprint provides an ideal geological setting for 101 investigating the effects of fluid-rock interaction and deformation on element mobility in 102 rutile. We observe a spatial association between strain, fluid infiltration and rutile distribution 103 in amphibolite-facies rocks. Through targeted sampling of rutile-rich high strain zones and 104 fractures, we show how combined microstructural and geochemical analyses of rutile can 105 constrain the hydration-deformation history of the Lindås Nappe amphibolite. Furthermore, 106 we explore the link between deformation and trace element distribution in rutile, which 107 highlights the benefit of incorporating microstructural analyses in future applications of the 108 Zr-in-rutile geothermometer.

#### 109 **2.**

#### 110

### **2. BACKGROUND AND OUTCROP DESCRIPTION**

## 0 **2.1. General background and previous geochronological work**

111 The Bergen Arcs lie within the hanging wall of the extensional Bergen Arc shear zone. 112 The arcs encompass the Øygards gneiss complex, structurally overlain by a set of nappes, 113 including the Lindås Nappe, which is the focus of this study (Figure 1a). The Lindås Nappe 114 mainly consists of an anorthosite-mangerite-charnockite-granite (AMCG) suite and banded 115 gneiss complexes. Pulse-like intrusion of the AMCG suite occurred between 1237 Ma and 951 Ma (Bingen, Davis, & Austrheim, 2001), followed by pervasive recrystallization at
granulite-facies conditions during the late Grenvillian-Sveconorwegian Orogeny at *c*. 930–
910 Ma (Bingen et al., 2001; Cohen, O'nions, Siegenthaler, & Griffin, 1988). Partial
transformation of the granulite-facies rocks to eclogite and amphibolite-facies mineral
assemblages advanced via localized fluid infiltration along fractures (Austrheim, 1987;
Jamtveit, Bucher-Nurminen, & Austrheim, 1990) between *c*. 440 Ma and 420 Ma during the
Caledonian Orogeny (Bingen et al., 2001).

123 Previously acquired geochronological data for the amphibolite-facies rocks of the 124 Bergen Arcs define a broad time-span for amphibolite-facies metamorphism and deformation 125 (Figure 1b). Amphibole Ar-Ar data record the oldest ages at  $455 \pm 2$  Ma and  $439 \pm 4$  Ma 126 (Boundy, Essene, Hall, Austrheim, & Halliday, 1996). However, the validity of these early ages has been questioned based on the interpretation that these Ar-Ar data might reflect the 127 128 presence of excess Ar in the amphiboles instead of constraining the cooling age below the 129 closure temperature for Ar diffusion (Kühn, Glodny, Austrheim, & Råheim, 2002; Roffeis, 130 Corfu, & Austrheim, 2012). Zircon generally records slightly younger U–Pb concordia ages 131 between  $426 \pm 4$  Ma and  $430 \pm 3$  Ma (Glodny, Kühn, & Austrheim, 2008; Roffeis et al., 132 2012). One U–Pb age of  $437 \pm 11$  was obtained from zircons that had been deformed by amphibolite-facies shear (Piazolo et al., 2012). A zircon U–Pb age of  $418 \pm 9$  Ma (Kühn et 133 134 al., 2002) and a  $433 \pm 3$  Ma biotite Ar-Ar age (Fossen & Dunlap, 1998) from a sheared 135 trondhjemite dyke are interpreted as directly recording the timing of amphibolite-facies 136 deformation. These ages define a span of c. 20 Ma for the amphibolite-facies deformation. 137 Rb-Sr multi-mineral isochron data provide the youngest age estimates at  $414 \pm 5$  Ma (apatite, feldspar, white mica; Glodny et al., 2008) and  $409 \pm 8$  Ma (apatite, amphibole, biotite; 138 139 Bingen et al., 2001), consistent with the reported low temperature resetting of the Rb-Sr

system in mica, giving an estimate for the timing of crystallization of these lower temperatureminerals.

142 Additional constraints on the timing of the amphibolite-facies metamorphism are given by biotite muscovite <sup>40</sup>Ar/<sup>39</sup>Ar plateau ages from the Ulriken Gneiss unit immediately 143 144 to the west of the Lindås Nappe (Fossen & Dunlap, 1998). Plateau ages of c. 410 Ma from 145 greenschist-facies shear zones constrain the time of nappe stacking in the Bergen Arcs, 146 marking the end of Caledonian contractional deformation. In addition to the amphibolite-147 facies rocks, pre- to syn-tectonic pegmatites have been dated by Rb-Sr and Sm-Nd multi-148 mineral isochron methods at c. 425 Ma (Kühn et al., 2002) and by U–Pb in zircons at 424±1 149 Ma (Jamtveit et al., 2018). These dates, interpreted as crystallization ages, indicate that the 150 mobility of hot silicate rich fluid was synchronous with metamorphism. However, the exact 151 relationship between the emplacement of the pegmatites and the amphibolite-facies alteration 152 remains unknown.

153 If interpreted as an isolated event, the geochronology derived from the amphibolite-154 facies mineral assemblages provides a sensible timeline. However, this apparent c. 430 Ma 155 amphibolite-facies episode overlaps with the 429  $\pm$  3 Ma (Glodny et al., 2008) and 423  $\pm$  4 156 Ma (Bingen, Austrheim, Whitehouse, & Davis, 2004) ages derived from eclogite-facies 157 assemblages that are intercalated with the amphibolites on Holsnøy (Figure 1b). This 158 overlapping of ages has been explained by a west-to-east pressure decrease during 159 simultaneous amphibolite- and eclogite-facies metamorphism (Roffeis et al., 2012). 160 However, the interpretation remains contentious, since the characteristics and timing of the 161 amphibolitization are somewhat ambiguous compared to the robustly constrained eclogite 162 facies metamorphism.

Here we focus on integrating rutile Zr-thermometry, U–Pb age determination, and
 EBSD microstructural analyses to establish a rigorous temperature-deformation-time history

of the amphibolite-facies event. With an emphasis on distinguishing the deformational and
metamorphic controls on the amphibolite-facies rutile temperature and age data.

167

## 2.2. Description of lithological relationships

168 The sampled outcrop, situated on Radøy in the northeastern part of the Lindås Nappe 169 (Figure 1a) (UTM zone 31V, 620260E, 6718524N), includes three main lithologies; 170 granulite, amphibolite, and leucocratic domains within the shear zone (Figure 2a). The 171 amphibolite occurs between the other two lithologies. The granulite, characterized as a lilac 172 corona-bearing medium-grained rock, shares an irregular and transitional contact with the 173 amphibolite. Where the granulite grades into the amphibolite the foliation becomes defined 174 by elongate amphibole clusters (1-2 cm in length), directly replacing coronitic diopside and 175 garnet that define the foliation in the granulite. Strain increases markedly throughout the 176 amphibolite towards the shear zone that contains additional leucocratic domains (Figure 2a). 177 Within 50 cm from the first appearance of leucocratic domains, the foliation of the 178 amphibolite grades into a 5-40 cm wide zone, where the foliation is defined by mafic and 179 felsic bands with 5 mm spacing, marking the outer limit of the shear zone. In the following 180 we refer to this part of the amphibolite as strained amphibolite. Within the shear zone, the 2-3 cm spaced foliation is defined by dark, amphibole- and feldspar-bearing amphibolite bands 181 182 that alternate with creamy white leucocratic domains dominated by feldspar and strongly 183 aligned clinozoisite.

In addition, the granulite contains two types of fractures (Figure 2b). Fracture type-I presents as a 1-2 cm wide plane hosting amphibole. Fracture type-II is apparent as 1-2 mm wide greenish fractures surrounded by white alteration haloes with a width of 1-2 cm, oriented subparallel to the high strain zone.

#### **3. METHODS**

#### **3.1. Sample preparation**

Polished thin sections were used for electron microprobe (EMP) analyses of rutile
grains from the amphibolite and fracture alteration haloes. Additional EMP analyses were
performed on plugs (3.8 mm diameter) drilled from lineation parallel (XZ) sections of
leucocratic domains. Plugs were mounted in epoxy resin, polished initially with 6 µm and 1
µm diamond suspension fluid and given a final chemomechanical polish with colloidal silica
for electron back-scatter diffraction (EBSD) analysis.

## **3.2. Imaging and quantification of rutile microstructures**

Back-scatter electron (BSE) imaging and EBSD analyses were carried out on the
Tescan Mira3 Variable Pressure Field Emission Scanning Electron Microscope (VP-FESEM)
at the John de Laeter Centre (JDL), Curtin University, Australia. For both BSE and EBSD
acquisition, the VP-FESEM was run at a high vacuum with an accelerating voltage of 20 kV
and a beam current of 5.0 nA.

202 EBSD patterns were acquired with a HKL NordlysNano high sensitivity EBSD 203 detector and indexed with AzTec analysis software (Oxford Instruments). The samples were 204 tilted to 70° and analysed at a working distance of 20 mm to 24.5 mm. The step size ranged 205 between 1 µm and 10 µm depending on the grain size and required spatial resolution. EBSD 206 data was processed using HKL's Channel 5 software. Non-indexed solutions were replaced 207 by the most common neighbour orientation to reduce data noise following the procedure 208 outlined in Prior, Wheeler, Peruzzo, Spiess, and Storey (2002), Bestmann and Prior (2003) 209 and Piazolo, Bestmann, Prior, and Spiers (2006).

210

Mineral abbreviations used in the figures follow Whitney and Evans (2010).

211

#### **3.3. U–Pb geochronology**

212 The sensitive high-resolution ion microprobe (SHRIMP) analytical procedure broadly 213 follows those described by Compston, Williams, and Meyer (1984) and Williams (1998). 214 SHRIMP rutile data were collected from one mount (N17-44) over one analytical session at 215 JDL. During this session, a 10-15  $\mu$ m diameter spot for grains 30 and 32 and a 20-25  $\mu$ m 216 diameter spot for grains 29, 31 and 33 were used (Table S4b), with a mass-filtered O<sup>-</sup>-217 primary beam of ~7.6-8.3 and 25.0-29.5 nA on rutile from a leucocratic domain sample, 218 respectively. Data for each spot was collected in sets of 6 scans on the rutile through the mass range of <sup>192</sup>Ti<sub>3</sub>O<sub>3</sub><sup>+</sup>, <sup>200</sup>WO<sup>+</sup>, <sup>204</sup>Pb<sup>+</sup>, Background, <sup>206</sup>Pb<sup>+</sup>, <sup>207</sup>Pb<sup>+</sup>, <sup>208</sup>Pb<sup>+</sup>, <sup>248</sup>ThO<sup>+</sup>, <sup>254</sup>UO<sup>+</sup>and 219 220 <sup>270</sup>UO<sub>2</sub><sup>+</sup>. The rutile age standard used was WH (Windmill Hill), a multigrain standard from an Archean quartzite, with a <sup>206</sup>Pb/<sup>238</sup>U age of 2625 Ma (Clark, Hensen, & Kinny, 2000). 221 222 WH has been calibrated against the Wodgina single rutile crystal described by Ewing (2011) 223 and Ewing, Rubatto, Beltrando, and Hermann (2015) using the procedures described therein. Although we note that a small proportion of rutile grains from WH have <sup>207</sup>Pb/<sup>206</sup>Pb ages 224 225 slightly older than the dominant age population (manuscript in preparation), we only observe this in the more precise <sup>207</sup>Pb/<sup>206</sup>Pb ages, not in the <sup>206</sup>Pb/<sup>238</sup>U data. These "older" grains 226 227 were avoided during analysis of the WH standard. No U standard was analysed during this 228 session although estimated U contents are shown in Table S4b and based on the average U concentration in WH of 164 ppm. The common Pb correction was based on the measured 229 <sup>208</sup>Pb due to the negligible Th contents on rutile. The programs SQUID II and Isoplot 230 231 (Ludwig, 2003, 2009) were used for rutile data processing.

232 Uncertainties  $(2\sigma)$  cited for individual analyses include uncertainties from counting 233 statistics and the common-Pb correction. Analyses for which the proportion of common Pb 234 was  $\geq 1.4\%$  were not considered in the age discussion or plotted on the Concordia diagram. 235 Weighted mean values for the mean age given on pooled analyses are at the 95% confidence 236 level.

237 Rutile grains from leucocratic domains were analysed for U-Pb ratios by LA-ICP-MS 238 at the Institut für Mineralogie, University of Münster, Germany. Ablation was done with an 239 excimer laser (Analyte G2, Photon Machines) connected to a Thermo Fisher Scientific 240 Element2 magnetic sector field single collector ICP-MS, at a repetition rate of 10 Hz and a 241 spot size of 50  $\mu$ m, producing a pit depth of ~ 30  $\mu$ m. Prior to sample analyses, the system was tuned for high sensitivity, stability, and low oxide-interference rates  $(^{232}\text{Th}^{16}\text{O}/^{232}\text{Th} <$ 242 0.05%). For rutile U–Pb measurements five unknowns were bracketed with two analyses of 243 244 R10, which was used as external standard (Luvizotto et al. 2009). Along with the unknowns, 245 secondary reference rutiles SP1 (Mezger et al., 1989, 911±2 Ma), Sugluk-4 (Bracciali, 246 Parrish, Horstwood, Condon, & Najman, 2013, 1723.0±6.8 Ma), and R-632 (Axelsson et al., 247 2018, 496±2 Ma) were analysed to check for precision and accuracy. Obtained results show 248 good agreement with the published values (917.2  $\pm$  8.5 Ma for SP1, 1729  $\pm$  14 Ma for 249 Sugluk-4, 505.9±6.4 Ma for R-632). U–Pb rutile data were corrected for common Pb and 250 processed following the procedures described in Kooijman, Berndt, and Mezger (2012) and 251 Zack et al. (2011). A common Pb correction was applied to analyses if the contribution of the estimated common <sup>206</sup>Pb to the total measured <sup>206</sup>Pb exceeded 1%. The isotope ratios for the 252 253 common Pb were calculated using the evolution model for terrestrial Pb by Stacey and 254 Kramers (1975).

255 LA-ICP-MS and SHRIMP data are presented in Concordia, weighted mean, and radial plots, produced in IsoplotR (Vermeesch, 2018). Ages in figures are <sup>206</sup>Pb/<sup>238</sup>U dates. 256

257

## **3.4. Trace elements and Zr-in-rutile thermometry**

258 Rutile trace element concentration measurements by EMP (n = 72) were carried out 259 on a field-emission JEOL 8530F Hyperprobe equipped with five Wavelength-Dispersive

260 Spectrometers at the Centre for Microscopy, Characterization and Analysis (CMCA), The University of Western Australia. Operating conditions were a beam energy of 25 keV, the 261 262 beam current was 60 nA, and the beam was fully focused. Elements were acquired using the 263 following analysing crystals: LiF for Ti Ka, V Ka, Cr Ka, Mn Ka, Fe Ka, and Ni Ka; TAP for Si Ka and Al Ka; and PETH for Ca Ka, Zr La and Nb La. The standards employed were 264 265 commercially available silicates, oxides, and metals. Counting time was 20 s for Ti Ka, 70 s for Cr Ka, Mn Ka, Fe Ka, Ni Ka, Hf La, Si Ka, Al Ka, Ca Ka, Zr La and Nb La, and 100 s 266 267 for V K $\alpha$ . Mean atomic number background corrections were employed throughout 268 (Donovan and Tingle, 1996). Unknown and standard intensities were corrected for dead time 269 and the ZAF algorithm was used for matrix absorption (Armstrong, 1988). On-peak 270 interference corrections were applied as appropriate (Donovan et al., 1993). Detection limits 271 ranged from 10 ppm for Ca to 40 ppm for Hf.

272 Rutile grains were analysed for trace elements also by LA-ICP-MS at the Institut für 273 Mineralogie, University of Münster. For trace element analyses the NIST 610 glass was used 274 as external standard while synthetic TNT666 and TNT777 glasses (Klemme et al., 2008) as 275 well as natural rutile R10 (Luvizotto et al., 2009) were measured along with the unknowns to 276 monitor precision and accuracy. Obtained results generally match the published range of concentrations given in the GeoReM database (version 23) (Jochum et al., 2005). 277 278 Approximately 25 sample measurements were bracketed by three analyses of the external standard. <sup>47</sup>Ti was used as internal standard element. Trace element data reduction was 279 performed using Glitter 4.4.4 (Griffin, 2008). For LA-ICP-MS the elemental concentrations 280 were analysed by measuring the following isotopes: <sup>238</sup>U, <sup>118</sup>Sn, <sup>121</sup>Sb, <sup>182</sup>W, <sup>95</sup>Mo, <sup>53</sup>Cr, <sup>66</sup>Zn, 281 <sup>51</sup>V, <sup>181</sup>Ta, <sup>93</sup>Nb, <sup>90</sup>Zr, and <sup>178</sup>Hf. The obtained trace element data were filtered to exclude all 282 measurements with > 800 ppm Si showing abnormally high Zr contents, a method adapted 283

from Zack et al. (2004) and Luvizotto and Zack (2009) in an effort to detect zircon-lamellae
rich rutile and zircon micro inclusions.



## 291 **4. RESULTS**

292

### 4.1. General microstructural description of the main lithologies

293 The transition from granulite to amphibolite is marked by an overall grain size 294 reduction and replacement of diopside-garnet coronas by amphibole clusters (Figures 3a and 295 b). The amphibolite features 1 cm-scale interlayering of two characteristic domains; an 296 amphibole-rich and a plagioclase-rich domain. The amphibole-rich domain is composed of 297 amphibole with minor amounts of chlorite, biotite, quartz, and kyanite (Figure 3b). Within 298 the amphibole-rich domain, trace amounts of rutile and pyrite are observed associated with 299 replaced diopside. The plagioclase-rich domain is composed of plagioclase with minor 300 amounts of zoisite, kyanite, quartz and CO<sub>3</sub>-rich scapolite (Figure 3b). The replacement of 301 diopside and garnet by amphibole is also observed in alteration haloes around fractures type-I 302 and II (Figure 4a).

