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4	Fe-Ti oxide micro-inclusions in clinopyroxene of oceanic gabbro: phase
5	content, orientation relations and petrogenetic implication
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18	pyroxene, Fe-Ti oxides, crystallographic orientation relations, optimal phase boundary theory,
19	oceanic gabbro
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22	INTRODUCTION
23	Oriented micro-inclusions of Fe-Ti oxides such as magnetite, ulvospinel, hematite,
24	and ilmenite that are hosted in rock-forming silicate minerals like plagioclase and pyroxene
25	are a widespread phenomenon in igneous rocks (Poldervaart and Gilkey, 1954; Divljan, 1960;
26	Neumann and Christie, 1962; Amburstmacher and Banks, 1974; Sobolev, 1990; Usui et al.,

2006; Wenk et al., 2011; Ageeva et al., 2016 and others). Due to their small size these
inclusions often have single magnetic domain state and unusually stable magnetization. Their
stable magnetization and protection from external influence by the mineral-host crystal make
these micro-inclusions important carriers of the paleomagnetic record (Hargraves and Young,
1969; Xu and others, 1997; Renne et al., 2002; Harrison et al., 2002; Smirnov et al., 2003;
Feinberg et al., 2005 and others).

33 The presence of Fe-Ti oxide micro-inclusions in rock-forming minerals is usually attributed to exsolution from the host phase. Special conditions predetermine their 34appearance. Initial crystallization, re-crystallization or alteration of the potential silicate host 35 36 mineral in a high-temperature magmatic or metamorphic environment allows iron and 37 titanium to be incorporated into the silicate lattice at high concentrations. Subsequent cooling, 38 possibly fluid mediated re-crystallization at lower temperature, or at different oxygen fugacity 39 may render the silicate phase supersaturated with respect to Fe-Ti oxide phases and may induce precipitation of Fe-Ti oxides within the silicate host crystal (Smith, 1974). 40

41 Once precipitated the micro-inclusions may undergo various changes like internal 42exsolution, re-crystallization, or chemical changes. It is known that Fe-Ti oxides are highly sensitive to redox state (Sauerzapf et al., 2008; Dégi et al., 2009). When enclosed in a more 4344stable silicate host, the Fe-Ti oxides are less strongly exposed to changes in their geochemical 45environment and somewhat more sheltered from hydration and low-temperature hydrothermal 46 alteration than the matrix phases. Therefore, the phase contents, chemical compositions and 47 microstructures of the inclusions likely reflect the evolution of pressure, temperature and 48 redox state during their primary crystallization and early evolution. Their shape orientation 49relationships (SORs) and their crystallographic orientation relationships (CORs) with their 50mineral hosts provide important constraints on the formation mechanisms and on transformations that the micro-inclusions may have undergone. Combined with the 5152mineralogical and chemical compositions of both the micro-inclusions and the mineral host, the SORs and CORs provide the basis for using Fe-Ti oxide micro-inclusions as indicators of
 post-crystallization conditions and for extracting petrogenetic, petromagnetic and
 paleomagnetic information.

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### Clinopyroxene hosted Fe-Ti oxide micro-inclusions

In this study we address Fe-Ti oxide micro-inclusions hosted in gabbroic 58 59clinopyroxene. In particular, we focus on the crystallographic- and shape orientation 60 relationships between the Fe-Ti oxide micro-inclusions and the host clinopyroxene and try to 61 link different types of the inclusions to specific stages of the petrogenetic evolution. It has 62 been shown earlier (Bown and Gay, 1959; Okamura et al., 1976; Harlow and Klimentidis, 63 1980; Fleet et al., 1980; Woensdregt et al., 1983; Doukhan et al., 1990; Renne et al., 2002; Feinberg, et al., 2004; Hwang et al., 2010) that: (i) there are only two types of shape 6465 orientation and corresponding CORs of needle-shaped magnetite inclusions in a 66 clinopyroxene host. Both types are represented by Fe-Ti micro-inclusions that are flattened parallel to the (010) planes of the clinopyroxene host; (ii) according to their elongation 67 68 subparallel either to clinopyroxene [100] or to clinopyroxene [001] these two types are 69 referred to as X- and Z-type inclusions, respectively (Fleet et al., 1980); (iii) based on the notion that the angle between the needle long-axes of the X- and Z-type inclusions depends on 70 71 their formation temperature, a geo-thermometer has been calibrated that reveals the 72 temperature of exsolution of the Fe-Ti oxide micro-inclusions from clinopyroxene (Fleet et al., 1980; Doukhan et al., 1990; Feinberg et al., 2004). This temperature is of pivotal 73 74 importance in the context of petrogenetic and paleomagnetic reconstructions.

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#### Fe-Ti oxides in the lower oceanic crust

77 The Fe-Ti oxide micro-inclusions in rock-forming minerals of modern oceanic crust 78 gabbros are particularly interesting, since these associations often show a multistage 79 magmatic history and interaction with evolved residual melts enriched in Fe and Ti (e.g., 80 Natland et al., 1991; Malpas and Robinson, 1997; Grimes et al., 2008; Hayman et al., 2011; Hekinian, 2014). This leads to the crystallization of Fe-Ti oxide macro-grains and possibly to 81 82 the enrichment of the rock-forming silicates in Fe and Ti. The crystallization of Fe-Ti oxide 83 macro-grains in oceanic gabbro and the provenance of residual liquids enriched in Fe and Ti, 84 as a result of MORB-type magma differentiation, redox state, and synmagmatic deformation have been debated for many years (e.g., Natland et al., 1991; Agar and Lloyd, 1997; Dick et 85 86 al, 2000). However, the detailed evolution of the Fe-Ti oxide micro-inclusions in the rock-87 forming silicates from oceanic gabbros does not seem to have received sufficient attention.