In the strained amphibolite, interlayering of amphibole and plagioclase domains occurs on a finer, 1 mm-scale (Figure 3c). Amphibole-rich and plagioclase-rich domains of the strained amphibolite differ from those in the amphibolite in that they contain a higher abundance of clinozoisite and zoisite. In the shear zone the foliation is defined by mm-scale interlayering of dark amphibole-rich with plagioclase-rich domains (Figure 3d), consistent with the lithological variation in the strained amphibolite. On the cm-scale, these dark bands are interlayered with porphyroblastic rutile-bearing leucocratic domains (Figures 3d and 5a). In areas of increased leucocratic component, small (< 100  $\mu$ m) plagioclase and zoisite grains dominate the rock and define the foliation (Figures 3d and 5b, c). In these areas clinozoisite and biotite laths are aligned with the foliation (S; Figure 3d) and form in shear bands at ~30° to S (C; Figure 5a).

314

## 4.2. General occurrence of rutile and its mineral association

Rutile occurs as an accessory phase (< 1 volume %) in all lithologies except for the granulite. Rutile always occurs together with quartz and zircon, except in the fracture alteration haloes where zircon is absent. In general, rutile overall abundance and grain size positively correlate with clinozoisite, biotite, quartz, CO<sub>3</sub>-rich scapolite and tschermakite abundances.

320

## 4.2.1. Amphibolite

321 Hydration of granulite to amphibolite proceeded through the replacement of granulite-322 facies diopside by secondary clusters of Mg-hornblende, tschermakite, quartz, carbonate and 323 rutile (Figures 3b and 4b). In the strained parts of the amphibolite rutile grains (<10  $\mu$ m) are 324 associated with amphibole clusters, whereas larger rutile grains (< 150  $\mu$ m) occur in 325 association with small (< 100  $\mu$ m) clinozoisite grains, very small zircon grains (< 20  $\mu$ m) and 326 large grains (< 1 mm) of biotite that are aligned with the foliation (Figure 4c).

327

#### 4.2.2. Fracture alteration haloes within granulite

The fracture filling assemblage of type-I fractures comprises quartz and tschermakite (Figure 4a). Alteration haloes around type-I fractures in the granulitic anorthosite, visible only in thin section, contain abundant rutile in amphibole clusters (Figure 4d). Here, rutile grains are equant and rounded with diameters of up to 30 µm. On the outer edge of the fracture type-I alteration halo, diopside is replaced by symplectites of magnesio-hornblende,
clinozoisite, quartz, and rutile (Figure 4e).

Type-II fractures predominantly contain quartz, with minor amounts of fine-grained (< 10  $\mu$ m) biotite and amphibole. Type-II fracture alteration haloes are visible at both the outcrop and thin section scale due to abundant zoisite inclusions in the host plagioclase, producing a milky white appearance. In the alteration halo, rutile is present in clinozoisiteand pargasite-filled fractures in diopside as clusters of rounded-cuspate shaped grains ranging in diameter between 10  $\mu$ m and 30  $\mu$ m (Figure 4f). Zircon is not observed in association with either fracture types.

341

### 4.2.3. Leucocratic domains

342 The leucocratic domains of the shear zone are the primary rutile-bearing lithology. In 343 the shear zone the mineralogical variation typical of the strained amphibolite is evidenced by 344 amphibole-rich foliation-parallel bands primarily consisting of Mg-hornblende with 345 tschermakitic rims, quartz and minor amounts of clinozoisite and rutile (< 20 µm in diameter) 346 (Figure 5f). Where the strained amphibolite is interlayered at a higher frequency with the 347 leucocratic domains, there is an increase in tschermakitic amphibole, coarse grained rutile (< 200 µm diameter), biotite and clinozoisite, concomitant with a decrease in Mg-hornblende 348 349 (Figure 5d). In areas where the amphibole domains of the strained amphibolite are no longer 350 interlayered with the leucocratic domains, the mafic component primarily consists of (< 1 351 mm) foliation-parallel laths of clinozoisite and biotite and porphyroblastic rutile (< 1 mm), 352 aligned with their long axes parallel to the foliation (Figure 5b).

In plagioclase-dominated areas of the leucocratic domains, porphyroblastic rutile, biotite, CO<sub>3</sub>-rich scapolite and clinozoisite occur in S and C foliation parallel bands (Figure 5a, e). Rutile also occurs as foliation-parallel grains in the felsic component of the leucocratic rock together with plagioclase, kyanite, zoisite and quartz but only minor amounts of biotite and clinozoisite (Figure 5c). Throughout the leucocratic domains zircon occurs as very small grains ( $< 10 \,\mu$ m) on the rim of rutile (Figure 5b and c) and, uncommonly, as inclusions within the rutile grain (Figure 5c).

360

## 4.3. Details of rutile microstructures and quantitative orientation analysis

361 Since the leucocratic domains are the main rutile-bearing lithology, most of this 362 section investigates rutile in the leucocratic domains unless stated otherwise. Rutile is 363 categorized based on grain size, morphology, structure and the mineral assemblage described 364 above. A first order division is made based on grain size into subsets of large (> 150  $\mu$ m) and 365 small grains (< 150  $\mu$ m).

Large porphyroblastic rutile is mostly present as substructure-free grains (Figure 6a, b) that exhibit an internal grain orientation spread (GOS) of < 1°. Porphyroblastic rutile has a highly irregular grain shape, typically displaying cuspate protrusions into the surrounding matrix (e.g. Figure 6a). Most commonly, these grains are observed in the leucocratic domains (Figures 6a and c) and only one occurrence was found in an amphibole cluster within the amphibolite (Figure 6b).

However, there are substructured large porphyroblastic rutile grains showing a GOS of > 1° (Figures 7a, e and h). For these grains, four types of substructures can be identified: (1) shear bands; (2) continuous lattice bending and low-angle boundaries; (3) distinct, straight growth twins and (4) deformation twins. To exclude the possibility that the observation of substructured versus substructure-free grains is due to an orientation effect, the c-axis orientations of all large grains have been plotted (Figure S1), demonstrating no preferred orientations for grains of either structure.

379 Shear bands are observed in large rutile (>  $150 \mu$ m) and are associated with a high 380 occurrence of recrystallized rutile and/or kyanite inclusions and are marked by a change in 381 trace element chemistry (Figure 8) and a  $\sim 4^{\circ}$  grain orientation change across the plane 382 (Figure 7a). Misorientation data for large substructured rutile grains identify three types of 383 lattice distortions (Types-I to -III) associated with low-angle boundaries and/or shear bands. 384 The first type, Type-I, is observed at low-angle boundaries associated with shear bands with a rotation axis near <112>, lying on the low-angle boundary wall (Figure 7b and c). Shear 385 386 bands are distinguished from continual lattice bending by their sharp misorientation profile. 387 The misorientation profile across the boundary (A-A'; Figure 7d) shows a sharp change in 388 orientation at the low-angle boundaries on the order of  $\sim 1^{\circ}/\mu m$ , without significant 389 orientation variation outside of the bounding low-angle boundaries.

390 Low-angle boundaries appear as subparallel discontinuous bands, which are most 391 commonly parallel to the short axis of the grain (Figure 7e). They may be associated with 392 continuous lattice bending on either side of the low-angle boundary (Figure 7g). Continuous 393 lattice bending is associated with Type-II lattice distortions. Type-II lattice distortions are 394 characterized by their lamellar, subparallel appearance (Figure 7e) and a rotation axis lying 395 approximately on the pole to the (110)-plane (Figure 7f). The misorientation profile across 396 lamellar boundaries (B-B'; Figure 7g) indicates stepwise misorientation on the order of 397  $0.6^{\circ}/\mu m$  at each boundary wall, resulting in a progressively increasing misorientation relative 398 to the original grain orientation.

While both twin types are identified as a 65° misorientation around [010], deformation twins are differentiated from growth twins based on their characteristic thin and tapered appearance (e.g. Figures 6a and 7a, e). In contrast, growth twin boundaries appear lobate and irregular (e.g. Figure 7h). Type III lattice distortions are commonly spatially related to growth twinning (Figure 7h). Type-III lattice distortions are exclusively present within the outer 100 µm of the grain and are characterized by a rotation axis lying approximately on the pole to the (100)-plane (Figure 7i). Overprinting relationships between the different lattice distortion 406 types indicate that deformation twinning preceded the formation of shear bands and growth
407 twins (Figure 7a), but occurred after at least some crystal lattice bending and is associated
408 with the formation of low-angle boundaries (Figure 7e).

Small rutile grains occur either associated with porphyroblastic biotite (Figures 4c and
5e) or around large porphyroblastic rutile grains (Figure 5b). These grains exhibit a GOS of <</li>
1° and very rarely show any low-angle boundaries or crystal lattice bending.

412

## 4.4. Trace element geochemistry

Fifty-one LA-ICP-MS trace element spot analyses were obtained from 39 rutile grains
in the leucocratic gneiss. Trace element concentrations are summarised in Table S2 and
plotted against distance to the grain boundary (Figure 8).

416 Hafnium, Zr and U concentrations show systematic changes in large grains vs. small 417 grains, shear bands, low-angle boundaries and twins (Figure 8a, c and d). Due to the thin 418 width of deformation twins and the resolution of the instruments used, trace element analyses 419 of deformation twins could not be performed, hence chemical characteristics of only the 420 growth twins are provided. The concentration of U is generally between 0.1 ppm and 18 ppm 421 with an average concentration of 6 ppm. In substructure-free grains, the average U 422 concentration is 8 ppm. In contrast, small grains and grains with irregular twins and shear 423 bands have less than 5 ppm U. Grains with low-angle boundaries have on average 3 ppm U. 424 The average concentrations of Zr and Hf of all analysed grains are 1260 ppm and 35 ppm, 425 respectively. If only substructure-free grains are considered, the average concentrations of Zr 426 and Hf are 1500 and 40 ppm, in comparison to an average of 950 ppm Zr and 30 ppm Hf for 427 spot analyses on grains containing low-angle boundaries and 265 ppm Zr and 11 ppm Hf 428 average concentrations in small grains and growth twins. Concentrations of V, Nb and Ta 429 vary non-systematically (Figure 8b, e and f).

430

431

#### 4.5. Zr-in-rutile thermometry

432 lithologies are summarised in Table S3 (Figure 9). The Al-hornblende pressure estimates 433 used in the calculation are derived from tschermakite, ranging from 10 kbar to 14 kbar (Table 434 S1). In the amphibolite, rutile contains between 146 ppm ( $607 \pm 27 \text{ °C}$ ) and 572 ppm Zr (727 435  $\pm$  17 °C) (Figure 9a). Small rutile from the strained amphibolite contains between 74 ppm Zr 436  $(557 \pm 35 \text{ °C})$  and 247 ppm Zr  $(655 \pm 22 \text{ °C})$  (Figure 9c). The Zr concentration in rutile from 437 type-I fracture alteration haloes varies between 125 ppm (588  $\pm$  28 °C) and 462 ppm (690  $\pm$ 438 18 °C), whereas rutile from type-II fracture haloes shows a narrower range between 238 ppm  $(640 \pm 23 \text{ °C})$  and 470 ppm  $(696 \pm 18 \text{ °C})$  (Figure 9b). 439 440 Rutile from the leucocratic domains shows the largest variation in Zr concentrations, ranging from 97 ppm (598  $\pm$  32 °C) to 2331 ppm (861  $\pm$  45 °C). Substructure-free areas in 441 442 large rutile grains show a slightly smaller spread in Zr concentrations between 230 ppm (637  $\pm$  23 °C) and 2331 ppm (861  $\pm$  45 °C) (Figure 9f). Shear bands have a Zr concentration of up 443 444 to 1330 ppm (797  $\pm$  31 °C), while low-angle boundaries contain between 184 ppm (620  $\pm$ 445 25°C) and 1690 ppm Zr ( $823 \pm 33$ °C) (Figure 9e). Small grains and growth twins in the 446 leucocratic domains record lower values, ranging between of 97 ppm (598  $\pm$  32 °C) to 262

Rutile Zr concentrations and calculated temperature estimates for all rutile-bearing

447 ppm (669 ± 22 °C) (Figure 9d) and 231 ppm (638 ± 26 °C) to 596 ppm (718 ± 37 °C) (Figure
448 9e), respectively.

449

### 4.6. Rutile U–Pb Geochronology

Ninety-eight analyses were obtained from 33 large grains in the leucocratic domains by LA-ICP-MS (n = 68; 28 grains) and SHRIMP (n = 30; 5 grains) (Table S4). The results are shown in Concordia and radial plots (Figure 10). All rutile grains have less than 10% common Pb, defined here as the percentage of common  $^{206}$ Pb on total  $^{206}$ Pb. Common Pb ranges between 1.7 to 9.9% for LA-ICP-MS analyses and 0.4 to 1.4% for SHRIMP analyses.

#### 455

#### 4.6.1. LA-ICP-MS analyses

All analyses yield overlapping concordant ages, producing a LA-ICP-MS Concordia age of 416.4  $\pm$  1.7 Ma (Figure 10a). By excluding LA-ICP-MS data points with more than 5% discordance (n = 4), the mean LA-ICP-MS age is 415.7  $\pm$  2.7 Ma with a mean square weighted deviation (MSWD) of 2.0 (Figure 10c). Radial plots indicate that the LA-ICP-MS data are consistent with multiple age groups. For ages obtained by LA-ICP-MS, 36% of the analyses correspond to an age of 438.2  $\pm$  5.9 Ma and 64% of the analyses correspond to an 462 age of 404.8  $\pm$  4.2 Ma (Figure 10e).

463

## 4.6.2. SHRIMP analyses

SHRIMP analyses produce a Concordia age of  $419.6 \pm 1.4$  Ma (Figure 10b). For the calculated SHRIMP mean age of  $419.2 \pm 3.3$  Ma all analyses were considered. However, the elevated MWSD of 5.6 (Figure 10d) indicates over-dispersion of the data relative to the estimated analytical uncertainty. Age data obtained by SHRIMP define two age populations of  $437.4 \pm 2.7$  Ma (39%),  $409.1 \pm 2.2$  Ma (57%) and one analysis with a younger date at  $379.9 \pm 7.2$  Ma (3.6%) (Figure 10f).

#### 470 **5. DISCUSSION**

### **5.1. Origin of rutile**

472 Rutile exhibits distinct textures and a heterogeneous distribution in the different 473 assemblages that result from localized deformation and/or hydration reactions. Large rutile is 474 particularly abundant in leucocratic domains, whereas only a small quantity of fine-grained 475 acicular to rounded rutile occurs in fracture alteration haloes and in the amphibolite (Figures 476 4, 5, and 11). The Ti required to form this fine-grained rutile in the amphibolite is most likely 477 derived from the breakdown of the diopside during hydration, as evidenced by the spatial 478 association between relict diopside and rutile (Figures 3b and 4b). Textural features, such as 479 the cuspate grain boundary morphology and the clustering of the rutile grains in the

480 leucocratic domains (Figures 5b - d) and fracture alteration haloes (Figures 4d and f) indicate 481 that rutile in these areas precipitated from a grain boundary fluid. This is consistent with the 482 general requirement of fluid for driving the retrogression of the granulite to amphibolite 483 (Austrheim & Robins, 1981). Furthermore, the correlated abundance of rutile with 484 tschermakite, clinozoisite, CO<sub>3</sub>-rich scapolite, quartz and biotite in both fracture alteration 485 haloes (Figures 4e, f) and leucocratic domains (Figure 5b) suggests a genetic link between 486 rutile precipitation and the composition of the alteration fluid resulting in the described 487 assemblage. In particular, the presence of quartz in fractures that are surrounded by rutile-rich 488 alteration haloes (Figure 4a) likely reflects cm-scale Ti mobilisation in a Si-bearing fluid. 489 Concomitant Si and Ti mobility is in agreement with experimental and natural observations 490 of enhanced rutile solubility in Na-Al-Si-bearing crustal fluids, indicating that complexing 491 with major rock-forming constituents is governing Ti mobility in fluids in addition to 492 pressure and temperature (Antignano & Manning, 2008; Gao, John, Klemd, & Xiong, 2007; 493 John, Klemd, Gao, & Garbe-Schönberg, 2008; Rapp, Klemme, Butler, & Harley, 2010). 494 Hence, the spatial affinity between rutile grains and their host lithology suggests that both 495 fractures and leucocratic domains acted as pathways channelizing Na-Al-Si-bearing fluid. 496 Without more data concerning the chemical properties of the Na-Al-Si-bearing fluid it is not 497 possible to make the distinction of whether it is in the form of a hydrous melt or an aqueous 498 solution, both of which may be stable at the determined PT conditions (see discussion below 499 for conditions) (Bureau & Keppler, 1999; Hermann, Spandler, Hack, & Korsakov, 2006; 500 Manning, 2004). Nevertheless, in remainder of the discussion the Na-Al-Si-bearing fluid is 501 distinguished from the fluid that forms the amphibolite due to a higher solute concentration.

502

#### 5.2. Deformation microstructures of rutile

Low-angle boundaries are consistent with subgrain boundaries, some of which may
have formed via crystal-plastic mechanisms. To date, very few examples of natural

505 deformation microstructures in rutile have been reported in the literature (e.g. Plavsa et al., 506 2018; Puelles et al., 2017). Nevertheless, experimental observations provide evidence for the 507 activation of two slip systems in rutile, the  $\{101\} < -101 >$  system and the  $\{110\} [001]$  system 508 (Ashbee & Smallman, 1963; Hirthe & Brittain, 1963). These earlier studies attribute the 509 activation of the different systems to the crystal orientation relative to the compressive axis 510 during deformation, independent of temperature. Following these initial investigations, 511 Blanchin, Bursill, and Lafage (1990) reported on the activation of the {101}<-101> and 512 {110}[001] slip systems at temperatures above 600°C and 900°C, respectively. However, for 513 the investigated substructured rutile grains the rotation axis orientations of [110] and [100] 514 (Figures 7f and i, respectively) combined with the subgrain boundary geometry indicate 515 activation of both the  $\{101\} < -101 >$  and the  $\{110\} [001]$  slip systems during deformation 516 (Figures 7f and i). The [112] rotation axis observed on the shear band (Figures 7b and c) may 517 be a result of the activation of multiple slip systems, unresolvable with the current EBSD 518 data. It is unlikely that temperatures exceeded 900 °C during deformation, supporting the 519 observation of temperature independence of slip system activation, thus highlighting the need 520 for further research on rutile deformation-induced microstructures.