88 In this communication we focus on the evolution of clinopyroxene-hosted Fe-Ti 89 oxide micro-inclusions in oceanic gabbro that was dredged from the Mid Atlantic ridge at 10°42.95'N, 41°34.60'W. An earlier study (Pertsev et al., 2015) suggests that the 90 91 petrogenetic history of the gabbro includes late magmatic crystallization of Fe-Ti oxides, 92 interaction between a crystal aggregate and residual liquid, and multistage hydrothermal 93 alteration. We report on the shape and crystallographic orientation relationships between Fe-94Ti oxide micro-inclusions, associated amphibole lamellae and a clinopyroxene host. Different 95 types of Fe-Ti oxide micro-inclusions are shown and their attribution to different formation 96 mechanisms is discussed. The temperature of primary formation of the inclusions is inferred 97 based on the geo-thermometer by Fleet et al. (1980) and Feinberg et al. (2004). Multistage 98 phase transformations of the Fe-Ti oxide inclusions are ascribed to changes in temperature 99 and redox regimes during the evolution of the gabbro. The genetic relations between the Fe-Ti 100 oxide micro-inclusions and the associated amphibole lamellae are discussed in terms of 101 possible processes of exsolution and intra-crystalline re-distribution of components in the clinopyroxene host during the late-magmatic and hydrothermal stages. Although the results 102 have been obtained from a single sample, the proposed approach is generally applicable and 103

helps to unravel the provenance and evolution of different gabbro assemblages from modernoceanic crust and ophiolites.

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107 SAMPLE

The gabbro sample – L2612-41 (gabbro-41) – was collected together with more abundant mantle-derived peridotites from the Vema lithospheric section, Central Atlantic (Auzende et al., 1989; Bonatti et al., 2003, 2005) by dredging at  $10^{\circ}42.95'$  N,  $41^{\circ}34.60'$  W, 5195-4620 mbsl (Cipriani et al., 2009). This gabbro-peridotite assemblage was formed close to or within the crust–mantle transition zone near the end of a slow-spreading segment, where thin igneous crust and underlying mantle were affected by intense fracturing and hydrothermal alteration during interaction with seawater (Pertsev et al., 2015).

A detailed mineralogical and petrographic description, including fluid inclusion data 115 116 of gabbro-41 is available in Pertsev et al. (2015). The main petrogenetic stages reconstructed 117 by the latter authors include: (i) early magmatic crystallization of plagioclase + augite  $\pm$ 118 enstatite; (ii) late magmatic formation of Ti-bearing hornblende, Fe-Ti oxides and local re-119 crystallization of plagioclase and augite related to reaction with residual melt at 800-900°C; 120 (iii) early hydrothermal alteration due to interaction with a reducing fluid that was generated 121 by seawater/peridotite interaction; hydrothermal alteration occurred at 560-640°C and caused 122 local crystallization of chlorine-bearing ferropargasite-ferroedenite, formation of olivine 123 coronas between enstatite and magnetite, local enrichment of the anorthite component in 124 plagioclase and replacement of augite by diopside; and (iv) late hydrothermal alteration 125 related to tectonic unroofing and infiltration of seawater or slightly modified seawater (3-9% 126 NaCl) which lead to the formation of albite, chlorite, and actinolite.

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128 METHODS

The main methods used in this study apart from optical polarization microscopy are Scanning Electron Microscopy (SEM) including Electron BackScatter Diffraction analysis (EBSD), Electron Probe MicroAnalysis (EPMA), and Universal stage measurements. All methods are described in detail in the Appendix 1.

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134**RESULTS** 

135 Gabbro mineralogy

The gabbro-41 sample is mainly composed of coarse-grained (3-7 mm sized) primary 136 magmatic plagioclase Pl<sub>1</sub> (An# 44-45), clinopyroxene Cpx<sub>1</sub> (augite), and minor 137 138 orthopyroxene, which all together represent more than 90% of the rock volume. Bent parting 139 cracks in Cpx<sub>1</sub>, banded optical extinction of Pl<sub>1</sub>, and local crystal-plastic deformation features in plagioclase and clinopyroxene indicate deformation, which probably occurred during the 140 141 late-magmatic stage. The late-magmatic mineral assemblage is represented by large interstitial grains of Fe-Ti oxides, a Hbl +  $Cpx_2$  + Pl<sub>2</sub> assemblage and by  $Cpx_2$  + Hbl ± Mt micro-142143aggregates partially replacing early magmatic clinopyroxene Cpx<sub>1</sub> (Pertsev et al., 2015). The 144 late magmatic hornblende (titanian pargasite) is brown and often occurs as blebs in a 145clinopyroxene host. The late-magmatic plagioclase has higher anorthite (An# 55-63) and 146 LREE contents than the early magmatic plagioclase Pl<sub>1</sub> (An#43) (Pertsev et al., 2015). Early 147 hydrothermal hornblende fills a network of cracks within plagioclase Pl<sub>1</sub> and is accompanied by re-deposited plagioclase Pl<sub>3</sub>. It clearly differs in color from late magmatic titanian 148 hornblende and shows pleochroism from dark green to greenish vellow. It is enriched in  $Fe^{2+}$ 149 (Mg# 0.5 to 0.25) and Cl (0.4-1.4 wt.%). This hornblende is mantled by re-deposited 150151 plagioclase, which is enriched in anorthite (An# 49-52) and Sr relative to Pl<sub>1</sub> (Pertsev et al., 2015). Late albite, chlorite, and actinolite are observed along grain boundaries and in parting 152153 cracks of pre-existing clinopyroxene and are ascribed to a late hydrothermal overprint.