521

#### 5.3. Conditions of rutile precipitation, deformation and recrystallization

522 The basic assumption made when applying the Zr-in-rutile thermometer is that 523 activities of Zr and Si are 1 during rutile crystallization and re-equilibration (Tomkins et al., 524 2007; Watson et al., 2006; Zack et al., 2004). This condition is usually satisfied if it can be 525 demonstrated that rutile precipitated in the presence of zircon and quartz, in which case the 526 Zr-in-rutile temperature is representative of the rutile formation temperature. By contrast, the 527 lack of zircon and/or quartz during rutile formation is equivalent to reduced Zr- and Siactivities, in which case the calculated temperature is either an overestimate ( $a_{Si} < 1$ ) or 528 529 underestimate ( $a_{Zr} < 1$ ). If the presence of zircon and quartz can be demonstrated, then the

variability of Zr concentration of rutile allows for evaluation of post-formation Zr mobilization. In our samples rutile always occurs together with quartz and zircon is only absent from the fracture halo assemblage (Figures 3, 4 and 5). Furthermore, rutile likely crystallized in the presence of a Na-Al-Si-bearing fluid. Therefore, we infer that the high Zrin-rutile temperatures reflect the temperature conditions during rutile growth and that the lower temperatures measured along deformation microstructures may be attributed to Zr mobilization during subsequent cooling.

537 Zr-in-rutile temperature estimates obtained for the amphibolite and the fracture 538 alteration haloes are between  $588 \pm 28^{\circ}$ C and  $727 \pm 17^{\circ}$ C (Figures 9a and b) and the pressure 539 range of 10-14 kbar for the crystallization of tschermakite has been estimated by amphibole 540 barometry (Table S1). This PT range is consistent with estimates of ~700°C and 9-11 kbar 541 obtained through thermodynamic modelling of the amphibolite assemblages in this outcrop 542 (Moore, Beinlich, Austrheim, & Putnis, 2019). They are also consistent with estimates of 543 690°C and 10-12 kbar for petrographically similar amphibolites of the Bergen Arcs (see 544 Figure 1a for locality; Boundy et al., 1996) that are based on the garnet-amphibole 545 thermometer of Graham and Powell (1984), the hornblende-plagioclase thermometer of 546 Blundy and Holland (1990) and garnet-rutile-ilmenite-plagioclase-silica (GRIPS) barometry 547 (Bohlen & Liotta, 1986). The consistency with previously reported metamorphic conditions 548 corroborates our PT estimate for the granulite hydration and indicates that rutile from the 549 amphibolite is likely to be in equilibrium with its host assemblage.

In contrast, Zr-in-rutile temperature estimates for the leucocratic domains in the shear zone are between  $598 \pm 32$  °C and  $861 \pm 45$ °C (Table S3; Figure 9f), which are higher than most previously reported temperatures for the amphibolites of the Lindås Nappe. However, similar values ( $874 \pm 26$ °C) are reported by Piazolo et al. (2012) using Ti-in-zircon thermometry on undeformed inherited zircons hosted within veins cross-cutting an 555 anorthosite body in the Lindås Nappe. While the authors report the possibility of an 556 erroneously high temperature due to potential integration of undetected rutile micro-557 inclusions during the zircon analysis, the similarity with our temperature estimate may also 558 represent evidence for a high temperature Si-rich fluid event in the Lindås Nappe. 559 Furthermore, temperature values obtained from deformed porphyroclastic zircon (~760 -560 820°C: Piazolo et al., 2012) are also similar to those obtained from low-angle boundaries of large rutile (~620 – 820°C; Figure 9e), reflecting deformation near peak metasomatic 561 562 temperatures.

563 The closure temperature for volume diffusion of Zr-in-rutile with a grain radius of 200 µm is between 635 °C to 725 °C, for an initial temperature of 800°C and cooling rates of 564 565 1 and 10°C/Ma, respectively (Cherniak et al., 2007; Dodson, 1973; Ganguly & Tirone, 1999). 566 Hence, even at fast cooling rates, large rutiles should have been modified by volume 567 diffusion of Zr during cooling and temperatures up to ~860°C should not have been 568 preserved. The lack of Zr diffusion profiles in rutile, i.e. Zr-depleted rims in large rutile 569 (Figure 8c) thus indicates that volume diffusion does not mobilize Zr during cooling. 570 Therefore, we infer that the high temperature recorded reflects the thermal condition during 571 rutile crystallization. However, we observe systematically lower Zr concentrations at low-572 angle boundaries compared to the undeformed parts of the same grains (Figure 8c). This 573 correlation may hint at enhanced geochemical exchange through fast-diffusion pathways in 574 areas of high dislocation density, as previously described for zircon (Timms, Reddy, Gerald, 575 Green, & Muhling, 2012), feldspar (Kramer & Seifert, 1991; Yund, Smith, & Tullis, 1981), 576 garnet (Büttner & Kasemann, 2007), and pyroxene (Chapman, Clarke, Piazolo, Robbins, & 577 Trimby; Lund, Piazolo, & Harley, 2006). Strain-enhanced diffusion occurs via 578 crystallographic ordering and disordering due to the increased atomic mobility around 579 dislocation cores (pipe diffusion). Both "static" and "dynamic" modals of strain-enhanced

580 diffusion have been proposed by previous workers. The dynamic model of strain-enhanced 581 diffusion may only occur during the creep of dislocations, elemental mobility being enabled 582 by dislocation climb or glide (Kramer & Seifert, 1991; Yund & Tullis, 1980). During creep, 583 dislocations organise into lower energy arrangements, i.e. low-angle boundaries, forming fast 584 diffusion pathways. This is the static model for strain-enhanced diffusion, where the grain 585 size is effectively decreased due to the introduction of fast diffusion pathways, lowering the closure temperature and allowing increased interaction with infiltrating fluids (Büttner & 586 587 Kasemann, 2007; Plümper et al., 2012; Reddy, Timms, Pantleon, & Trimby, 2007; Timms et 588 al., 2011). While the models are not mutually exclusive, only the dynamic model of strain-589 enhanced diffusion is in agreement with the sustained record of high temperatures (up to 590 ~820°C) along low-angle boundaries, as these temperatures indicate that diffusion of Zr 591 along dislocation cores was not effective during post-deformational cooling but during active 592 deformation. These results indicate that while volume diffusion may not effectively 593 equilibrate Zr-in-rutile, the distortion of the crystal-lattice during deformation may enhance 594 the mobility of trace elements within the affected lattice.

595 Calculated temperatures from small rutile grains from the leucocratic domains (~610 -596  $670^{\circ}$ C) and rutile in the strained amphibolite (~ $560 - 660^{\circ}$ C) are distinctly lower than those 597 of large rutile in the leucocratic domains (Figures 9c and d). These small grains, mostly 598 present within biotite porphyroblasts (Figures 4c and 5e) and around large porphyroblastic 599 rutile grains (Figure 5b), have a textural positioning that is consistent with late stage growth. 600 The low Zr-content and the textural positioning of small grains suggest that these are new 601 rutiles that precipitated at lower temperatures. Thus, the variability in Zr-in-rutile within the 602 amphibolite-facies shear zone is attributed to initial growth of rutile at high temperatures 603 followed by the remobilization of Zr along high diffusivity pathways during lattice distortion, 604 and finally, late stage growth of small rutiles at lower temperatures.

Growth twins exhibit trace element concentrations that are more similar to those
measured on low-angle boundaries (Figures 8 and 9e) than to substructure-free grains,
suggesting that twinning also enhances Zr mobility. Even though the idea of twin boundaries
acting as fast diffusion-pathways in rutile has been suggested previously (Zack & Kooijman,
2017), data with higher spatial resolution is still needed for further confirmation.

610

#### 5.4. Timing of rutile crystallization and deformation

611 We consider the two age populations defined by SHRIMP U-Pb data (Figure 10f) 612  $(437.4 \pm 2.7 \text{ Ma}, 409.1 \pm 2.2 \text{ Ma})$  as statistically valid. While the statistical analysis of the U– 613 Pb data obtained from LA-ICP-MS and SHRIMP yielded consistent age populations, the 614 relationship between age and grain microstructure can only be resolved with confidence by 615 SHRIMP data due to its superior precision and spatial resolution (Figure 10). The 616 combination of EBSD and SHRIMP data shows an apparent positive correlation of age with 617 GOS, i.e. the apparently younger grains (n = 2) exhibit a larger orientation spread than the 618 "older" undeformed grain (Figure 10f). The relationship between grain microstructure and 619 LA-ICP-MS ages is consistent with the correlation found for the SHRIMP ages (Figure 10e; 620 Table S4).

621 The resetting of U–Pb ages due to diffusive transport of mainly Pb from the rutile into 622 the surrounding matrix is controlled by temperature and the diffusion length scale (Dodson, 623 1986). Consequently, closure temperatures are grain size dependent, while the cooling rate 624 determines the timescale for diffusion to occur above the closure temperature (Cherniak, 625 2000; Kooijman et al., 2010; Vry & Baker, 2006). In addition, enhanced diffusion of U and 626 Pb as has been demonstrated to occur at low-angle boundaries in zircon (Piazolo et al., 2016; 627 Timms et al., 2011) and enhanced elemental diffusion in other deformed silicates such as pyroxene, feldspar and garnet (e.g. Chapman, Clarke, Piazolo, Robbins, & Trimby, 2019; 628 629 Lund et al., 2006; Yund et al., 1981). The continued post-deformational loss of Pb is in

agreement with a static strain-enhanced diffusion model rather than a dynamic one (as
described above for Zr). The presence of low-angle boundaries in rutile grains is expected to
reduce the diffusion length scale, thus enhancing U and Pb transport, equivalent to lowering
the closure temperatures for deformed grains versus undeformed grains of equal size. In such
a scenario low-angle boundaries enhance element mobility and enable continuous reequilibration during fluid-assisted deformation.

636 Indeed, U concentrations at low-angle boundaries and in growth twins (Figure 8a) are 637 too low for reliable age determination. This indicates a likely mobility of U associated with 638 the formation of low-angle boundaries subsequent to lattice distortion, suggesting that U 639 mobility was syn-deformational. Due to its smaller ionic radii, Pb should diffuse much faster 640 than U (Dowty, 1980; Fortier & Giletti, 1989), as a result Pb-loss should have continued after 641 crystal-plasticity of rutile became inactive, if the rocks remained at mid-crustal temperatures. 642 This does not however, reconcile the static diffusion of Pb versus the dynamic diffusion of U 643 and Zr, the contrasting modes of mobility insinuating that there must be a further 644 crystallographic influence on the diffusion of these elements, unresolvable with the data 645 presented here. Thus, deformation-induced open system behaviour of both U and Pb provides 646 an explanation for the bimodality of the measured rutile age data, the younger age reflecting the eventual closure for Pb diffusion in substructured grains. 647

648

## 5.5. Temperature-time-deformation path

649 The field relationships, temperature conditions, fluid composition and the

650 geochronological data can be integrated to develop a comprehensive thermal and deformation

- 651 chronology for the amphibolites of the Lindås Nappe. The rutile microstructures and
- 652 corresponding U–Pb and Zr-in-rutile analyses are illustrated in Figure 11.

653 5.5.1. Stage 1 – Syn-tectonic fluid infiltration at lower to mid-crustal conditions The location of type-I and –II fractures in the outcrop (Figure 2b) together with 654 655 assemblages present within the fractures and the surrounding alteration haloes suggest that 656 they either represent (1) a record of initial fluid infiltration along brittle fractures and were 657 subsequently overprinted by hydration and deformation and/or (2) localized sites of brittle 658 failure during hydration within the dry protolith. Both scenarios are consistent with syn-659 deformation fluid infiltration as has been suggested as the general mechanism for hydration 660 of the Lindås Nappe (Austrheim, 1987; Jamtveit et al., 1990). Syn-tectonic fluid infiltration is 661 further supported by the subparallel alignment of biotite, clinozoisite and rutile 662 porphyroblasts in the leucocratic domains (Figures 5a, b, and e). Furthermore, the presence of 663 similar mineral assemblages and the higher rutile abundance in the fracture haloes and the 664 leucocratic domains suggest alteration of the granulite by a similar or even the same fluid. 665 This relationship between the presence of the rutile-bearing assemblage and localization of 666 fluid and deformation in the shear zone, producing the leucocratic domains, is summarised 667 schematically in Figure 11a. Since the interlayering and consistent foliation of the 668 amphibolite and the leucocratic domains (Figures 2a and 5a) indicate contemporaneous 669 deformation of both lithologies, hydration is assumed to begin with the breakdown of the 670 diopside in the granulite to form the amphibolite (Figure 11a). Where syn-deformational 671 reaction continues at amphibolite-facies conditions, the rutile-bearing assemblage 672 (tschermakite, clinozoisite, guartz and biotite) dominates.

Following rutile crystallization, large grains underwent crystal-plastic deformation in the presence of fluid (Figures 11a and b). Evidence of element transport associated with crystal-plastic deformation of rutile indicates that syn-tectonic fluid infiltration along fractures initiated the formation of the shear zone. Following initial fluid infiltration, the continuous syn-deformational re-equilibration of the rutile Zr concentrations is evidenced by the spread of Zr-in-rutile temperatures between 620 to 820°C measured on rutile low-angle
boundaries in the leucocratic domains (Figures 9 and 11b).

680 Rutile lattice deformation concomitant with re-equilibration of U resulted in the 681 observed low concentrations at low angle boundaries (Figure 8a). Since the temperature 682 during deformation was significantly higher than the Pb diffusion closure temperature of 683 approximately 600°C (Cherniak, 2000; Vry & Baker, 2006), it is unlikely that U-Pb in both 684 substructured and substructure-free rutile reached closure until the end of deformation. The 685  $437 \pm 2.7$  Ma age is therefore interpreted to reflect the minimum age of deformation, 686 recorded in large substructure-free rutile in the leucocratic domains (Figure 11b). 687 Ages of  $455 \pm 2$  Ma and  $439 \pm 4$  Ma recorded by Ar-Ar in amphibole (Boundy et al., 688 1996) have previously been interpreted to be erroneous due to possible excess Ar in the 689 amphibole (Kühn et al., 2002; Roffeis et al., 2012). Alternatively, they may also reflect the 690 first stage of fluid infiltration as recorded by rutile in the leucocratic domains. A similar re-691 interpretation of phengitic muscovite ages in Lindås Nappe eclogites ( $433\pm1$  to  $429\pm1$ ; 692 Boundy et al., 1996) was suggested by Bingen et al. (2004), based on evidence for the 693 retention of radiogenic Ar in phengite in high-pressure environments at relatively high 694 temperatures (> 500°C) (Di Vincenzo, Tonarini, Lombardo, Castelli, & Ottolini, 2006; 695 Giorgis, Cosca, & Li, 2000; Rodriguez, Cosca, Ibarguchi, & Dallmeyer, 2003).

However, the coeval formation of leucocratic domains and amphibolite does not reconcile with the distinct temperature values recorded by rutile from the two lithologies. Zrin-rutile temperatures range from 640 to 860°C for large substructure-free grains in the leucocratic domains and 610 to 730 °C for the amphibolite (Figures 9a, f). Since a 130 °C temperature gradient over a distance of only 0.5 m appears inconsistent with a regional-scale thermal state, further explanation is required to account for the high temperatures recorded in the shear zone. Three explanations are considered: (1) Zr is undersaturated in the amphibolite, resulting in an underestimation of the temperature by Zr-in-rutile thermometry
for the statically hydrated wall rock of the shear zone; (2) rutile temperatures from the shear
zone reflect infiltration of an externally derived high temperature fluid; (3) the high
temperatures recorded by rutile from the shear zone reflect rutile formation during shear
heating.

708 In the first explanation, temperature is controlled by the regional thermal state but 709 rutile in the static amphibolite formed in Zr-unsaturated conditions and was therefore unable 710 to reach the equilibrium concentration of Zr consistent with the temperature of formation 711 (Watson et al., 2006). In this scenario, localized flow of Si-Al-Na-bearing fluid within the 712 leucocratic domains facilitates transport of normally immobile elements through the 713 deforming rock volume, thus enhancing the availability of Zr within Si-Al-Na-bearing fluid 714 hosted zones. In contrast to option two, which requires an external fluid for heat input, in this 715 option a hydrous fluid with low thermal mass could infiltrate the rock and produce an 716 internally derived Si-Na-Al-bearing fluid along fractures pathways. The Si-Na-Al-bearing 717 fluid, being of higher viscosity than the hydrous fluid, would be restricted to these fracture 718 pathways, resulting in temperature and consequently textural and mineralogical differences 719 that are coupled to strain variations as observed between the amphibolite and leucocratic 720 domains and localized re-equilibration of trace element concentrations.

The second explanation is that the localized occurrence of high-temperature rutile is due to the channelization of externally derived hot Na-Al-Si-bearing fluid along a deformation-induced high permeability zone (e.g. Daczko, Piazolo, Meek, Stuart, & Elliott, 2016). Modelling of heat transfer by fluids suggests that conduction or advection of heat requires a large, channelized, continuous flux and/or repeated pulses of high temperature fluid (Bickle & McKenzie, 1987; Brady, 1988; Hoisch, 1991; Thompson & Connolly, 1990). Fluid channelization is consistent with the distribution of leucocratic domains, appearing as 728 highly localized zones of distinct composition in outcrop and thin section (Figures 2a and 4a). 729 Fluid channelization most effectively occurs under high strain, where the partitioning of 730 strain into discrete zones of deformation couples with fluid-flow (Mancktelow, 2006). To 731 account for the coupling between high strain and fluid flow the initial infiltration must have been syn-tectonic, resulting in localized mechanical weakening and subsequent fluid 732 733 channelization along optimally orientated pathways. This link between deformation and syn-734 tectonic fluid channelling has been recently shown to occur in the lower continental crust 735 (Daczko et al., 2016; Meek, Piazolo, & Daczko, 2019; Stuart, Meek, Daczko, Piazolo, & 736 Huang, 2018).