In gabbro-41 Fe-Ti oxide micro-inclusions are observed in minerals of different genetic groups including (i) early magmatic plagioclase and clinopyroxene, (ii) late-magmatic plagioclase and clinopyroxene, and (iii) late-hydrothermal actinolite lamellae in clinopyroxene. In this paper we present the results from the clinopyroxene-hosted inclusions. The abbreviations of mineral names correspond to those used by Pertsev et al. (2015).

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#### The Fe-Ti oxide micro-inclusions in clinopyroxene: types, distribution and SORs

161 Rock-forming clinopyroxene of gabbro-41 contains micro-inclusions of Fe-Ti 162 oxides, which mostly take the form of needles or elongated plates or blades flattened parallel 163 to and lying in the (010) plane of the clinopyroxene host (Fig. 1, Fig. 2). The inclusions 164 consist of composite Fe-Ti oxide assemblages containing magnetite + ulvospinel, magnetite + 165 ilmenite + ulvospinel, or magnetite + ilmenite assemblages. Pure magnetite inclusions are 166 rare. Typically, the inclusions are only 0.2 - 2 micrometers thick in the [010] direction of their 167 mineral host, a few micrometers wide and 10-100 micrometers long. In sections parallel to the 168 (010) plane of the clinopyroxene host the Fe-Ti oxide micro-inclusions show two distinct 169 elongation directions. The more abundant elongation direction is sub-parallel to the [001]-170 direction of the clinopyroxene host, the less abundant elongation direction is sub-parallel to 171 the [100]-direction of the clinopyroxene host. Following Bown and Gay (1959) we refer to the 172 two elongation directions as X-type and Z-type inclusions, respectively.

The distribution of the Fe-Ti oxide micro-inclusions is not uniform. The more abundant Z-type inclusions tend to be closely associated with 0.01 to 5 micrometers wide hornblende (Hbl) lamellae extending parallel to the (010) plane of the clinopyroxene host (Fig. 1b, Fig. 2a, c). The amphibole lamellae reach their maximum width near the Fe-Ti oxide micro-inclusions and taper out at some distance from the inclusions. On a larger scale, the Ztype inclusions tend to be concentrated along (100) planes of clinopyroxene (Fig. 1b, Fig. 3b). The less abundant X-type inclusions are primarily observed near big cracks or near the 180 contacts of the clinopyroxene host to large interstitial grains of Fe-Ti oxides, where they occur 181 together with Z-type inclusions (Fig. 1a). Like the Z-type inclusions, the X-type inclusions are 182 usually also accompanied by hornblende lamellae.

A separate generation of homogeneous ilmenite inclusions is associated with the actinolite lamellae lying in the (100) plane of the clinopyroxene host. The ilmenite inclusions take the form of large plates lying in the (100) plane of the actinolite, which are 50 - 100 microns wide. The shape orientation relationships of hornblende (Hbl) and actinolite (Ac) lamellae in the clinopyroxene host as well as the shape orientation relationships of the Fe-Ti microinclusions are shown schematically in Figure 1 b.

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### Crystallographic and shape orientation relations

The (010)-oriented hornblende and (100)-oriented actinolite lamellae have identical crystallographic orientations and show strong CORs with the clinopyroxene host. The (010) and (100) planes of clinopyroxene and amphibole are parallel to each other (Table 1), and the (001) plane of the clinopyroxene is parallel to the (10-1) plane of the amphiboles.

195 The crystallographic orientation relationships between the Fe-Ti oxide phases in the 196 inclusions and the clinopyroxene host are also strong (Table 1, Fig. 4, Fig. 5, Fig. 6). In both 197 the X- and Z- type inclusions the (110) plane of magnetite is parallel to the (010) plane of the 198 clinopyroxene host. In addition, one of the (111) planes of magnetite is parallel to the (-101) 199 plane of the clinopyroxene host in the X-type inclusions, and one of the (111) planes of 200 magnetite is parallel to the (100) plane of the clinopyroxene host in the Z-type inclusions. These crystallographic orientation relationships together with the X- and Z-type shape 201 202 orientations were first described by Bown and Gay (1959), and have been documented 203 repeatedly since then (Bown and Gay, 1959; Okamura et al., 1976; Harlow and Klimentidis, 1980; Fleet et al., 1980; Woensdregt et al., 1983; Doukhan et al., 1990; Renne et al., 2002; 204 Feinberg, et al., 2004; Hwang et al., 2010). The symmetrically equivalent CORs between X-205

and Z-type magnetite inclusions and the clinopyroxene host are summarized in Table 1 and are illustrated in Figure 4.