737 In the third option, increased temperature coupled with strain is accounted for by shear heating. Shear heating occurs at relatively high shear stresses (>100 MPa) and strain 738 rates  $(10^{-11} - 10^{-12} \text{ s}^{-1})$  (Molnar & England, 1990) and is the result of the conversion of 739 740 mechanical energy into heat during progressive deformation (Brun & Cobbold, 1980). 741 Temperature increases in shear zones as result of shear heating have been inferred to be in the 742 order of 100 – 200°C above ambient conditions (Camacho, McDougall, Armstrong, & Braun, 743 2001; Whittington, Hofmeister, & Nabelek, 2009). The coincidence here between the 744 deformation zone and higher temperatures (up to ~860°C; Figures 9 and 11b) is in agreement 745 with these previous estimates. At ambient crustal temperatures exceeding ~600°C heat 746 produced by shear heating may be retained due to thermal insulation (Whittington et al., 747 2009). In fact, thermo-mechanical modelling indicates that during intermittent stress 748 relaxation the shear zone temperature remains elevated and progressively increases toward 749 the peak temperature with successive shear heating events (Kelemen & Hirth, 2007). This is 750 consistent with the range of Zr-in-rutile temperatures recorded in substructure-free areas of 751 deformed porphyroblastic rutile (620-820°C; Figures 9e and 11b). In contrast to the second

explanation, Si-Al-Na-bearing fluid is produced internally by dissolution of the rock duringdeformation.

754 Based on the data presented in this study none of the above explanations can be 755 excluded with certainty. However, the consistency of temperatures of 610 - 730°C recorded 756 by rutile in the statically hydrated amphibolite with previous estimates of regional peak-757 metamorphic conditions (~690 °C) of the Bergen Arcs (Bhowany et al., 2018; Boundy et al., 758 1996; Glodny et al., 2008) supports a scenario where rutile formed above ambient 759 temperature within the shear zone, requiring either shear heating or heat advection through an 760 externally derived fluid. Crystallization ages of 428±8 Ma and 422±17 Ma (Kühn et al., 761 2002) and a U-Pb zircon age of 424±1 Ma (Jamtveit et al., 2018) for pegmatites within the 762 Lindås Nappe further support the transport of Na-Al-Si-bearing fluids at a time approximate 763 to the amphibolite-facies event. However, whether the emplaced pegmatites are derived 764 externally or from internal reactions remains ambiguous.

765

#### 5.5.2. Stage 2 – Exhumation to the upper-crust

The  $404.8 \pm 4.5$  Ma and  $409.1\pm 2.2$  Ma LA-ICP-MS and SHRIMP rutile ages agree 766 with reported Rb-Sr ages of 413±4 Ma (Glodny et al., 2008) and 409±8 Ma (Bingen et al., 767 768 2001). These ages are interpreted to record the cessation of diffusional re-equilibration of Pb 769 along low-angle boundaries during exhumation. According to estimated closure temperatures 770 for Pb-loss from rutile, the investigated rocks must have resided at temperatures in the range 771 of ~500-700°C (Cherniak, 2000; Kooijman et al., 2010; Vry & Baker, 2006). Any temperature in excess of this range would have resulted in complete resetting of the 772 773 substructure-free grains, indicating Pb-loss at mid-crustal temperature conditions for a period 774 of c. 30 My following Stage 1 deformation. In addition, the c. 410-405 Ma age for exhumation is additionally consistent with  ${}^{40}\text{Ar}/{}^{39}\text{Ar}$  plateau ages of *c*. 410 Ma from Fossen 775 and Dunlap (1998), interpreted as the last stage of nappe stacking and cooling below ~500°C. 776

The eventual cessation of Pb-loss at *c*. 410-405 Ma therefore marks the exhumation to uppercrustal conditions where thermally driven Pb loss is no longer operative.

## 779 6. CONCLUSIONS/IMPLICATIONS

780 Rutile from the Bergen Arcs amphibolite-facies shear zone records not only the cooling 781 history of the amphibolites but also the timing and temperature of deformation. Combined 782 microstructural and geochemical analyses of rutile allow a reconstruction of the multi-stage 783 history of the shear zone. While mid-crustal conditions (~610–730°C) are recorded in the 784 statically hydrated wall rock of the shear zone, syn-tectonically precipitated rutile in the 785 dynamically hydrated shear zone records a localized temperature increase to ~860°C. 786 Whether this temperature increase was driven by externally derived high-temperature fluids 787 or by shear heating remains to be investigated. Progressive fluid-assisted deformation 788 resulted in the localized resetting of U-Pb ages and slightly lower Zr-in-rutile temperatures of 789  $\sim 820^{\circ}$ C along deformation structures in deformed grains from the shear zone. Finally, 790 crystal-plastic deformation subsides at conditions consistent with the statically hydrated rock 791 (~ 560–670°C) accompanied by the formation of small (< 150  $\mu$ m) rutile in the strained 792 lithologies. This is estimated to be concomitant with the cessation of U mobility along low-793 angle boundaries at  $437.4 \pm 2.7$  Ma. The final closure of Pb diffusion from rutile is recorded 794 at c. 410–405 Ma, indicating that the rocks remained at mid-crustal conditions (~500–700°C) 795 during the interim period.

Our findings indicate that U–Pb ages and trace element concentrations in rutile may be strongly dependent on microstructure. In this particular instance both U–Pb and Zr-inrutile show systematic changes with crystal-lattice distortion. Young ages are consistently correlated with significant crystal-plastic deformation and low-angle boundaries have lower Zr concentrations than substructure-free domains in rutile. Low-angle boundaries are therefore proposed to act as fast diffusion pathways in rutile, allowing for enhanced element

802	mobility. Therefore, in instances where the trace element distribution and U-Pb ratios are
803	controlled by fast diffusion due to deformation, rutile can be a useful tool for constraining a
804	near complete deformation and cooling history of the rock.

805

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- 1128 LIST OF SUPPORTING INFORMATION
- 1129 Supporting information is in the form of data tables and can be found in the document
- 1130 Supdataset.xlsx. The data tables provided are as follows:
- 1131 Figure S1 c-axis orientation of large rutile
- 1132 Table S1 Representative amphibole
- 1133 Table S2 LA-ICP-MS trace element
- 1134 Table S3 EMPA Major and trace element
- 1135 Table S4a LA-ICP-MS U-Pb
- 1136 Table S4b SHRIMP U-Pb

#### 1137 FIGURE CAPTIONS

1138 FIGURE 1 – (a) Regional setting of study area at Radøy in the Lindås Nappe of the Bergen

1139 Arcs, Western Norway. The outcrop locality is indicated by the white star. The locality of

1140 amphibolite-facies samples collected for age data and P-T estimates by previous workers are 1141 annotated on the map with symbols corresponding to those used in (b) and numbered 1142 according to the references listed below. MCTZ = Main Caledonian Thrust Zone. BASZ = 1143 Bergen Arcs Shear Zone. Modified from Boundy, Mezger, and Essene (1997), Glodny et al. 1144 (2008) and Centrella, Austrheim, and Putnis (2015). (b) Summary of previous 1145 geochronological work for Caledonian age events affecting the Lindås Nappe. Amphibolite 1146 zircon U–Pb data are from <sup>1</sup>Kühn et al. (2002), <sup>2</sup>Roffeis et al. (2012), <sup>3</sup>Piazolo et al. (2012), and <sup>4</sup>Glodny et al. (2008). Multi-mineral Rb-Sr and Sm-Nd data for amphibolites are from 1147 1148 <sup>5</sup>Glodny, Kühn, and Austrheim (2002), <sup>4</sup>Glodny et al. (2008) and <sup>8</sup>Austrheim (1990). 1149 Amphibole and white mica Ar-Ar data are from <sup>6</sup>Boundy et al. (1996), biotite Ar-Ar data are 1150 from <sup>7</sup>Fossen and Dunlap (1998). Rb-Sr and Sm-Nd Pegmatite ages were determined by <sup>1</sup>Kühn et al. (2002) and the U–Pb zircon age is from <sup>9</sup>Jamtveit et al. (2018). Eclogite U–Pb 1151 1152 data are from the following sources: U-Pb zircon (Bingen et al., 2004; Bingen et al., 2001; 1153 Glodny et al., 2008), titanite-epidote-zircon U-Pb (Boundy et al., 1997). Error bars represent 1154  $2\sigma$  uncertainty. 1155 FIGURE 2 – Lithological relationships in outcrop. (a) The porphyroblastic rutile-bearing

1155 FIGURE 2 – Enhological relationships in outcrop. (a) The porphyroblastic ruthe-bearing
1156 lithology, the leucocratic domains, are observed interlayered with the amphibolite in the shear
1157 zone. (b) Fracture type-I and type-II in the granulite in outcrop. Sample locations are
1158 indicated by red boxes, annotated with image figure numbers.

1159 FIGURE 3 – Photomicrographs of the main lithologies (a) granulite (b) amphibolite, here

1160 incompletely reacted, (c) strained amphibolite and (d) the shear zone rock comprised of the

amphibolite interlayered with leucocratic domains. Scale bar is 5 mm.

1162 FIGURE 4 – Rutile occurrence in amphibolite and in association with fracture haloes in the

1163 granulite. (a) Fracture type-I, hosting quartz and tschermakite, crosscutting horizontal

foliation in partially replaced granulite. BSE images (b-f) showing (b) rutile needles in

amphibole clusters in the amphibolite (c) rutile associated with both amphibole clusters and
biotite and clinozoisite in the strained amphibolite (d) rutile in amphibole clusters in
alteration halo of fracture type-I, and, (e) rutile forming in a symplectitic assemblage of
magnesio-hornblende quartz and zoisite replacing diopside on the edge of the fracture type-I
alteration halo. (f) Rutile in a clinozoisite and pargasite filled fracture in diopside, occurring
in the alteration halo of fracture type-II.

1171 FIGURE 5 - Rutile distribution in leucocratic domains, interlayered with strained 1172 amphibolite in the shear zone. (a) Photograph of sample showing the location of 1173 microtextural analyses. Note the bottom to top increase in the proportion of the leucocratic 1174 component. BSE images (b-f) illustrate the mineralogical changes with increased leucocratic 1175 component. (f) Original amphibole clusters display a lower abundance of rutile and biotite 1176 then (d) amphibole clusters interlayered with the leucocratic component. Porphyroblastic 1177 rutiles are apparent in both (b) the mafic (c) and plagioclase dominated components of the 1178 leucocratic vein. Within leucocratic domains porphyroblastic clinozoisite and biotite form in 1179 (b) foliation parallel mafic clusters and in (e) foliation parallel bands. All images are aligned 1180 with the S foliation parallel to that shown in (a).

1181**FIGURE 6** – Microstructures of substructure-free rutile associated with leucocratic domains;1182rutiles are characterized by low GOS (< 1°) and lack of subgrain boundaries; (a-d) EBSD</td>1183phase maps with grain, subgrain and twin boundaries in rutile show (a, c) grain morphology1184and phase associations of large substructure- free grains in both the plagioclase dominated1185leucocratic domain and, (b) in the preserved amphibole banding, as well as, (d) small1186substructure-free grains in the extensional veinlets. GOS: Grain orientation spread (°). Grain1187numbering applies to grains with reported U–Pb data (see Table S4).

1188 **FIGURE 7** – Microstructures of large substructured rutile grains in the leucocratic domains.

1189 For visualisation of substructures, representative large substructured grains are presented as

1190 EBSD maps showing the deviation of each pixel from the mean orientation of the grain (a, e 1191 & h). Presented here are grains showing (a) shear bands, (e) lamellar subgrain boundaries and 1192 (h) low-angle boundaries related to twinning at grain edges. Lower hemisphere equal area 1193 plots (b, f, & i) exhibit disorientation axes relative to the crystallographic directions. (b) 1194 disorientation axes for the entire grain shown in (a); (f and i) disorientation axes for selected 1195 areas in (e) and (h) that are outlined by stippled boxes. For (a) the shear band the (b) 1196 disorientation axes cluster on the <112> axis but show no correlation with known slip 1197 systems while for (f) disorientation axes plot at <110>, consistent with operation of the 1198 <001>{110} slip system and (i) and at <100> consistent with the {101}<-101> slip system. 1199 Plotted grain orientation axes (c), coloured according to the misorientation in the EBSD map, 1200 further exhibit that the principal rotation axis for the sheared grain is  $\sim <112>$ . Cumulative 1201 misorientation profile (d, g) showing the misorientation across the subgrain boundaries 1202 associated with the shear band (d) and the lamellar subgrain boundaries (g). GOS: Grain 1203 orientation spread (°). Grain numbering applies to grains with reported U–Pb data (see Table 1204 S4).

1205 FIGURE 8 – LA-ICP-MS trace element spot analyses plotted by distance from grain 1206 boundary. Individual analyses are coloured to represent the grain structure of the analysis 1207 spot. Shaded grey areas indicate the spread of the data from low-angle boundaries (LAB) 1208 (darker) and substructure free grains (lighter). The weighted average concentration for trace 1209 elements measured from substructure free spots within 80 µm of the grain boundary in large 1210 grains (grey stippled line) is compared to the weighted average value of spots containing low-1211 angle boundaries (cyan stippled lines). Dotted lines represent the mean  $2\sigma$  uncertainty. 1212 FIGURE 9 - Frequency histograms displaying Zr-in-rutile temperature estimates for 1213 individual EMP and LA-ICP-MS analyses in all lithologies (a-f).

1214 FIGURE 10 - U-Pb data for porphyroblastic rutile in leucocratic domains obtained via LA-1215 ICP-MS (a, c, and e) and SHRIMP (b, d, and f) analysis. (a, b) U–Pb concordia diagrams, 1216 ellipses representing the 2-sigma uncertainty. (c, d) Weighted mean age of individual 1217 analyses. (e, f) Radial plot exhibiting dispersion of U-Pb ages, coloured according to GOS; 1218 for graphical method see Galbraith (1988); Galbraith (1990). The grey coloured analyses in 1219 (f) represent a grain with no corresponding EBSD data. In weighted mean and radial plots 1220 data are restricted to analyses within 5% discordance, eliminating 4 of the LA-ICP-MS 1221 analyses.

1222 FIGURE 11 - Characteristic textural setting of rutile in each lithology, showing correlation 1223 between rutile growth, recorded temperature and strain. (a) Schematic illustration of (i) the 1224 microstructure of rutile in the amphibolite where it forms as part of a symplectitic assemblage 1225 replacing diopside, (ii) rutile in strained and shear zone amphibolite where it occurs with 1226 biotite and quartz in amphibole and (iii) large and small rutile replacing amphibole in the 1227 leucocratic domains of the shear zone due to increasing deformation and reaction. (b) 1228 Schematic of deformation microstructures in large rutile in the leucocratic domains and box 1229 plots showing the corresponding spread in Zr-in-rutile temperatures for microstructures and 1230 small grains.

1231























#### (a) Textural setting of rutile by lithology





Figure S1 - C-axis orientation of all large rutile grains in respect to the cutting plane.

Litholology	Fracture type-I halo	Fra	acture type-II halo Ai	mphibolite	Str	Strained amphibolite		
Calcic amphibole classification	tschermakite	Mg-hornblende	pargasite	tschermakite	Mg-hornblende	Mg-hornblende		
Technique	EDS	EDS	WDS	WDS	WDS	WDS		
P estimate (kbar)								
Johnson and Rutherford, 1989 (±0.5 kbar)	11.6	6.0	12.4	13.6	7.8	12.7		
Schmidt 1991 (±0.6 kbar)	10.6	4.3	11.5	12.8	6.3	11.8		
Wt% oxide								
SiO <sub>2</sub>	43.2	48.8	42.2	41.5	48.0	44.7		
TiO <sub>2</sub>	0.330	0.650	0.143	0.147	0.0836	0.144		
Al <sub>2</sub> O <sub>3</sub>	17.0	9.20	17.6	19.4	11.6	16.8		
Cr <sub>2</sub> O <sub>3</sub>	b.d.l.	b.d.l.	b.d.l.	0.0348	b.d.l.	b.d.l.		
Fe <sub>2</sub> O <sub>3</sub>	4.55	3.45	0.594	0.939	1.73	#REF!		
FeO	7.03	3.23	9.75	9.71	7.55	9.02		
MnO	b.d.l.	b.d.l.	0.116	0.117	0.166	0.206		
MgO	12.8	18.0	11.4	10.9	14.6	11.8		
CaO	11.9	13.1	12.1	12.2	12.0	11.3		
Na <sub>2</sub> O	1.60	0.690	1.50	1.48	1.08	1.72		
K <sub>2</sub> O	0.270	0.250	0.720	0.503	0.217	0.745		
CI	0.260	b.d.l.	0.341	0.206	0.0714	0.324		
F	b.d.l.	b.d.l.	0.01	b.d.l.	b.d.l.	b.d.l.		
0	n.m.	n.m.	0.00	n.m.	n.m.	n.m.		
H <sub>2</sub> O	2.02	2.14	1.98	2.02	2.10	2.01		
Total	101	99.5	98.5	99.1	99.3	100		

Litholology Calcic amphibole classification Technique	Leucocratic domain <b>tschermakite</b> WDS	<b>Mg-hornblende</b> WDS
P estimate (kbar)		
Johnson and Rutherford, 1989 (±0.5 kbar)	12.7	11.1
Schmidt 1991 (±0.6 kbar)	11.9	10.1
Wt% oxide SiO <sub>2</sub>	42.1	44.6
TiO <sub>2</sub>	0.246	0.390
Al <sub>2</sub> O <sub>3</sub>	18.0	16.1
Cr <sub>2</sub> O <sub>3</sub>	0.0177	b.d.l.
Fe <sub>2</sub> O <sub>3</sub>	2.07	#REF!
FeO	11.2	11.2
MnO	0.180	0.206
MgO	9.90	10.8
CaO	11.7	11.7
Na <sub>2</sub> O	1.50	1.44
K <sub>2</sub> O	0.489	0.543
CI	0.238	0.205
F	b.d.l.	0.06
0	n.m.	0.00
H <sub>2</sub> O	2.00	2.00
Total	99.6	100

		Growth twin						Shear plane		
Grain number**		25		No U-Pb		No U-Pb		6		11
	Elements (ppm) (MDL filtered)	7_G1-1	±2σ	7_B-3	±2σ	2_H-1	±2σ	1_5-7	±2σ	3_K-4
Analyses	Si	498	180	346	150	252	150	311	170	446
	Fe	4400	940	5010	1200	6860	990	7220	1000	6000
	U	4.26	1.10	1.26	0.34	0.22	0.040	3.65	0.56	3.04
	V	777	190	479	130	336	53	807	130	790
	Zr	260	61	596	150	231	35	322	48	1090
	Hf	7.59	1.8	25.6	6.6	10.3	1.5	11.5	1.7	41.7
	Cr	49.8	12	42.1	11	16.5	3.2	47.1	7.8	75.0
	Nb	374	88	938	240	1840	270	854	130	1542
	Та	16.8	4.0	46.0	12	87.3	13	36.0	5.4	92.9
	Zn	136	36	4.78	4.5	8.72	4.1	12.6	4.7	6.00
	Мо	180	42	55.4	14	33.5	5.1	58.2	8.8	49.7
	W	58.3	14	47.1	12	76.8	12	31.4	4.9	44.7
	Sb	1.57	0.44	1.40	0.42	2.44	0.44	1.34	0.28	1.34
	Sn	8.72	2.2	11.0	3.0	10.2	1.7	10.5	1.7	13.5
Ratios	Nb/Ta	22.2		20.4		21.1		23.7		16.6
	Zr/Hf	34.2		23.3		22.5		27.9		26.1
Thermometry	Ln (Zr)	5.56		6.39		5.44		5.77		6.99
	P (Kbars)	12		12		12		12		12
	Temperature (°C)	647	33	718	37	638	26	664	26	776
Distance from grain bo	undary (µm)	0.1		0.1		45		0.1		24
	GROD	56		3.4		1.9		4.46		1.5

					Low angle b	oundaries					
Grain number**			11		4		7		No U-Pb		No U-Pb
	Elements (ppm) (MDL filtered)	±2σ	3_K-6	±2σ	1_4-5	±2σ	2_D-1	±2σ	4_BI-4	±2σ	7_K-1
Analyses	Si	170	364	150	642	200	1084	290	1703	450	329
	Fe	890	5480	820	3610	510	3440	500	3740	610	3730
	U	0.50	1.79	0.30	1.23	0.19	4.25	0.68	2.60	0.48	0.09
	V	130	790	130	825	130	515	83	456	83	625
	Zr	170	1330	210	184	27	1030	150	1690	290	631
	Hf	6.4	48.9	7.6	5.45	0.80	30.4	4.6	37.2	6.4	16.3
	Cr	12	61.0	10	13.0	2.6	30.6	5.3	43.1	8.1	13.1
	Nb	240	1702	260	481	69	1500	230	1330	230	255
	Та	14	94.0	15	13.6	2.0	64.7	9.8	69.7	12	13.1
	Zn	4.1	3.68	3.8	1.28	3.0	11.9	4.5	4.07	4.1	9.53
	Мо	7.8	57.9	9.1	4.66	0.78	59.6	9.1	18.0	3.2	17.2
	W	7.2	42.4	6.8	77.1	12	49.6	7.8	99.4	18	57.2
	Sb	0.28	1.52	0.30	2.37	0.42	1.72	0.34	2.21	0.46	2.26
	Sn	2.3	15.3	2.5	6.34	1.0	13.8	2.2	5.82	1.1	7.55
Ratios	Nb/Ta		18.1		35.3		23.2		19.1		19.4
	Zr/Hf		27.2		33.8		33.8		45.5		38.8
Thermometry	Ln (Zr)		7.19		5.22		6.94		7.43		6.45
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	29	797	31	620	25	770	28	823	33	723
Distance from grain bo	undary (µm)		46		9		20		0.1		7
	GROD		2.47		1.21		6.99		24.6		6.33

									Small grains	5	
Grain number**			No U-Pb		No U-Pb		22		No U-Pb		No U-Pb
	Elements (ppm) (MDL filtered)	±2σ	3_l-1	±2σ	5_B-1	±2σ	6_H3-3	±2σ	4_BI-1	±2σ	4_G-1
Analyses	Si	150	282	140	445	190	486	180	66.9	130	1366
	Fe	790	3330	500	3890	700	6920	1400	4130	660	2790
	U	0.024	0.301	0.054	0.21	0.050	9.41	2.20	0.06	0.016	b.d.l
	V	150	1110	180	355	71	718	160	473	84	525
	Zr	150	267	42	618	120	1580	350	166	28	153
	Hf	3.8	11.4	1.8	27.2	5.2	42.9	9.5	8.37	1.4	6.90
	Cr	3.6	32.3	5.7	86.2	18	66.6	16	42.4	7.9	16.5
	Nb	59	632	99	939	180	873	190	1810	310	353
	Та	3.1	21.6	3.4	58.7	11	33.5	7.5	105	18	32.3
	Zn	4.9	6.38	4.0	8.41	5.4	7.07	4.4	3.33	3.7	2.31
	Мо	4.0	13.5	2.2	40.8	7.9	78.5	17	23.4	4.1	3.85
	W	14	101	16	37.3	7.4	43.5	9.9	78.0	14	103
	Sb	0.60	2.59	0.50	2.24	0.52	1.00	0.28	1.92	0.40	1.49
	Sn	1.9	8.2	1.4	11.4	2.4	13.2	3.1	8.23	1.5	3.73
Ratios	Nb/Ta		29.3		16.0		26.0		17.3		10.9
	Zr/Hf		23.3		22.7		36.8		19.8		22.2
Thermometry	Ln (Zr)		5.59		6.43		7.37		5.11		5.03
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	35	649	26	721	31	816	39	613	26	607
Distance from grain boundary	(µm)		19		13		79		6		1
	GROD		1.07		5.17		0.225		22.9		0.265

			Amphibole c		Large grains in leucocratic domain						
Grain number**			24		24		1		2		2
	Elements (ppm) (MDL filtered)	±2σ	7_E-3	±2σ	7_E-4	±2σ	1_1-1	±2σ	1_2-2	±2σ	1_2-4
Analyses	Si	380	516	190	276	140	307	160	718	250	435
	Fe	490	4310	980	3620	820	4950	700	6650	940	5740
	U		7.28	1.9	2.21	0.58	18.4	2.8	14.1	2.1	9.32
	V	100	682	170	669	170	787	120	891	140	881
	Zr	28	2330	580	2080	520	1690	240	1390	200	1270
	Hf	1.3	50.5	13	49.9	13	44.9	6.5	43.2	6.3	42.4
	Cr	3.7	14.4	4.1	13.3	3.9	32.0	5.4	46.2	7.7	42.1
	Nb	65	207	51	202	50	966	140	1040	150	1060
	Та	6.1	7.20	1.8	7.38	1.9	47.8	7.0	44.2	6.5	46.6
	Zn	4.1	1.79	4.7	16.7	6.6	4.40	3.5	6.64	4.7	5.43
	Мо	0.86	35.8	8.8	30.4	7.6	60.4	8.9	64.8	9.7	65.2
	W	20	11.7	3.0	13.0	3.4	25.9	3.9	32.5	4.9	32.7
	Sb	0.36	1.14	0.34	1.38	0.40	1.05	0.22	1.20	0.26	1.16
	Sn	0.80	15.8	4.1	14.6	3.9	15.5	2.4	15.5	2.5	14.2
Ratios	Nb/Ta		28.8		27.3		20.2		23.5		22.7
	Zr/Hf		46.2		41.7		37.6		32.2		30.0
Thermometry	Ln (Zr)		7.75		7.64		7.43		7.24		7.15
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	27	861	45	847	43	823	30	802	30	792
Distance from grain bo	undary (µm)		67		27		36		165		36
	GROD		0.125		0.595		0.585		0.865		0.255

Grain number**				3		4		5		5	
	Elements (ppm) (MDL filtered)	±2σ	1_3-1	±2σ	1_4-1	±2σ	1_5-2	±2σ	1_5-4	±2σ	2_D-3
Analyses	Si	210	354	170	705	210	1280	390	612	290	492
	Fe	810	8660	1200	7160	1000	7650	1100	5160	730	4900
	U	1.4	8.31	1.3	12.3	1.8	10.6	1.6	4.14	0.64	8.22
	V	140	285	45	870	130	927	140	895	140	557
	Zr	180	1280	190	1730	250	2100	310	1160	170	1670
	Hf	6.2	41.6	6.2	45.0	6.5	50.3	7.4	31.5	4.6	42.4
	Cr	7.1	31.0	5.4	18.4	3.3	45.3	7.8	46.1	8.0	32.3
	Nb	150	1330	200	2050	300	1230	180	1230	180	1540
	Та	6.9	66.3	9.9	155	23	53.8	7.9	53.0	7.9	65.7
	Zn	4.7	5.92	4.2	b.d.l.	3.0	13.5	6.7	12.4	6.8	12.6
	Мо	9.7	66.3	10	68.3	10	92.0	14	77.0	12	73.6
	W	5.0	41.2	6.4	42.3	6.3	55.6	8.5	56.0	8.6	52.8
	Sb	0.24	1.03	0.22	1.11	0.22	1.11	0.26	1.73	0.36	1.16
	Sn	2.3	15.3	2.5	17.6	2.7	17.1	2.7	15.1	2.4	15.4
Ratios	Nb/Ta		20.1		13.2		22.9		23.2		23.4
	Zr/Hf		30.8		38.4		41.7		36.8		39.4
Thermometry	Ln (Zr)		7.15		7.46		7.65		7.06		7.42
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	29	793	30	826	30	849	31	783	28	822
Distance from grain bo	undary (µm)		108		152		113		10		17
	GROD		0.485		2.04		0.295		0.415		1.23

Grain number**			8		9		10		11		No U-Pb
	Elements (ppm) (MDL filtered)	±2σ	2_G-1	±2σ	3_C-1	±2σ	3_G2-1	±2σ	3_K-1	±2σ	4_BII-1
Analyses	Si	180	277	150	536	180	988	280	331	160	462
	Fe	710	5480	800	3980	600	6970	1000	6700	990	4990
	U	1.3	11.4	1.8	4.31	0.72	6.43	1.1	15.2	2.4	0.86
	V	90	964	150	978	160	438	72	800	130	491
	Zr	250	1240	190	1470	230	1820	290	1140	170	743
	Hf	6.4	36.7	5.5	43.0	6.8	41.9	6.5	41.9	6.4	34.9
	Cr	5.5	60.3	9.9	48.9	8.4	34.3	6.1	69.7	12	38.1
	Nb	230	899	130	1310	210	1670	260	1700	260	1860
	Та	10	42.8	6.5	67.7	11	97.7	15	92.1	14	91.7
	Zn	4.6	13.1	4.6	2.01	3.9	6.45	4.9	6.52	4.3	2.42
	Мо	11	69.0	11	50.4	8.1	70.0	11	76.9	12	40.3
	W	8.3	32.5	5.1	42.6	7.0	49.0	7.9	42.4	6.7	62.2
	Sb	0.24	1.17	0.24	1.39	0.28	1.16	0.26	1.20	0.24	1.60
	Sn	2.5	15.0	2.4	14.0	2.4	17.3	2.9	17.2	2.8	12.1
Ratios	Nb/Ta		21.0		19.4		17.1		18.5		20.3
	Zr/Hf		33.8		34.2		43.4		27.2		21.3
Thermometry	Ln (Zr)		7.12		7.29		7.51		7.04		6.61
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	31	790	29	808	31	832	32	781	29	738
Distance from grain bou	undary (µm)		94		31		90		117		14
	GROD		0.185		0.35		0.475		1.99		0.305

Grain number**				13		14		15		16	
	Elements (ppm) (MDL filtered)	±2σ	4_l-1	±2σ	4_M-2	±2σ	4_N-5	±2σ	4_NII-2	±2σ	5_A-1
Analyses	Si	170	1050	300	377	190	514	180	1768	470	317
	Fe	820	6370	1100	6030	1000	4940	840	4480	770	5330
	U	0.16	5.51	1.1	2.11	0.40	9.40	1.8	8.83	1.7	3.46
	ν	90	1090	210	853	160	1020	190	943	180	667
	Zr	130	939	170	614	110	2030	370	1800	330	1770
	Hf	6.1	32.3	6.0	31.2	5.5	55.3	10	52.5	9.6	46.1
	Cr	7.3	38.8	7.8	64.3	12	67.7	13	77.6	15	42.9
	Nb	320	1560	290	1520	270	1440	260	1370	250	1440
	Та	16	77.5	14	73.5	13	60.8	11	56.0	10	69.2
	Zn	3.8	14.2	5.2	b.d.l.	5.1	6.24	4.0	10.8	4.5	5.05
	Мо	7.1	67.3	12	59.6	11	70.5	13	65.9	12	68.9
	W	11.2	49.8	9.5	39.4	7.3	41.2	7.7	44.7	8.5	44.7
	Sb	0.34	1.15	0.28	1.29	0.32	1.26	0.28	1.50	0.34	1.00
	Sn	2.3	15.0	3.0	14.1	2.7	14.2	2.8	16.8	3.3	14.9
Ratios	Nb/Ta		20.1		20.7		23.7		24.5		20.8
	Zr/Hf		29.1		19.7		36.7		34.3		38.4
Thermometry	Ln (Zr)		6.84		6.42		7.62		7.50		7.48
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	30	761	31	720	29	845	35	831	35	829
Distance from grain bou	undary (μm)		66		33		79		26		51
	GROD		0.615		0.405		1.02		1.706		0.295

Grain number**				No U-Pb			19		20		20
	Elements (ppm) (MDL filtered)	±2σ	6_A-1	±2σ	6_B-1	±2σ	6_G2-1	±2σ	6_H1-2	±2σ	6_H1-3
Analyses	Si	150	678	220	544	200	520	190	476	170	401
	Fe	950	4440	910	4600	940	7300	1300	5140	1000	4050
	U	0.70	3.56	0.84	4.57	1.1	13.5	2.8	12.4	2.8	5.86
	V	130	422	98	740	170	637	130	871	190	865
	Zr	340	2060	460	1090	240	1820	360	1460	310	894
	Hf	8.8	46.3	10	29.0	6.5	44.9	8.9	37.3	8.1	30.3
	Cr	8.9	26.0	6.3	46.7	11	63.0	13	78.1	18	69.9
	Nb	270	763	170	1240	280	928	180	751	160	833
	Та	13	33.6	7.6	91.4	21	40.1	8.1	27.9	6.1	29.7
	Zn	4.6	8.29	4.5	5.94	4.6	6.75	4.8	2.53	3.6	4.44
	Мо	13	48.6	11	59.6	13	73.6	15	73.7	16	52.3
	W	8.8	28.9	6.7	50.7	12	44.3	9.1	46.2	10	47.9
	Sb	0.26	1.06	0.30	1.20	0.32	1.08	0.28	1.09	0.28	1.23
	Sn	3.0	15.5	3.7	12.9	3.1	13.3	2.8	14.2	3.3	12.2
Ratios	Nb/Ta		22.7		13.6		23.1		26.9		28.0
	Zr/Hf		44.5		37.6		40.5		39.2		29.6
Thermometry	Ln (Zr)		7.63		6.99		7.51		7.29		6.80
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	36	846	40	776	36	832	37	807	38	756
Distance from grain bo	undary (µm)		26		39		58		50		47
	GROD		0.385		0.565		0.125		0.335		0.255

Grain number**			23 N		No U-Pb No U-Pb				25		
	Elements (ppm) (MDL filtered)	±2σ	6_J1-1	±2σ	6_J5-1	±2σ	7_B-1	±2σ	7_C-1	±2σ	7_G1-3
Analyses	Si	160	939	270	753	230	406	160	278	130	553
	Fe	810	4110	700	5390	980	4510	1000	3830	860	5000
	U	1.3	7.96	1.6	5.60	1.1	1.86	0.50	0.0643	0.020	8.18
	V	190	725	150	746	150	486	130	229	58	814
	Zr	190	2300	440	1620	320	1400	350	811	200	872
	Hf	6.6	46.7	9.0	42.0	8.2	38.6	9.8	23.2	5.7	24.2
	Cr	16	57.4	12	46.5	9.8	47.4	13	8.4	2.6	51.1
	Nb	180	1450	280	1420	280	975	250	893	220	903
	Та	6.5	68.1	13	61.6	12	45.9	12	69.3	17	40.9
	Zn	3.8	4.76	4.0	8.08	4.4	3.35	4.3	4.50	4.3	4.45
	Мо	11	49.5	9.6	75.4	15	65.2	16	14.4	3.6	81.9
	W	11	62.4	13	52.4	11	40.0	11	54.2	14	47.1
	Sb	0.32	1.90	0.44	1.69	0.40	1.27	0.38	1.74	0.50	1.11
	Sn	2.8	12.0	2.5	13.4	2.8	13.7	3.7	8.12	2.1	15.9
Ratios	Nb/Ta		21.3		23.0		21.2		12.9		22.1
	Zr/Hf		49.3		38.6		36.3		34.9		36.0
Thermometry	Ln (Zr)		7.74		7.39		7.24		6.70		6.77
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	35	859	37	819	36	803	42	747	37	754
Distance from grain bo	undary (µm)		29		34		46		12		147
	GROD		0.385		0.335		4.52		2.38		2.43
Grain number**			25		26		26		No U-Pb	27	
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	Elements (ppm) (MDL filtered)	±2σ	7 G1-8	±2σ	7 G2-1	±2σ	7 G2-2	±2σ	7 K-2	±2σ	7 L1-3
Analyses	Si	190	610	210	341	140	197	120	403	160	695
	Fe	1100	4680	1000	4750	1000	4690	1000	4010	860	8170
	U	2.0	7.94	2.0	13.1	3.3	9.52	2.4	0.483	0.12	10.6
	V	200	789	190	506	130	510	130	627	150	735
	Zr	210	1160	280	1940	470	1720	420	1350	320	1500
	Hf	5.8	31.9	7.6	46.1	11	44.3	11	33.3	7.8	39.0
	Cr	13	44.2	11	53.6	14	62.2	16	20.0	5.2	52.5
	Nb	210	921	220	992	240	1050	250	462	110	996
	Та	9.8	41.3	10	43.4	11	42.1	10	18.3	4.3	44.6
	Zn	4.2	b.d.l.	4.1	2.27	4.0	0.641	4.1	11.6	5.3	9.38
	Мо	19	76.1	18	86.4	21	80.9	19	35.2	8.2	86.7
	W	12	41.6	10	48.2	12	48.1	12	18.9	4.6	48.9
	Sb	0.32	1.19	0.34	0.895	0.26	0.91	0.26	1.23	0.34	1.22
	Sn	4.0	14.8	3.8	13.9	3.5	13.9	3.6	15.1	3.7	15.5
Ratios	Nb/Ta		22.3		22.9		24.9		25.3		22.3
	Zr/Hf		36.4		42.1		38.9		40.5		38.5
Thermometry	Ln (Zr)		7.06		7.57		7.45		7.21		7.31
	P (Kbars)		12		12		12		12		12
	Temperature (°C)	36	783	38	839	42	825	42	799	39	810
Distance from grain bou	ndary (µm)		71		51		89		15		88
	GROD		2.22		0.805		1.25		0.535		0.125