Despite the fact that the CORs between the Fe-Ti oxide micro-inclusions and the clinopyroxene host are strong, they do not correspond to the shape elongation directions of the Fe-Ti oxide micro-inclusions. Optical measurements with the universal stage microscope showed that the angles between the long axes of the two sets of needle-shaped magnetite Xand Z-type inclusions vary in the narrow range of 106-107°.

The homogeneous ilmenite grains take the form of plates extending parallel to the (001) plane of the ilmenite and oriented parallel to the actinolite lamella boundaries (Fig. 2 b). The ilmenite inclusions show strong CORs with their actinolite host, where the CORs correspond to those of the Z-type inclusions (Fig. 6, Table 1).

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Shape and crystallographic orientation relationships inside of heterogeneous micro inclusions

In the composite Fe-Ti oxide micro-inclusions the intergrown phases show strong crystallographic orientation relationships. Magnetite and ulvospinel have identical crystallographic orientations. Ilmenite adheres to the common ilmenite – magnetite mutual COR with one of the (111) planes of magnetite parallel to the (001) plane of ilmenite and three of the (110) planes of magnetite parallel to the three (100) planes of ilmenite.

The inclusions that consist of magnetite-ulvospinel intergrowths show internal graphic microstructure (Fig. 2 b). The magnetite-ulvospinel phase boundaries are mainly parallel to the {100} planes of the magnetite, which is the most common interface orientation in magnetite-ulvospinel intergrowths (Ramdohr, 1955).

Whereas the ulvospinel phase of the Usp-Mt intergrowths is homogeneous, the magnetite shows a lamellar internal microstructure, which is due to the presence of submicron wide lamellae of supposedly ulvospinel oriented parallel to one of the {100} planes of

the magnetite host (Fig. 2 b). The phase boundaries between the inclusions and the pyroxene host as well as the phase boundaries between the intergrown magnetite and ulvospinel phases inside the inclusions are generally straight. In contrast, when magnetite is accompanied by ilmenite instead of or in addition to ulvospinel, the phase boundaries within the inclusions are uneven and give the impression that the phases are corroded (Fig. 2 c, Fig. 3 a). The corrosion of the magnetite-ilmenite inclusions is particularly pronounced in the vicinity of cracks following the clinopyroxene parting planes.

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#### Mineral host composition

The Fe-Ti oxide inclusions are mainly located in large grains of clinopyroxene, which were identified as the early-magmatic ' $Cpx_1$ ' generation by Pertsev et al., (2015) (Table 2, an. 1, 2). The Fe-Ti micro-inclusions in  $Cpx_1$  are spatially related to amphibole lamellae. The (010) amphibole lamellae (Table 2, an. 5) that are linked to the multiphase Xand Z-type inclusions are closely similar to the Al- and Ti-rich hornblende (titanian pargasite) "Hbl<sub>2</sub>" (Table 2, an. 6) described in (Pertsev et al., 2015).

In the domains where the hornblende lamellae are associated with the "X"- and "Z"type inclusions, the composition of the clinopyroxene host is slightly changed compared to the bulk Cpx<sub>1</sub> and in terms of composition corresponds to the late-magmatic clinopyroxene  $Cpx_2$ " (Table 2, an. 3, 4). Both Cpx<sub>1</sub> and Cpx<sub>2</sub> are classified as augite. However, Cpx<sub>2</sub>, which is associated with the hornblende lamellae, is slightly depleted in Al, Ti and Na and enriched in Si, Ca and Mg with respect to Cpx<sub>1</sub> (Table 2, an.1 and 3; Fig. 7). Nevertheless the concentration of Fe is the same in Cpx<sub>1</sub> and Cpx<sub>2</sub>.

The amphibole lamellae (Table 2, an. 9) that have a shape preferred orientation parallel to the Cpx (100) plane and which host the platy ilmenite inclusions are classified as actinolite ( $Al^{IV}$  0.1 to 0.2, atoms per formula unit). Yet, actinolite lamellae contain irregularly distributed domains of variable hornblende composition with higher concentrations of Al, Fe, and Cl (up to 7.9, 26.0, and 0.02 wt.% respectively (Table 2, an. 7). The ilmenite microinclusions in actinolite (Table 2, an. 12) are more Ti-rich than the lamellar ilmenite (Table 2, an. 11) in the macro-grains of magnetite (Table 2, an. 10)

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#### 262 DISCUSSION

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### 1. Crystallographic- and shape-orientation relationships

Regular crystallographic and shape orientation relationships were documented between rock-forming clinopyroxene and the amphibole lamellae it contains as well as among all phases present in the Fe-Ti oxide micro-inclusions and their clinopyroxene hosts (Fig. 4 a, Fig. 5, Fig. 6).

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### 1.1. Amphiboles

270 The lamellar hornblende and actinolite have similar crystallographic orientation relationships to their clinopyroxene host (Fig. 5, Fig. 6), which most likely reflect the 271 272 geometric (angular) correspondence between the amphibole and clinopyroxene crystal structures (Table 1, Appendix 1). In contrast, the shape orientations of the hornblende and the 273 274actinolite lamellae are different (Fig. 1b) and probably reflect a different formation 275 mechanism. The orientation of the hornblende lamellae parallel to the (010) plane and 276 perpendicular to the "b" axis of clinopyroxene minimizes the lattice misfit, because the largest 277 difference in lattice dimensions between clinopyroxene and amphibole occurs along the "b" 278 axis (Table 1, Appendix 1). The (010)-orientation of hornblende in clinopyroxene is preferentially selected during exsolution (Smith, 1977; Yamaguchi et al., 1978) and may be 279 280 taken as an indication for an exsolution origin of the hornblende lamellae. In contrast, the 281 shape orientation of the actinolite lamellae parallel to the (100) plane of the clinopyroxene 282 does not appear to be controlled by lattice fit. It is rather related to the cracks of parting, 283 where clinopyroxene breaks along planes of structural weakness or along twin planes. Such parting cracks may provide access for fluids allowing hydration and fluid mediated alteration
of pyroxene (Yamaguchi et al., 1978), which is in line with the inferred hydrothermal origin
of the actinolite lamellae.