Grain number**			28				
	Elements (ppm) (MDL filtered)	±2σ	7 L2-1	±2σ			
Analyses	Si	230	674	220			
	Fe	1710	4330	900			
	U	2.5	8.69	2.1			
	V	170	536	130			
	Zr	340	1680	390			
	Hf	8.9	40.2	9.3			
	Cr	13	50.3	12			
	Nb	230	1030	240			
	Та	10	44.5	10			
	Zn	4.9	5.40	4.2			
	Мо	20	77.9	18			
	W	12	48.5	12			
	Sb	0.34	0.932	0.26			
	Sn	3.8	14.3	3.5			
Ratios	Nb/Ta		23.1				
	Zr/Hf		41.8				
Thermometry	Ln (Zr)		7.43				
	P (Kbars)		12				
	Temperature (°C)	39	823	40			
Distance from grain bo	undary (µm)		64				
	GROD		0.325				

Lithology	Amphibolite					
Туре						
Analyses	17FJ05_R1_1	17FJ05_R1_2	17FJ05_R2_1	17FJ05_R2_2	17FJ05_R2_3	17FJ05_R2_4
Wt% oxide						
SiO <sub>2</sub>	0.200	0.758	0.415	0.0636	0.652	0.137
TiO <sub>2</sub>	99.1	96.3	99.7	101	96.6	98.9
Al <sub>2</sub> O <sub>3</sub>	0.0227	0.0610	0.144	0.0138	0.153	0.0375
Cr <sub>2</sub> O <sub>3</sub>	b.d.l.	b.d.l.	0.0658	0.0556	0.0469	0.0393
FeO	0.485	0.606	0.376	0.276	0.337	0.322
MnO	0.0160	0.0152	b.d.l.	b.d.l.	b.d.l.	b.d.l.
NiO	b.d.l.	0.00736	b.d.l.	b.d.l.	b.d.l.	b.d.l.
CaO	0.709	1.05	0.698	0.463	1.12	0.714
Nb <sub>2</sub> O <sub>5</sub>	0.0275	0.0179	0.0184	0.0123	0.0133	0.0167
ZrO <sub>2</sub>	0.0527	0.0456	0.0276	0.0234	0.0305	0.0197
HfO <sub>2</sub>	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V <sub>2</sub> O <sub>3</sub>	0.0464	b.d.l.	0.0824	0.0914	0.0569	0.0931
Total	101	98.8	102	102	99.0	100
Elements (ppm)						
Si	936	3540	1940	297	3050	640
AI	120	323	762	73	808	199
Cr	b.d.l.	b.d.l.	451	381	321	269
Fe	3770	4710	2930	2150	2620	2500
Ni	b.d.l.	57.8	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V	260	b.d.l.	462	512	319	522
Nb	192	125	129	86.0	92.7	117
Zr	390	337	205	173	226	146
Thermometer (Tomk	ins et al 2007)					
In (Zr ppm)	5.97	5.82	5.32	5.15	5.42	4.98
P (Kbar)	13	13	13	13	13	13
Temperature (°C)	685	672	632	620	640	607
Error (2σ)*	19	20	24	25	23	27

Туре	_							
Analyses	17FJ05_R2	17FJ05_R2	17FJ05_R3	17FJ05_R3	17FJ05_R3	17FJ05_R3	17FJ05_R3	H2 R15
Wt% oxide								
SiO <sub>2</sub>	0.0807	0.878	0.0546	0.0428	0.0385	0.0429	0.361	0.581
TiO <sub>2</sub>	99.8	99.3	100	101	101	101	99.8	98.3
Al <sub>2</sub> O <sub>3</sub>	0.00519	0.306	0.0198	b.d.l.	0.00781	0.0103	0.110	0.254
Cr <sub>2</sub> O <sub>3</sub>	0.0539	0.0404	0.0505	0.0415	0.0708	0.0583	0.0533	b.d.l
FeO	0.413	0.667	0.383	0.359	0.327	0.323	0.387	0.421
MnO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	0.0106	0.00611	b.d.l.	0.00693
NiO	b.d.l.	0.00988						
CaO	0.548	0.744	0.314	0.270	0.268	0.286	0.557	1.00
Nb <sub>2</sub> O <sub>5</sub>	0.0182	0.0156	0.0196	0.0140	0.0191	0.0152	0.0130	0.0447
ZrO <sub>2</sub>	0.0254	0.0209	0.0557	0.0340	0.0291	0.0469	0.0564	0.0712
HfO <sub>2</sub>	b.d.l.	b.d.l.						
V <sub>2</sub> O <sub>3</sub>	0.0974	0.0610	0.0573	0.0757	0.0732	0.0600	0.0667	0.0187
Total	101	102	101	101	102	102	101	101
Elements (ppm)								
Si	377	4110	255	200	180	201	1690	2720
AI	27.5	1620	105	b.d.l.	41.3	54.5	582	1340
Cr	369	276	346	284	485	399	364	b.d.l.
Fe	3210	5180	2970	2790	2550	2510	3010	3270
Ni	b.d.l.	77.7						
V	545	342	321	424	410	336	374	105
Nb	128	109	137	97.9	134	106	90.8	313
Zr	188	155	412	252	216	347	418	527
Thermometer (Tomk	i							
ln (Zr ppm)	5.24	5.04	6.02	5.53	5.37	5.85	6.03	6.27
P (Kbar)	13	13	13	13	13	13	13	13
Temperature (°C)	626	612	689	648	636	675	690	715
Error (2σ)*	25	26	19	22	24	20	19	18

Lithology

Lithology		Strained ar	nphibolite									
Туре												
Analyses	H2 R16	FJ7-2 R1	FJ7-2 R2	FJ7-2 R2	FJ7-2 R7	FJ7-2 R9	FJ7-2 R6	FJ7-2 R10				
Wt% oxide												
SiO <sub>2</sub>	0.0997	0.0416	0.0411	0.0859	0.274	0.104	0.254	0.0853				
TiO <sub>2</sub>	100	100	101	101	99.4	100	101	101				
Al <sub>2</sub> O <sub>3</sub>	0.00759	b.d.l.	b.d.l.	0.0198	0.0912	0.0361	0.0745	0.0247				
Cr <sub>2</sub> O <sub>3</sub>	b.d.l	0.0370	b.d.l	0.0254	0.0341	0.0621	0.0248	0.0378				
FeO	0.385	0.412	0.396	0.504	0.637	0.480	0.650	0.564				
MnO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	0.00988	0.0114	b.d.l.				
NiO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.				
CaO	1.00	0.0244	0.0276	0.0398	0.513	0.244	0.525	0.334				
Nb <sub>2</sub> O <sub>5</sub>	0.0476	0.0890	0.0681	0.218	0.0899	0.142	0.0878	0.1635				
ZrO <sub>2</sub>	0.0772	0.0196	0.0261	0.0100	0.0175	0.0241	0.0334	0.0112				
HfO <sub>2</sub>	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.				
V <sub>2</sub> O <sub>3</sub>	b.d.l.	0.0234	0.0329	0.0165	0.0267	0.0341	b.d.l.	b.d.l.				
Total	102	101	101	102	101	101	103	103				
Elements (ppm)												
Si	466	195	192	401	1280	486	1190	399				
AI	40.2	b.d.l.	b.d.l.	105	482	191	394	131				
Cr	b.d.l.	253	b.d.l.	174	233	425	170	259				
Fe	2990	3200	3080	3920	4950	3730	5060	4390				
Ni	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.				
V	b.d.l.	131	184	92.4	149	191	b.d.l.	b.d.l.				
Nb	333	622	476	1530	628	994	614	1140				
Zr	572	145	193	73.9	129	178	247	83				
Thermometer (Tomk	i											
ln (Zr ppm)	6.35	4.97	5.26	4.30	4.86	5.18	5.51	4.42				
P (Kbar)	13	12	12	12	12	12	12	12				
Temperature (°C)	727	603	624	557	595	618	655	580				
Error (2σ)*	17	26	24	35	27	25	22	34				

Lithology	Fracture type-I hal	0			
Туре					
Analyses	17FJ02A_R2_1	17FJ02A_R2_2	17FJ02A_R2_3	17FJ02A_R3_1	17FJ02A_R3_2
Wt% oxide					
SiO <sub>2</sub>	0.0446	0.0352	0.0444	0.0598	0.0522
TiO <sub>2</sub>	98.4	99.7	100	99.7	101
Al <sub>2</sub> O <sub>3</sub>	b.d.l	b.d.l	b.d.l	b.d.l	b.d.l
Cr <sub>2</sub> O <sub>3</sub>	0.140	0.139	0.165	0.0861	0.0853
FeO	0.322	0.299	0.322	0.383	0.377
MnO	0.0107	b.d.l.	b.d.l.	b.d.l.	b.d.l.
NiO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
CaO	0.304	0.250	0.311	0.261	0.222
Nb <sub>2</sub> O <sub>5</sub>	0.0351	0.0252	0.0211	0.0657	0.0573
ZrO <sub>2</sub>	0.0198	0.0291	0.0336	0.0276	0.0265
HfO <sub>2</sub>	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V <sub>2</sub> O <sub>3</sub>	0.0435	0.0503	0.0559	0.0876	0.0703
Total	99.4	100	101	101	102
Elements (ppm)					
Si	208	165	207	279	244
AI	b.d.l	b.d.l	b.d.l	b.d.l.	b.d.l.
Cr	959	952	1130	589	584
Fe	2500	2320	2500	2980	2930
Ni	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V	244	282	313	491	394
Nb	245	176	148	459	401
Zr	147	215	249	204	196
Thermometer (Tomk	i				
ln (Zr ppm)	4.99	5.37	5.52	5.32	5.28
P (Kbar)	11	11	11	11	11
Temperature (°C)	599	628	639	624	621
Error (2σ)*	27	23	22	24	24

туре					
Analyses	17FJ02A_R3_3	17FJ02A_R3_4	17FJ02A_R3_7	17FJ02A_R3_8	17FJ02A_R4_2
Wt% oxide					
SiO <sub>2</sub>	0.0532	0.0514	0.109	0.0972	0.0423
TiO <sub>2</sub>	100	99.0	99.8	98.4	101
Al <sub>2</sub> O <sub>3</sub>	b.d.l	b.d.l	0.0106	b.d.l	b.d.l
Cr <sub>2</sub> O <sub>3</sub>	0.108	0.105	0.0813	0.0992	0.101
FeO	0.369	0.373	0.424	0.495	0.372
MnO	b.d.l.	0.00920	b.d.l.	b.d.l.	b.d.l.
NiO	b.d.l.	0.00615	b.d.l.	b.d.l.	b.d.l.
CaO	0.212	0.210	0.557	0.495	0.206
Nb <sub>2</sub> O <sub>5</sub>	0.0512	0.0635	0.0482	0.0729	0.0283
ZrO <sub>2</sub>	0.0169	0.0242	0.0328	0.0622	0.0337
HfO <sub>2</sub>	b.d.l.	b.d.l.	0.0233	b.d.l.	0.0102
V <sub>2</sub> O <sub>3</sub>	0.0814	0.0797	0.0586	0.0720	0.0843
Total	101	99.9	101	99.8	101
Elements (ppm)					
Si	249	240	511	454	198
AI	b.d.l.	b.d.l.	55.96	b.d.l.	b.d.l.
Cr	737	719	556	679	690
Fe	2870	2900	3290	3850	2890
Ni	b.d.l.	48.3	b.d.l.	b.d.l.	b.d.l.
V	456	446	328	404	472
Nb	358	444	337	509	198
Zr	125	179	242	460	249
Thermometer (Tomk	i				
ln (Zr ppm)	4.83	5.19	5.49	6.13	5.52
P (Kbar)	11	11	11	11	11
Temperature (°C)	588	614	637	690	639
Error (2σ)*	28	25	23	18	22

Туре	_				
Analyses	17FJ02A_R4_3	17FJ02A_R4_4	17FJ02A_R4_5	17FJ02A_R4_6	17FJ02A_R4_7
Wt% oxide					
SiO <sub>2</sub>	0.0406	0.0597	0.0920	0.0794	0.0867
TiO <sub>2</sub>	100	101	99.5	101	101
Al <sub>2</sub> O <sub>3</sub>	b.d.l	b.d.l	b.d.l	b.d.l	b.d.l
Cr <sub>2</sub> O <sub>3</sub>	0.105	0.102	0.0983	0.0888	0.0933
FeO	0.364	0.439	0.473	0.480	0.511
MnO	b.d.l.	b.d.l.	0.00622	0.0125	b.d.l.
NiO	b.d.l.	b.d.l.	0.00603	b.d.l.	b.d.l.
CaO	0.252	0.320	0.441	0.413	0.467
Nb <sub>2</sub> O <sub>5</sub>	0.0326	0.0345	0.0397	0.0353	0.0513
ZrO <sub>2</sub>	0.0486	0.0325	0.0373	0.0290	0.0505
HfO <sub>2</sub>	b.d.l.	0.0168	b.d.l.	b.d.l.	b.d.l.
V <sub>2</sub> O <sub>3</sub>	0.0837	0.0648	0.0433	0.0669	0.0733
Total	101	102	101	103	102
Elements (ppm)					
Si	190	279	430	371	405
AI	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
Cr	717	695	673	607	638
Fe	2830	3410	3680	3730	3970
Ni	b.d.l.	b.d.l.	47.4	b.d.l.	b.d.l.
V	469	363	242	375	411
Nb	228	241	278	246	359
Zr	360	241	276	215	374
Thermometer (Tomk	i				
In (Zr ppm)	5.89	5.48	5.62	5.37	5.92
P (Kbar)	11	11	11	11	11
Temperature (°C)	669	637	647	628	672
Error (2σ)*	19	23	22	23	19

Lithology

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17FJ02A_R4_8	17FJ02A_R4_9	17FJ02A_R5_2	17FJ02A_R5_3	17FJ02A_R6_3
0.0752	0.0582	0.106	0.186	0.0662
100	99.5	101	98.9	101
0.00592	b.d.l	0.00713	0.0169	b.d.l
0.0832	0.0754	0.231	0.252	0.307
0.475	0.525	0.390	0.430	0.325
b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
0.392	0.385	0.475	0.567	0.380
0.0502	0.0380	0.0539	0.0615	0.0282
0.0407	0.0293	0.0421	0.0624	0.0331
b.d.l.	0.0198	b.d.l.	b.d.l.	b.d.l.
0.0684	0.0337	0.0456	0.0653	0.0654
102	101	102	101	102
352	272	497	871	310
31.34	b.d.l	37.74	89.64	b.d.l.
569	516	1578	1724	2100
3690	4080	3030	3340	2530
b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
383	189	255	366	366
351	266	377	430	197
301	217	312	462	245
i				
5.71	5.38	5.74	6.14	5.50
11	11	11	11	11
654	629	657	690	638
20	23	20	18	23
	17FJ02A_R4_8 0.0752 100 0.00592 0.0832 0.475 b.d.l. b.d.l. 0.392 0.0502 0.0407 b.d.l. 0.0684 102 352 31.34 569 3690 b.d.l. 383 351 301 5.71 11 654 20	17FJ02A_R4_8 17FJ02A_R4_9   0.0752 0.0582   100 99.5   0.00592 b.d.l   0.0832 0.0754   0.475 0.525   b.d.l. b.d.l.   0.392 0.385   0.0502 0.0380   0.0407 0.0293   b.d.l. 0.0198   0.0684 0.0337   102 101   352 272   31.34 b.d.l   569 516   3690 4080   b.d.l. b.d.l   351 266   301 217   5.71 5.38   11 11   654 629   20 23	17FJ02A_R4_8   17FJ02A_R4_9   17FJ02A_R5_2     0.0752   0.0582   0.106     100   99.5   101     0.00592   b.d.I   0.00713     0.0832   0.0754   0.231     0.475   0.525   0.390     b.d.I   b.d.I.   b.d.I.     b.d.I.   b.d.I.   b.d.I.     b.d.I.   b.d.I.   b.d.I.     b.d.I.   b.d.I.   b.d.I.     0.392   0.385   0.475     0.0502   0.0380   0.0539     0.0407   0.0293   0.0421     b.d.I.   0.0198   b.d.I.     0.0684   0.0337   0.0456     102   101   102     352   272   497     31.34   b.d.I.   3030     b.d.I.   b.d.I.   b.d.I.     3690   4080   3030     b.d.I.   b.d.I.   b.d.I.     383   189   255     351   266   377	17FJ02A_R4_8 17FJ02A_R4_9 17FJ02A_R5_2 17FJ02A_R5_3   0.0752 0.0582 0.106 0.186   100 99.5 101 98.9   0.00592 b.d.l 0.00713 0.0169   0.0832 0.0754 0.231 0.252   0.475 0.525 0.390 0.430   b.d.l. b.d.l. b.d.l. b.d.l.   0.392 0.385 0.475 0.567   0.0502 0.0380 0.0539 0.0615   0.0407 0.0293 0.0421 0.0624   b.d.l. 0.0198 b.d.l. b.d.l.   0.0684 0.0337 0.0456 0.0653   102 101 102 101   352 272 497 871   31.34 b.d.l. 37.74 89.64   569 516 1578 1724   3690 4080 3030 3340   b.d.l. b.d.l. b.d.l. b.d.l.   383 189 255 366   351 266 <