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### 1.2. Fe-Ti oxide micro-inclusions

For both the X- and Z- type inclusions one of the (110) planes of magnetite is 289 290 parallel to the (010) plane of the clinopyroxene host (Table 1). However, the X- and Z-type 291 inclusions show different orientation relations to the host mineral in all other crystallographic 292 directions, which corresponds to an about 7° rotation of the X- and Z-type magnetite crystal 293lattices with respect to one another around their common [110] direction, which is parallel to 294the "b" axis of the clinopyroxene host (Fig. 4 a). In such mutual orientation, one of the {111} 295planes of magnetite of the X-type inclusions is parallel to the (-101) plane of the 296 clinopyroxene host, and one of the {111} planes of magnetite of the Z-type inclusions is 297 parallel to the (100) plane of the clinopyroxene host. These planes are important in the context 298 of some similarity between the clinopyroxene and magnetite crystal structures. In 299clinopyroxene, densely packed oxygen layers occur parallel to both the (-101)- and the (100)-300 planes, and the {111} planes of magnetite are densely packed oxygen layers as well. Thus, 301 parallel alignment of densely packed oxygen layers is supposedly the prime control on the 302 crystallographic orientation relationships between the studied Fe-Ti oxide micro-inclusions of 303 both the X- and Z-types and their clinopyroxene hosts. This is in line with the findings of 304 Fleet at al. (1980), who infer that the densely packed oxygen layers provide the link between 305 the crystal structures of the inclusion and host phases.

306 Despite of their consistent crystallographic orientations, the elongation directions of 307 the X- and Z-type inclusions are defined by inclusion-host interfaces that follow "irrational" 308 planes in the lattices of both phases. The inclusion-host interfaces neither show accordance 309 with low indexed lattice planes of the magnetite nor of the clinopyroxene. Moreover, the

shape orientations of the X- and Z-type inclusions differ by as much as 106° to 107° (Fig. 4 310 311 b), while the crystallographic orientations only differ by 7° (Fig. 4 a). This phenomenon was 312 first described by Bown and Gay (1959) and since then has been found by several authors (Okamura et al., 1976; Harlow and Klimentidis, 1980; Fleet et al., 1980; Woensdregt et al., 313 3141983; Doukhan et al., 1990; Renne et al., 2002; Feinberg, et al., 2004; Hwang et al., 2010). 315 Fleet et al. (1980) applied the "Optimal phase boundary theory" of Bollmann and Nissen 316 (1968) and suggested that the phase boundaries between the magnetite inclusions and 317 clinopyroxene host that follow "irrational" planes in both lattices are determined by the best 318 geometrical fit between the two crystal lattices at the interface. A detailed discussion of the 319 theory is presented in the Appendix 2. Figure 1 (Appendix 2) is drawn for the best 320 geometrical fit between (111)<sub>mt</sub> and (100)<sub>cpx</sub> in the X-type inclusions and between (-1-11)<sub>mt</sub> 321 and (-101)<sub>cpx</sub> in the Z-type inclusions as is indicated by our EBSD data.

322 The orientation of the optimal magnetite-clinopyroxene phase boundary can be 323 calculated using the equations of Robinson et al. (1977). Combined with thermal expansion 324 coefficients for both magnetite and clinopyroxene, the orientation of the optimum magnetite-325 clinopyroxene phase boundary was calculated for temperatures from 0 to 1000°C (Fleet et al., 326 1980, Doukhan et al., 1990, Feinberg et al., 2004). Based on the temperature dependence of 327 the calculated interface orientation, the angle between the elongations of the X- and Z-type 328 inclusions and the angle between Z-type inclusions and the [001] direction of clinopyroxene 329 can be used as a thermometer for magnetite exsolution in clinopyroxene.

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## 2. Temperatures of inclusions formation

Referring to the "X-Z-angle - Temperature" diagram of Feinberg et al. (2004) the observed angle between the sets of X- and Z-type inclusions of 106,5° indicates that the exsolution of magnetite from clinopyroxene occurred at a temperature in the range of 800 -900°C, thereby suggesting that the inclusions were formed during cooling of either the early336 or late-magmatic clinopyroxene. The spatial association of the magnetite X- and Z-type 337 inclusions with titanian pargasite lamellae and the chemical alteration of the clinopyroxene 338 host in the domains close to the titanian pargasite lamellae, with a composition shift towards 339 the composition of late magmatic clinopyroxene Cpx<sub>2</sub> (according to Pertsev et al., 2015), 340 suggests that the X- and Z-type inclusions were formed during the late magmatic stage of 341 gabbro-41. Application of the amphibole-plagioclase geothermometer of Holland and Blundy 342 (1994) on the compositions of coexisting titanian pargasite and plagioclase yielded 343 temperatures of 830-930° (Pertsev et al., 2015), in line with our estimate from the orientation 344 relations between the X- and Z- type inclusions.

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#### 3. Formation mechanisms of the Fe-Ti oxide micro-inclusions

3.1. X- and Z- types micro-inclusions

348 The close spatial association of the Fe-Ti oxide inclusions with the (010) oriented 349 amphibole lamellae in clinopyroxene suggests their genetic relation. Lamellar intergrowth of 350 pyroxene and amphibole has been described earlier. Amphibole lamellae parallel to the (010) 351 plane of the clinopyroxene host were interpreted as a result of exsolution, implying the 352 existence of a solid-solution between amphibole and clinopyroxene (Smith, 1977; Yamaguchi 353 et al., 1978). The existence of a solid-solution between amphibole and orthopyroxene has 354 been similarly suggested by Desnoyers (1975). Coupled precipitation of (010) oriented 355 lamellae of tremolitic amphibole and magnetite inclusions in metamorphic diopside was 356 explained by a balanced reaction that involves hydrogen as reactant (Doukhan et al., 1990; Feinberg et al. 2004). 357

358 Okamura et al. (1976) described orthopyroxenes containing clinopyroxene lamellae 359 and spinel inclusions. They suggested an exsolution mechanism, where during cooling of an 360 originally non-stoichiometric pyroxene the oxygen framework remains intact, while excess Al 361 cations become increasingly unstable on interstitial and tetrahedral sites and migrate to octahedral sites producing domains with spinel-like substructure. Eventually such domains may reach the critical size for producing stable nuclei and lead to the subsequent growth of spinel. According to Okamura et al. (1976) the passive enrichment of Ca in orthopyroxene next to the growth front of the Ca-free spinel and the originally Ca-bearing orthopyroxene resulted in the formation of clinopyroxene lamellae.

367 The mechanism suggested by Okamura et al. (1976) may also be applied to the 368 formation of the X- and Z-type Fe-Ti oxide inclusions and the associated hornblende lamellae 369 in the clinopyroxene of gabbro-41. In the inclusion-bearing domains, the clinopyroxene host 370 is enriched in Ca, Mg and Si and depleted in Al, Ti and Na relative to the inclusion-free 371 domains. These differences reflect the local re-distribution of Al, Ti, Mn, and K into the 372 hornblende lamellae (Fig. 7) that show titanian pargasite composition typical of magmatic 373 amphibole in oceanic gabbro (e.g., Tribuzio et al., 2000; Coogan et al., 2001). We suggest that 374 the iron accumulation in evolved residual liquid during the late-magmatic stage led to the 375 incorporation of Fe into the lattice of the previously formed clinopyroxene (Cpx<sub>1</sub>). During 376 cooling, precipitation of the excess Fe according to the mechanism suggested by Okamura et 377 al. (1976) resulted in the formation of the magnetite inclusions. During the partial replacement 378 of the clinopyroxene host by magnetite, Ca, Mg and Si were liberated. These components 379 cannot be accommodated in the magnetite structure, and were re-deposited in the remaining 380 clinopyroxene domains along the phase boundaries. This produced an enrichment of the 381 clinopyroxene in Ca, Mg and Si and a concomitant passive depletion of Al, Ti and Na, which 382 share the same crystallographic sites in clinopyroxene. The decrease of the Al, Ti and Na contents and concomitant increase of Ca and Mg in the octahedral "M1" and "M2" sites and 383 384 of Si in the tetrahedral "T" site in the late-magmatic clinopyroxene (Cpx<sub>2</sub>) calculated based on 385 Morimoto (1988) is shown (Table 2). Re-deposition of Al, Ti, and Na (with H<sub>2</sub>O addition 386 from the late-magmatic liquid) in turn resulted in the formation of titanian pargasite. This 387 model is corroborated by the observation that the titanian pargasite lamellae reach their maximum thickness in the vicinity of the Fe-Ti oxide micro-inclusions and taper out away from the inclusions. Alternative mechanisms that suggest exsolution of an amphiboleclinopyroxene solid-solution accompanied by substitution of certain oxygen positions by hydroxyl and partial substitution of  $Ca^{2+}$  by Na<sup>+</sup> and of Si<sup>4+</sup> by Al<sup>3+</sup> (Yamaguchi et al., 1978) cannot explain the observed association of the hornblende lamellae with the Fe-Ti oxide micro-inclusions.

394 Fe-Ti oxide macro-grains crystallized from iron-titanium rich interstitial melt, whereas titanian hornblende seems to form at the same late magmatic stage by interaction 395 396 between gabbro crystal aggregate and magmatic aqueous fluid, as was proposed by Pertsev et 397 al. (2015) based on the Zr abundances in clinopyroxene and plagioclase compositional trends. 398 Since the evolved iron-titanium rich melt and magmatic aqueous fluid appeared to be in 399 equilibrium, the regular association of compositionally similar titanian hornblende and 400 titanomagnetite micro-inclusions seems to be a micron-scale manifestation of the same late 401 magmatic process. Consequently the appearance of Fe-Ti oxide micro-inclusions associated 402 with (010) titanian hornblende lamellae may indicate a definite type of late magmatic gabbro 403 evolution.