Lithology

Lithology			Fracture type-II hal	0	
Туре					
Analyses	17FJ02A_R6_4	17FJ02A_R6_5	17FJ06B_R1_1	17FJ06B_R1_2 1	7FJ06B_R1_3
Wt% oxide					
SiO <sub>2</sub>	0.0831	0.105	0.0827	0.0330	0.0501
TiO <sub>2</sub>	99.4	99.5	101	99.5	99.4
Al <sub>2</sub> O <sub>3</sub>	b.d.l	b.d.l	b.d.l	b.d.l.	b.d.l
Cr <sub>2</sub> O <sub>3</sub>	0.271	0.279	0.0288	b.d.l.	0.0361
FeO	0.338	0.383	0.465	0.437	0.453
MnO	b.d.l.	b.d.l.	b.d.l.	0.0114	b.d.l.
NiO	b.d.l.	b.d.l.	0.0102	b.d.l.	b.d.l.
CaO	0.408	0.524	0.594	0.543	0.582
Nb <sub>2</sub> O <sub>5</sub>	0.0321	0.0279	0.0808	0.0772	0.0731
ZrO <sub>2</sub>	0.0178	0.0431	0.0322	0.0394	0.0485
HfO <sub>2</sub>	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V <sub>2</sub> O <sub>3</sub>	0.0766	0.0444	0.0305	0.0268	0.0268
Total	101	101	102	101	101
Elements (ppm)					
Si	388	489	387	154	234
AI	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l
Cr	1853	1910	197	b.d.l.	247
Fe	2630	2980	3610	3400	3520
Ni	b.d.l.	b.d.l.	80.2	b.d.l.	b.d.l.
V	429	249	171	150	150
Nb	225	195	565	539	511
Zr	132	319	238	291	359
Thermometer (Tomk	ci				
In (Zr ppm)	4.88	5.77	5.47	5.67	5.88
P (Kbar)	11	11	12	12	12
Temperature (°C)	592	659	640	656	673
Error (2σ)*	27	20	23	22	19

Lithology					Leucocratic domain
Туре					Small
Analyses	17FJ06B_R1_4	17FJ06B_R1_5	17FJ06B_R2_2	17FJ06B_R2_3	FJ6a-1 R2
Wt% oxide					
SiO <sub>2</sub>	0.0951	0.204	0.722	0.188	0.0513
TiO <sub>2</sub>	98.8	99.5	99.9	100	101
Al <sub>2</sub> O <sub>3</sub>	0.0140	0.155	b.d.l	0.0316	0.00889
Cr <sub>2</sub> O <sub>3</sub>	0.0320	0.0127	0.0177	0.101	0.0304
FeO	0.496	0.500	0.194	0.193	0.342
MnO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
NiO	b.d.l.	b.d.l.	0.0586	0.0125	b.d.l.
CaO	0.887	0.840	0.410	0.515	0.129
Nb <sub>2</sub> O <sub>5</sub>	0.0706	0.0723	0.0102	0.0197	0.166
ZrO <sub>2</sub>	0.0379	0.0423	0.0635	0.0530	0.0223
HfO <sub>2</sub>	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V <sub>2</sub> O <sub>3</sub>	0.0513	0.0396	0.0834	0.114	0.127
Total	101	101	102	102	102
Elements (ppm)					
Si	444	953	3370	877	240
AI	74.18	822.18	b.d.l	167.10	47.07
Cr	219	87.0	121	692	208
Fe	3860	3890	1510	1500	2660
Ni	b.d.l.	b.d.l.	461	98.6	b.d.l.
V	287	222	467	637	710
Nb	493	506	71.6	138	1160
Zr	281	313	470	392	165
Thermometer (Tomk	i I				
In (Zr ppm)	5.64	5.75	6.15	5.97	5.11
P (Kbar)	12	12	12	12	12
Temperature (°C)	653	662	696	681	612
Error (2σ)*	22	20	18	19	25

Lithology								
Туре	Large	Large	Large	Large	Large	LAB	LAB	LAB
Analyses	FJ6a-1 R5	FJ6a-1 R7	FJ6a-1 R9	FJ6a-1 R10	FJ6a-1 R11	FJ6a-1 R14	FJ6a-1 R15	FJ6a-1 R17
Wt% oxide								
SiO <sub>2</sub>	b.d.l.	0.00691	0.0134	0.0147	0.0116	b.d.l.	0.00778	b.d.l
TiO <sub>2</sub>	101	101	101	101	101	101	100	101
Al <sub>2</sub> O <sub>3</sub>	b.d.l	b.d.l	b.d.l	0.0292	b.d.l	b.d.l	b.d.l	b.d.l
Cr <sub>2</sub> O <sub>3</sub>	0.0282	0.0456	0.0501	0.0570	0.0334	0.0349	0.0269	0.0293
FeO	0.550	0.441	0.446	0.372	0.288	0.381	0.384	0.363
MnO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	0.00894
NiO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	0.00288	0.00593	b.d.l.
CaO	0.00440	0.00467	0.0202	0.0145	0.0246	0.00362	0.0180	0.00312
Nb <sub>2</sub> O <sub>5</sub>	0.205	0.191	0.193	0.190	0.176	0.0685	0.0728	0.0612
ZrO <sub>2</sub>	0.1230	0.1173	0.0445	0.0440	0.122	0.110	0.122	0.0698
HfO <sub>2</sub>	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V <sub>2</sub> O <sub>3</sub>	0.109	0.119	0.140	0.134	0.195	0.257	0.278	0.269
Total	102	102	101	102	102	102	101	102
Elements (ppm)								
Si	b.d.l.	32.3	62.7	68.7	54.3	b.d.l.	36.3	b.d.l
AI	b.d.l	b.d.l	b.d.l	154.59	b.d.l	b.d.l	b.d.l	b.d.l
Cr	193	312	343	390	229	239	184	201
Fe	4270	3430	3470	2890	2240	2960	2990	2820
Ni	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	22.6	46.6	b.d.l.
V	613	667	783	751	1090	1440	1560	1510
Nb	1440	1340	1350	1330	1230	479	509	428
Zr	911	869	329	326	906	813	903	516
Thermometer (Tomk	i -							
In (Zr ppm)	6.81	6.77	5.80	5.79	6.81	6.70	6.81	6.25
P (Kbar)	12	12	12	12	12	12	12	12
Temperature (°C)	758	753	666	665	758	747	757	705
Error (2σ)*	16	16	20	20	16	16	16	18

Table S3 - EMPA major and trace element data for all lithologies

Lithology							
Туре	LAB	LAB	Large	Large	Small	Small	Large
Analyses	FJ6a-1 R19	FJ6a-1 R20	FJ6a-1 R21	FJ6a-1 R22	FJ6a-1 R1	FJ6a-1 R3	FJ6a-1 R4
Wt% oxide							
SiO <sub>2</sub>	0.0132	0.00717	0.0148	0.0238	0.0246	0.0387	0.0250
TiO <sub>2</sub>	101	100	100	101	102	102	101
Al <sub>2</sub> O <sub>3</sub>	b.d.l	b.d.l	b.d.l	b.d.l	b.d.l	b.d.l	0.00635
Cr <sub>2</sub> O <sub>3</sub>	0.0420	0.0064	b.d.l	0.0191	0.0585	0.0205	0.0591
FeO	0.440	0.407	0.316	0.315	0.354	0.362	0.493
MnO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	0.00767	0.00859	0.00822
NiO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
CaO	0.0088	0.0143	0.0587	0.0524	0.0189	0.0823	0.0141
Nb <sub>2</sub> O <sub>5</sub>	0.0720	0.0718	0.0383	0.0224	0.121	0.0970	0.186
ZrO <sub>2</sub>	0.123	0.104	0.0439	0.0310	0.0353	0.0131	0.0855
HfO <sub>2</sub>	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V <sub>2</sub> O <sub>3</sub>	0.250	0.250	0.162	0.163	0.150	0.132	0.117
Total	102	101	101	101	103	103	102
Elements (ppm)							
Si	61.6	33.5	69.2	111	115	181	117
AI	b.d.l	b.d.l	b.d.l	b.d.l	b.d.l	b.d.l	33.6
Cr	287	43.7	b.d.l	131	400	140	404
Fe	3420	3170	2460	2450	2750	2810	3830
Ni	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V	1400	1400	908	915	838	738	657
Nb	503	502	268	157	845	678	1300
Zr	909	767	325	230	262	97.1	633
Thermometer (Tomk	i						
In (Zr ppm)	6.81	6.64	5.78	5.44	5.57	4.58	6.45
P (Kbar)	12	12	12	12	12	12	12
Temperature (°C)	758	741	665	637	669	598	755
Error (2σ)*	16	17	20	23	22	32	17

Table S3 - EMPA major and trace element data for all lithologies

Lithology					
Туре	Large	LAB	LAB	LAB	LAB
Analyses	FJ6a-1 R6	FJ6a-1 R12	FJ6a-1 R13	FJ6a-1 R16	5 FJ6a-1 R18
Wt% oxide					
SiO <sub>2</sub>	0.0120	0.0146	0.00742	b.d.l.	0.00590
TiO <sub>2</sub>	101	101	102	101	101
Al <sub>2</sub> O <sub>3</sub>	b.d.l	0.00627	b.d.l	b.d.l	0.0175
Cr <sub>2</sub> O <sub>3</sub>	0.0346	0.0530	0.0332	0.0177	0.0289
FeO	0.478	0.311	0.257	1.13	0.381
MnO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
NiO	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
CaO	0.0160	0.0143	0.00788	0.00346	0.00253
Nb <sub>2</sub> O <sub>5</sub>	0.192	0.0534	0.0584	0.0282	0.0651
ZrO <sub>2</sub>	0.0650	0.0472	0.0788	0.0390	0.0928
HfO <sub>2</sub>	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V <sub>2</sub> O <sub>3</sub>	0.151	0.281	0.273	0.293	0.296
Total	102	102	102	102	102
Elements (ppm)					
Si	56.2	68.3	34.7	b.d.l.	27.6
AI	b.d.l	33.2	b.d.l	b.d.l.	92.7
Cr	236	362	227	121	198
Fe	3720	2410	2000	8790	2960
Ni	b.d.l.	b.d.l.	b.d.l.	b.d.l.	b.d.l.
V	845	1570	1530	1640	1660
Nb	1340	373	408	197	455
Zr	481	350	584	288	687
Thermometer (Tomk	i -				
In (Zr ppm)	6.18	5.86	6.37	5.66	6.53
P (Kbar)	12	12	12	12	12
Temperature (°C)	734	710	761	702	786
Error (2σ)*	18	19	17	21	17

 $\begin{array}{ccc} Grain & Analyses & Distance from grain ~U~(ppm) & Pb_c & Ratios \\ & boundary~(\mu m) & \end{array}$ 

			f206%	<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>206</sup> Pb	±2σ	rho	<sup>206</sup> Pb/ <sup>238</sup> U
1 1_1-1	77	8.99	4.9	0.477	0.040	0.0644	0.0020	0.0537	0.0041	0.37	402
1_1-2	69	4.96	8.0	0.470	0.056	0.0643	0.0019	0.0530	0.0061	0.25	402
2 1_2-1	31	5.06	7.5	0.488	0.062	0.0645	0.0018	0.0549	0.0068	0.22	403
1_2-2	167	8.70	4.1	0.480	0.047	0.0637	0.0024	0.0547	0.0049	0.39	398
1_2-3	173	10.5	3.6	0.483	0.036	0.0641	0.0021	0.0546	0.0037	0.43	401
1_2-4	10	8.6	4.8	0.479	0.038	0.0645	0.0019	0.0538	0.0040	0.37	403
1_2-5	10	8.01	5.8	0.472	0.051	0.0626	0.0026	0.0548	0.0055	0.38	391
3 1_3-1	55	3.39	6.8	0.570	0.079	0.0734	0.0030	0.0563	0.0075	0.30	456
1_3-2	95	7.80	3.7	0.508	0.053	0.0667	0.0041	0.0553	0.0047	0.59	416
4 1_4-1	97	23.5	1.7	0.458	0.038	0.0584	0.0037	0.0569	0.0030	0.77	366
1_4-2	114	3.72	8.9	0.628	0.076	0.0715	0.0024	0.0637	0.0074	0.28	445
5 1_5-1	80	6.25	5.4	0.476	0.071	0.0634	0.0027	0.0545	0.0077	0.29	396
1_5-2	126	8.12	3.9	0.484	0.043	0.0647	0.0023	0.0542	0.0044	0.40	404
1_5-3	105	7.94	3.9	0.489	0.041	0.0645	0.0029	0.0550	0.0039	0.53	403
1_5-4	10	5.22	5.9	0.494	0.057	0.0650	0.0038	0.0550	0.0054	0.50	406
6 1_5-5	64	8.68	6.0	0.451	0.045	0.0613	0.0023	0.0534	0.0050	0.37	383
1_5-6	154	9.32	3.8	0.487	0.037	0.0650	0.0023	0.0543	0.0037	0.46	406
1_5-8	92	7.03	4.4	0.488	0.044	0.0641	0.0037	0.0552	0.0038	0.65	401
7 2_D-5	36	13.8	2.7	0.479	0.040	0.0645	0.0041	0.0538	0.0029	0.77	403
8 2_G-1	90	7.51	4.9	0.480	0.032	0.0639	0.0018	0.0545	0.0034	0.41	399
9 3_C-2	80	5.12	5.9	0.542	0.068	0.0641	0.0047	0.0613	0.0062	0.58	401
10 3_G2-2	84	7.74	4.0	0.499	0.056	0.0660	0.0033	0.0549	0.0055	0.44	412
3_G2-3	40	8.47	3.3	0.498	0.050	0.0660	0.0029	0.0547	0.0050	0.43	412
11 3_K-1	86	7.78	3.9	0.525	0.040	0.0686	0.0021	0.0556	0.0039	0.39	428
3_K-2	142	6.95	4.3	0.519	0.061	0.0698	0.0024	0.0539	0.0060	0.30	435
3_K-3	89	5.42	6.0	0.502	0.068	0.0654	0.0026	0.0557	0.0073	0.29	408
12 4_BI-3	22	4.35	8.8	0.508	0.059	0.0667	0.0039	0.0552	0.0056	0.50	417
13 4_I-1	33	5.26	6.9	0.562	0.066	0.0726	0.0038	0.0562	0.0059	0.44	451

			f206%	<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>206</sup> Pb	±2σ	rho	<sup>206</sup> Pb/ <sup>238</sup> U
14 4_M-2	69	4.33	4.6	0.458	0.054	0.0643	0.0044	0.0517	0.0049	0.59	402
15 4_N-5	33	8.74	3.9	0.485	0.046	0.0674	0.0020	0.0523	0.0048	0.31	420
4_N-6	74	7.22	4.7	0.510	0.051	0.0699	0.0019	0.0529	0.0051	0.27	436
16 4_NII-2	36	8.00	5.0	0.541	0.048	0.0712	0.0034	0.0552	0.0041	0.54	443
17 5_D-1	47	11.3	3.0	0.497	0.044	0.0646	0.0034	0.0558	0.0040	0.59	404
5_D-2	49	3.42	9.9	0.496	0.072	0.0671	0.0029	0.0536	0.0074	0.30	419
5_D-3	160	17.4	2.3	0.492	0.042	0.0656	0.0026	0.0544	0.0041	0.47	410
5_D-4	20	6.50	4.7	0.573	0.046	0.0738	0.0029	0.0563	0.0039	0.49	459
5_D-5	46	13.0	2.9	0.484	0.030	0.0682	0.0025	0.0516	0.0026	0.60	425
18 6_B-2	43	4.35	7.7	0.603	0.079	0.0773	0.0036	0.0566	0.0069	0.36	480
19 6_G2-2	27	10.9	3.1	0.544	0.029	0.0718	0.0017	0.0550	0.0026	0.45	447
6_G2-3	36	6.13	5.2	0.544	0.048	0.0704	0.0022	0.0560	0.0046	0.36	439
6_G2-4	28	4.43	7.1	0.577	0.088	0.0742	0.0022	0.0564	0.0084	0.19	461
20 6_H1-1	24	12.0	2.7	0.526	0.040	0.0687	0.0015	0.0555	0.0041	0.29	428
6_H1-2	73	12.6	2.8	0.521	0.029	0.0672	0.0019	0.0563	0.0027	0.50	419
6_H1-3	70	14.4	2.5	0.530	0.032	0.0686	0.0026	0.0560	0.0026	0.63	428
6_H1-4	67	14.0	2.6	0.555	0.038	0.0684	0.0023	0.0589	0.0036	0.48	426
21 6_H1-6	46	9.90	3.4	0.526	0.043	0.0691	0.0029	0.0552	0.0039	0.51	431
6_H1-7	9	12.1	3.0	0.523	0.041	0.0686	0.0020	0.0553	0.0041	0.37	427
22 6_H3-1	33	9.93	3.5	0.493	0.030	0.0683	0.0021	0.0523	0.0028	0.50	426
6_H3-3	46	13.7	2.7	0.503	0.044	0.0699	0.0049	0.0522	0.0027	0.81	435
23 6_J1-2	37	11.9	3.4	0.491	0.057	0.0642	0.0053	0.0555	0.0045	0.71	401
6_J1-3	73	5.37	7.1	0.497	0.068	0.0647	0.0028	0.0558	0.0072	0.32	404
24 7_E-1	34	4.35	6.2	0.526	0.058	0.0670	0.0025	0.0570	0.0059	0.34	418
25 7_G1-3	252	9.79	4.8	0.445	0.031	0.0622	0.0013	0.0520	0.0034	0.31	389
7_G1-4	144	8.31	3.1	0.530	0.037	0.0697	0.0025	0.0552	0.0033	0.51	434
7_G1-5	60	12.5	2.2	0.483	0.027	0.0639	0.0025	0.0548	0.0023	0.68	400
7_G1-6	170	9.28	2.8	0.500	0.032	0.0648	0.0021	0.0560	0.0030	0.52	405
7_G1-7	71	9.09	3.1	0.447	0.034	0.0598	0.0019	0.0541	0.0038	0.42	375
26 7_G2-1	35	7.36	3.8	0.521	0.044	0.0653	0.0020	0.0578	0.0045	0.37	408
7_G2-2	56	5.80	4.9	0.484	0.038	0.0642	0.0018	0.0547	0.0040	0.37	401