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#### 3.2. Ilmenite micro-inclusion

406 Formation of the ilmenite plates in the actinolite lamellae may be attributed to a late 407 hydrothermal alteration process. The actinolite lamellae are oriented parallel to the (100) 408 plane, which is one of the major parting planes of the clinopyroxene host (Fig. 1 c; Fig. 2 b, c; Fig. 7 b). Formation of the actinolite lamellae was likely mediated by access of hydrothermal 409410 fluids along these weak zones. Since the high-field strength element Ti is hardly mobile in hydrothermal solutions (Antignano and Manning, 2008) the Ti originally contained in 411 clinopyroxene (up to 1.1 wt.% TiO<sub>2</sub>, according to Pertsev et al., 2015) was precipitated 412 413 directly in place in the form of ilmenite during the replacement of clinopyroxene by actinolite.

414 Probably the patches with hornblende composition within the actinolite lamellae 415 (Table 2, an. 7) reflect an earlier and higher-temperature hydrothermal hornblende being 416 developed along the same (100) parting planes in clinopyroxene. Such a hydrothermal event 417 with the formation of ferropargasite 'Hbl<sub>3</sub>' (Table 2, an. 8) at about 560-640°C was suggested 418 by Pertsev et al. (2015). Formation of the actinolite-hosted ilmenite plates can thus be 419 explained by re-deposition of the Ti that was liberated during the replacement of the Ti-420 bearing clinopyroxene or a Ti-bearing precursor amphibole by actinolite in the form of 421 ilmenite.

The actinolite-hosted ilmenite plates show crystallographic orientation relationships corresponding to those of the Z-inclusions with the densely packed oxygen layers in the (001) planes of ilmenite aligned with the densely packed oxygen layers parallel to the (100) planes of the actinolite host (Table 1). The observed spatial association of the Z-inclusions and ilmenite plates (Fig. 3 b) may indicate that the Z-inclusions provided nucleation sites for the formation of the actinolite-hosted ilmenite plates.

428 Despite of the fact that the formation of the (100)-oriented actinolite lamellae is 429attributed to late-stage hydrothermal alteration, the same (100) parting planes may have 430 provided access for aqueous magmatic fluid, which was not in equilibrium with the early-431 magmatic clinopyroxene, already during the late-magmatic stage. This may explain the 432 observed large-scale distribution of the Z-type inclusions along the (100) clinopyroxene 433 planes and their spatial association with the actinolite lamellae (Fig. 3 b, Fig. 5). The X-type 434 inclusions seem to form in addition to the Z- type inclusions in areas, where the clinopyroxene host underwent particularly intensive iron enrichment such as in the immediate 435436 vicinity of big interstitial grains of late magmatic Fe-Ti oxides (Fig. 1a).

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### 3.3. Fe-Ti oxide phase content evolution

439 The exceedingly small size and preferred orientation of the X- and Z-type micro-440 inclusions hampered the determination of mineral compositions and of the spatial distribution 441 of ulvospinel and/or ilmenite within the magnetite grains. It was thus not possible to infer the 442 evolution of redox state from the compositions of coexisting Fe-Ti oxide phases. According to 443 O'Neill and Navrotsky, (1984) and Wechsler et al., (1984) the temperature of magnetite-444ulvospinel unmixing depends on fO<sub>2</sub>, cooling rate and the Ti content in the original 445homogeneous titano-magnetite and is always lower than 600°C. Based on the crystallographic 446 orientation relations, the morphology of the phase intergrowth within the individual multiphase Fe-Ti oxide micro-inclusions and the position of the multiphase Fe-Ti oxide 447 448micro-inclusions within the clinopyroxene host we suggest that at the high temperature of the 449late magmatic stage some Ti was dissolved in the primary late-magmatic magnetite precipitates. During post-magmatic cooling, the Ti-bearing magnetite decomposed into an 450451oriented magnetite-ulvospinel intergrowth (Fig. 2 b) with straight, supposedly coherent phase 452boundaries between the intergrown phases. The boundaries occur along the {100} planes of both minerals, which is consistent with magnetite-ulvospinel exsolution microstructures 453454(Price, 1980, Feinberg et al., 2005).

455In the ilmenite-magnetite composite inclusions, ilmenite and magnetite show CORs 456with (111)<sub>mt</sub> parallel to (001)<sub>ilm</sub> and (110)<sub>mt</sub> parallel to (100)<sub>ilm</sub>, but have uneven phase 457 boundaries (Fig. 2 c, Fig. 5). Their spatial distribution is generally controlled by contacts of the micro-inclusions with the clinopyroxene hosted actinolite lamellae (Fig. 5). In the vicinity 458459 of pronounced parting planes the ilmenite and magnetite grains appear strongly corroded (Fig. 460 2 c). Hence, hydrothermal alteration is probably responsible for the appearance of ilmenite in 461 the X- and Z-type composite inclusions. As shown above, the CORs of the homogeneous 462 ilmenite plates were inherited from adjoined Z-type inclusions and may indicate hydrothermal recrystallization of the ilmenite phase of the Z-type inclusions, which was linked to the re-463

464 deposition of Ti in sites, where the Ti-bearing clinopyroxene host was replaced by Ti-free465 actinolite.