Table S4a - LA-ICP-MS U-Pb data for large rutile grains in leucocratic domains

			f206%	<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>206</sup> Pb	±2σ	rho	<sup>206</sup> Pb/ <sup>238</sup> U
7_G2-3	62	12.2	2.3	0.511	0.026	0.0679	0.0017	0.0546	0.0024	0.50	424
7_G2-4	33	3.90	7.8	0.538	0.060	0.0711	0.0020	0.0549	0.0059	0.25	443
27 7_L1-1	56	7.07	3.4	0.484	0.045	0.0643	0.0033	0.0546	0.0042	0.55	402
7_L1-2	21	12.4	2.5	0.472	0.037	0.0665	0.0026	0.0515	0.0035	0.51	415
7_L1-3	143	13.9	2.1	0.499	0.040	0.0660	0.0030	0.0548	0.0037	0.56	412
7_L1-4	26	12.1	2.4	0.518	0.031	0.0679	0.0020	0.0553	0.0028	0.50	423
7_L1-6	6	9.21	2.6	0.489	0.038	0.0650	0.0036	0.0546	0.0031	0.70	406
28 7_L2-1	30	8.04	3.4	0.617	0.050	0.0788	0.0026	0.0568	0.0042	0.41	489
7_L2-2	60	12.1	2.8	0.542	0.045	0.0708	0.0044	0.0555	0.0031	0.74	441

Grain	Analyses				Discordance (%)	Peak age on radial plot (Fi 10e)	Mean grain g. age		Mean grain age		Mean ratios
		±2σ	<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>207</sup> Pb/ <sup>235</sup> U) / ( <sup>206</sup> Pb/ <sup>238</sup> U)		<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>235</sup> U
	1 1_1-1	12	396	27	1.6	1	394	22	402	8.3	0.475
	1_1-2	11	391	39	2.7	1					
	2 1_2-1	11	403	43	-0.15	1	398	14	400	5.7	0.480
	1_2-2	15	398	32	-0.024	1					
	1_2-3	12	400	25	0.19	1					
	1_2-4	11	397	26	1.5	1					
	1_2-5	16	393	35	-0.42	1					
	3 1_3-1	18	458	51	-0.33	2	431	29	442	15	0.527
	1_3-2	25	417	36	-0.27	1					
	4 1_4-1	23	383	26	-4.6	exc.	409	23	422	12	0.490
	1_4-2	15	495	48	-11	1					
	5 1_5-1	17	396	49	0.10	1	403	17	402	8.5	0.487
	1_5-2	14	400	29	0.9	1					
	1_5-3	18	404	28	-0.34	1					
	1_5-4	23	407	38	-0.27	1					
	6 1_5-5	14	378	32	1.4	1	393	20	396	8.9	0.477
	1_5-6	14	403	25	0.77	1					
	1_5-8	23	404	30	-0.75	1					
	7 2_D-5	25	397	27	1.5	1	397	27	403	25	0.479
	8 2_G-1	11	398	22	0.29	1	398	22	399	11	0.480
	9 3_C-2	28	440	45	-9.8	exc.	440	45	401	28	0.542
	10 3_G2-2	20	411	38	0.13	1	411	25	412	13	0.499
	3_G2-3	17	410	34	0.46	1					
	11 3_K-1	12	429	27	-0.28	2	427	22	425	8.1	0.519
	3_K-2	15	425	40	2.4	2					
	3_K-3	16	413	46	-1.2	1					
	12 4_BI-3	24	417	40	-0.21	1	417	40	417	24	0.508
	13 4_l-1	23	453	43	-0.34	2	453	43	451	23	0.562

#### Table S4a - LA-ICP-MS U-Pb data for large rutile grains in leucocratic domains

				<sup>207</sup> Pb/ <sup>235</sup> U) /						
	±2σ	<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	( <sup>206</sup> Pb/ <sup>238</sup> U)		<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>235</sup> U
14 4_M-2	27	383	37	4.7	1	383	37	402	27	0.458
15 4_N-5	12	402	32	4.4	2	409	23	428	8.2	0.496
4_N-6	11	418	34	4.0	2					
16 4_NII-2	21	439	32	0.88	2	439	32	443	21	0.541
17 5_D-1	20	410	30	-1.5	1	415	13	411	10	0.504
5_D-2	17	409	49	2.3	1					
5_D-3	16	407	29	0.77	1					
5_D-4	17	460	30	-0.26	2					
5_D-5	15	401	21	5.6	exc.					
18 6_B-2	22	479	50	0.16	2	479	50	480	22	0.603
19 6_G2-2	10	441	19	1.3	2	443	16	449	7.0	0.547
6_G2-3	13	441	32	-0.51	2					
6_G2-4	13	463	57	-0.27	2					
20 6_H1-1	9.1	429	27	-0.15	2	433	11	426	5.8	0.531
6_H1-2	11	426	19	-1.6	2					
6_H1-3	15	432	21	-0.92	2					
6_H1-4	14	448	25	-5.1	exc.					
21 6_H1-6	17	429	29	0.30	2	428	20	429	10	0.524
6_H1-7	12	427	28	0.13	2					
22 6_H3-1	13	407	21	4.5	2	409	17	424	11	0.496
6_H3-3	30	414	30	5.0	exc.					
23 6_J1-2	32	406	39	-1.2	1	407	30	403	15	0.494
6_J1-3	17	410	46	-1.5	1					
24 7_E-1	15	429	39	-2.7	1	429	39	418	15	0.526
25 7_G1-3	8.1	374	22	3.8	1	398	10	390	5.5	0.479
7_G1-4	15	432	24	0.50	2					
7_G1-5	15	400	19	-0.15	1					
7_G1-6	13	412	21	-1.8	1					
7_G1-7	12	375	24	-0.086	1					
26 7_G2-1	12	426	29	-4.3	1	418	12	419	7.8	0.509
7_G2-2	11	401	26	0.042	1					

Table S4a - LA-ICP-MS U-Pb data for large rutile grains in leucocratic domains

				<sup>207</sup> Pb/ <sup>235</sup> U) /						
	±2σ	<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	( <sup>206</sup> Pb/ <sup>238</sup> U)		<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>235</sup> U
7_G2-3	10	419	17	1.1	2					
7_G2-4	12	437	40	1.3	2					
27 7_L1-1	20	401	31	0.16	1	409	11	415	8.3	0.495
7_L1-2	16	393	26	5.3	exc.					
7_L1-3	18	411	27	0.24	1					
7_L1-4	12	424	21	-0.11	2					
7_L1-6	22	404	26	0.39	1					
28 7_L2-1	16	488	31	0.22	2	462	22	477	13	0.580
7 L2-2	26	440	30	0.32	2					

Table S4a - LA-ICP-MS U-Pb data for large rutile grains in leucocratic domains

Grain	Analyses				Mean rho	Grain diameter (µm)	Grain orientation spread (°)
		±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ			
	1 1_1-1	0.032	0.064	0.001	0.310	352	0.35
	1_1-2						
	2 1_2-1	0.020	0.064	0.001	0.356	660	0.3
	1_2-2						
	1_2-3						
	1_2-4						
	1_2-5						
	3 1_3-1	0.044	0.071	0.041	0.442	366	0.44
	1_3-2						
	4 1_4-1	0.860	0.068	0.077	0.524	512	4.4
	1_4-2						
	5 1_5-1	0.025	0.064	0.001	0.432	481	0.38
	1_5-2						
	1_5-3						
	1_5-4						
	6 1_5-5	0.024	0.063	0.005	0.490	679	3.7
	1_5-6						
	1_5-8						
	7 2_D-5	0.040	0.065	0.004	0.765	511	3.0
	8 2_G-1	0.032	0.064	0.002	0.415	396	0.88
	9 3_C-2	0.068	0.064	0.005	0.585	337	0.82
	10 3_G2-2	0.037	0.066	0.002	0.439	459	0.53
	3_G2-3						
	11 3_K-1	0.030	0.068	0.005	0.327	666	2.1
	3_K-2						
	3_K-3						
	12 4_BI-3	0.059	0.067	0.004	0.503	224	6.1
	13 4_l-1	0.066	0.073	0.004	0.444	368	0.42

### Table S4a - LA-ICP-MS U-Pb data for large rutile grains in leucocratic domains

Table S4a - I	LA-ICP-MS U-P	b data for l	arge rutile	drains in	leucocratic	domains
		b dulu ioi i	arge rame	grams m	leacoolatio	aomanio

	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ			
14 4_M-2	0.054	0.064	0.004	0.589	361	0.54
15 4_N-5	0.034	0.069	0.016	0.288	428	1.5
4_N-6						
16 4_NII-2	0.048	0.071	0.003	0.541	229	0.16
17 5_D-1	0.043	0.068	0.004	0.490	471	0.38
5_D-2						
5_D-3						
5_D-4						
5_D-5						
18 6_B-2	0.079	0.077	0.004	0.357	276	1.4
19 6_G2-2	0.024	0.072	0.004	0.333	311	0.52
6_G2-3						
6_G2-4						
20 6_H1-1	0.017	0.068	0.001	0.474	464	0.34
6_H1-2						
6_H1-3						
6_H1-4						
21 6_H1-6	0.030	0.069	0.002	0.440	315	1.5
6_H1-7						
22 6_H3-1	0.025	0.069	0.002	0.654	392	0.51
6_H3-3						
23 6_J1-2	0.044	0.065	0.002	0.516	328	0.52
6_J1-3						
24 7_E-1	0.058	0.067	0.003	0.343	376	0.5
25 7_G1-3	0.042	0.063	0.004	0.490	774	4.7
7_G1-4						
7_G1-5						
7_G1-6						
7G1-7						
26 7_G2-1	0.018	0.067	0.005	0.371	588	1.1
7 G2-2						

Table S4a - LA-ICP-MS U-Pb data for large rutile grains in leucocratic domains

	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ			
7_G2-3						
7_G2-4						
27 7_L1-1	0.017	0.066		0.537	521	0.38
7_L1-2						
7_L1-3						
7_L1-4						
7_L1-6						
28 7_L2-1	0.480			0.740	320	0.29
7_L2-2						

 $\label{eq:Grain} \begin{array}{cc} \mbox{Grain} & \mbox{Analyses} & \mbox{U}\ (\mbox{ppm}) & \mbox{Pb}_{c} & \mbox{Ratios} \end{array}$ 

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Ages
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		f206%	<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>206</sup> Pb	±2σ	rho	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>235</sup> U
 29 N17-44A1-3	12.3	0.41	0.524	0.013	0.0659	0.0011	0.0577	0.0011	0.64	411	6.4	428
N17-44A.1-6	8.90	0.58	0.484	0.013	0.0644	0.00097	0.0545	0.0013	0.52	402	5.9	401
N17-44A.1-7	8.47	0.79	0.506	0.014	0.0659	0.00099	0.0556	0.0013	0.50	411	6.0	416
N17-44A.1-9	8.70	0.81	0.456	0.013	0.0636	0.00095	0.0521	0.0013	0.49	397	5.8	381
N17-44A.1-11	9.01	0.77	0.504	0.015	0.0665	0.0014	0.0549	0.0013	0.63	415	8.4	414
30 N17-44B.2-2	6.92	1.2	0.500	0.024	0.0642	0.0013	0.0565	0.0024	0.38	401	7.8	412
N17-44B.2-10	5.11	1.1	0.515	0.043	0.0675	0.0014	0.0554	0.0044	0.25	421	8.6	422
31 N17-44C.1-2	10.9	0.92	0.491	0.021	0.0669	0.0013	0.0532	0.0020	0.42	417	8.1	406
N17-44C.1-3	11.0	0.60	0.508	0.014	0.0672	0.0012	0.0549	0.0012	0.61	419	7.3	417
N17-44C.1-4	7.89	0.73	0.517	0.020	0.0663	0.0021	0.0565	0.0014	0.76	414	12	423
N17-44C.1-5	7.14	0.75	0.509	0.020	0.0652	0.0019	0.0566	0.0016	0.68	407	11	418
N17-44C.1-6	6.25	0.95	0.520	0.023	0.0662	0.0030	0.0570	0.0018	0.76	413	18	425
N17-44C.1-8	7.75	1.1	0.522	0.017	0.0673	0.0015	0.0562	0.0015	0.64	420	9.4	426
N17-44C.1-9	5.27	1.3	0.516	0.019	0.0654	0.0016	0.0572	0.0018	0.53	408	9.5	422
N17-44C.1-16	9.47	0.73	0.518	0.017	0.0674	0.0017	0.0557	0.0012	0.72	420	10	424
32 N17-44E.1-2	13.6	0.51	0.476	0.019	0.0636	0.0014	0.0543	0.0018	0.53	397	8.5	395
N17-44E.1-3	13.8	0.46	0.498	0.020	0.0649	0.0015	0.0556	0.0018	0.56	405	9.0	410
N17-44E.1-4	13.0	0.66	0.474	0.018	0.0606	0.0011	0.0567	0.0019	0.44	379	6.6	394
N17-44E.1-5	13.5	0.66	0.489	0.030	0.0634	0.0016	0.0559	0.0031	0.42	396	10	404
33 N17-44G.1-10	10.2	0.65	0.553	0.017	0.0706	0.0016	0.0569	0.0013	0.77	440	9.4	447
N17-44G.1-12	10.8	0.66	0.524	0.013	0.0689	0.0010	0.0551	0.0011	0.37	430	6.2	428
N17-44G.1-13	8.44	0.43	0.544	0.013	0.0703	0.0012	0.0561	0.0010	0.31	438	7.2	441
N17-44G.1-16	7.59	0.41	0.533	0.011	0.0711	0.0010	0.0543	0.00087	0.51	443	6.0	434
N17-44G.1-17	11.1	0.38	0.528	0.011	0.0704	0.00092	0.0544	0.00082	0.66	439	5.5	430
N17-44G.1-2	8.24	1.3	0.526	0.023	0.0697	0.0013	0.0547	0.0021	0.67	434	8.0	429
N17-44G.1-3	9.58	1.1	0.523	0.017	0.0690	0.0019	0.0550	0.0012	0.56	430	11	427
N17-44G.1-4	11.7	0.50	0.521	0.019	0.0685	0.0013	0.0552	0.0019	0.68	427	7.9	426
N17-44G.1-5	15.2	1.4	0.532	0.024	0.0732	0.0014	0.0527	0.0023	0.63	455	8.4	433
N17-44G.1-6	15.8	0.89	0.527	0.014	0.0706	0.0024	0.0541	0.0016	0.64	440	14	430

Grain	Analyses	U (ppm)	Pbc	Ratios							Ages		
			f206%	<sup>207</sup> Pb/ <sup>235</sup> U	±2σ	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>206</sup> Pb	±2σ	rho	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ	<sup>207</sup> Pb/ <sup>235</sup> U
	N17-44G.1-7	9.46	0.86	0.578	0.016	0.0719	0.0015	0.0583	0.0012	0.40	448	9.1	463

Table S4b - SHRIMP U-Pb data for large rutile grains in leucocratic domains

Grain	Analyses	I	Discordance (%)	Mean grain age		Grain diamete (µm)	r Grain orientation spread (°)
			( <sup>206</sup> Pb/ <sup>238</sup> U - <sup>207</sup> Pb/ <sup>235</sup> U	)/			
		±2σ	( <sup>206</sup> Pb/ <sup>238</sup> U)	<sup>206</sup> Pb/ <sup>238</sup> U	±2σ		
29	N17-44A1-3	8.7	-4.0	406	2.8	384	3.9
	N17-44A.1-6	8.9	0.38				
	N17-44A.1-7	9.6	-1.1				
	N17-44A.1-9	8.9	4.0				
	N17-44A.1-11	10	0.15				
30	N17-44B.2-2	16	-2.6	410	7.0	n.m	n.m
	N17-44B.2-10	29	-0.17				
31	N17-44C.1-2	14	2.8	416	3.5	896	2.8
	N17-44C.1-3	9.2	0.52				
	N17-44C.1-4	14	-2.3				
	N17-44C.1-5	13	-2.6				
	N17-44C.1-6	16	-2.9				
	N17-44C.1-8	12	-1.6				
	N17-44C.1-9	13	-3.5				
	N17-44C.1-16	11	-0.79				
32	N17-44E.1-2	13	0.54	393	5.4	n.m	n.m
	N17-44E.1-3	14	-1.2				
	N17-44E.1-4	12	-3.9				
	N17-44E.1-5	20	-2.0				
33	N17-44G.1-10	11	-1.6	437	2.4	483	0.69
	N17-44G.1-12	8.7	0.40				
	N17-44G.1-13	8.6	-0.71				
	N17-44G.1-16	7.4	2.0				
	N17-44G.1-17	7.0	1.8				
	N17-44G.1-2	15	1.2				
	N17-44G.1-3	12	0.69				
	N17-44G.1-4	13	0.30				
	N17-44G.1-5	16	4.9				
	N17-44G.1-6	9.5	2.3				

### Table S4b - SHRIMP U-Pb data for large rutile grains in leucocratic domains

#### Table S4b - SHRIMP U-Pb data for large rutile grains in leucocratic domains

Grain	Analyses		Discordance (%)	Mean grain age		Grain diameter (µm)	Grain orientation spread (°)
		±2σ	( <sup>206</sup> Pb/ <sup>238</sup> U - <sup>207</sup> Pb/ <sup>235</sup> U ( <sup>206</sup> Pb/ <sup>238</sup> U)	) / <sup>206</sup> Pb/ <sup>238</sup> U	±2σ		
	N17-44G.1-7	10	-3.5				