- 466
- 467 CONCLUSIONS

468 Strong crystallographic orientation relationships (CORs) among Fe-Ti oxide micro-469 inclusions, associated lamellar amphiboles, and rock-forming clinopyroxene host were 470 established using the EBSD technique. The CORs are determined by the similarity of lattice 471 geometry for the intergrowth of the monoclinic clinopyroxene and amphibole, and by the 472parallel alignment of close-packed oxygen layers of the Fe-Ti oxide micro-inclusions and the 473 clinopyroxene host. The differences in shape orientations between the (010)-oriented titanian 474pargasite and the (100)-oriented actinolite lamellae in clinopyroxene are attributed to 475formation by exsolution and by hydrothermal alteration, respectively.

476 Based on phase content, orientation relationships and associated lamellar amphibole, 477 two genetic types of micro-inclusions were distinguished. A first generation of Fe-Ti oxide 478 micro-inclusions is represented by needle-shaped inclusions represented by intergrowth of 479 magnetite and ilmenite or/and ulvospinel associated with fine lamellae of titanian pargasite. 480 Both the micro-inclusions and hornblende are oriented parallel to the (010) plane of the 481 clinopyroxene host and are attributed to an exsolution reaction, which lead to the 482 redistribution of excess Fe and distinct behavior of major and minor elements in non-483stoichiometric late-magmatic clinopyroxene during cooling. The needle-shaped Fe-Ti oxide 484 micro-inclusions show two distinct elongation directions, with the group of more abundant Ztype inclusions showing elongation sub-parallel to the [001] direction of the clinopyroxene 485 486 host and the group of less abundant X-type inclusions showing elongation sub-parallel to the 487 [100] direction of the clinopyroxene host. The observed elongation directions imply inclusion-host interfaces that follow "irrational" planes of both the minerals. 488

489 Based on the optimal phase boundary geothermometer the angle between the 490 elongation directions of the two sets of needle-shaped micro-inclusions indicates an 491 exsolution temperature of 800-900°C confirming their attribution to the late-magmatic stage. 492 The formation of the Fe-Ti oxide inclusions and of the titanian pargasite lamellae was 493probably related to the same process involving introduction of Fe via a late magmatic fluid or 494 melt, and micro-scale intra-crystalline re-distribution of components, which determines the 495 specific chemical composition of the late-magmatic hornblende and clinopyroxene. A second 496 generation of Fe-Ti oxide micro-inclusions is represented by plate-shaped homogenous 497 ilmenite within actinolite lamellae, which are attributed to a hydrothermal process. The 498 ilmenite is considered to be the result of the re-deposition of Ti during hydrothermal 499replacement of the late magmatic Ti-bearing clinopyroxene or high-temperature lamellar 500 hornblende by actinolite along the (100) parting planes of the clinopyroxene host.

501 Clinopyroxene-hosted Fe-Ti oxide micro-inclusions associated with lamellar 502 amphiboles can serve as reliable indicators of major petrogenetic events in the host rock 503 history. Our example (gabbro-41) shows that Fe-Ti oxide micro-inclusions formed as a result 504 of chemical alteration and phase transformations of primary rock-forming minerals indicating 505 late-magmatic and hydrothermal processes. The evolution of the silicate-hosted Fe-Ti oxide 506 micro-inclusions seems useful to constrain the provenance of different gabbro assemblages 507 from modern oceanic crust and ophiolites.

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### 701 **APPENDIX 1**

702 METHODS

### 703 Electron Backscatter Diffraction Analysis

A mechanically and chemo-mechanically polished carbon-coated thin section was used for Electron Back-Scatter Diffraction (EBSD) analysis. EBSD measurements were performed at the Laboratory for Scanning Electron Microscopy and Focused Ion Beam Applications (Faculty of Geosciences, Geography and Astronomy) at the University of Vienna (Austria) using an FEI Quanta 3D FEG instrument. This FIB-SEM is equipped with a Field Emission Electron Source (Schottky-Emitter) and a Digiview IV EBSD camera, which is mounted at 0° azimuth and 5° elevation angle. Electron beam settings during EBSD data 711 collection were at 15kV accelerating voltage and approximately 4nA probe current (in 712 analytic electron beam mode), while the sample was positioned at 14 mm working distance 713 and at 70° tilted surface leading to a 20° beam incidence angle. The pattern center calibration 714 was carefully fine-tuned based on an excellent pattern taken at a position in the center of the 715 FSE image. The EDAX OIM Data Collection Software versions 6.2 and 7.3 were used for 716 EBSD data collection. Crystallographic orientation maps were processed by EDAX OIM 717 Analysis version 7.3, whereas the single point data were processed using the MTEX toolbox 718 for Matlab (version 4.1.4).

719 A total of 72 single point analyses (17 magnetite/ulvospinel, 36 ilmenite, 16 720 diopside, 3 amphibole data points) were collected manually using a 2x2 binning of the EBSD 721 camera resolution. Each indexing result was optimized by adjusting the Hough settings. The 722 Rho-fraction, minimum Hough peak intensity, minimum peak distance in Hough space as 723 well as the maximum and minimum number of bands used for indexing were varied for each 724 measurement in order to optimize the indexing statistics. The reference structure files listed in 725 Table 1 (Appendix 1), were used for indexing EBSD patterns. Ilmenite showed significant 726 pseudosymmetric misindexing when using the correct reference structure file with trigonal 727 rhombohedral symmetry (H-M Symbol -3). In order to eliminate misindexing, the interplanar angle tolerance was set to 2° and the EBSD band-width additionally was considered when 728 729 indexing ilmenite. For indexing all other phases only the band center position in the EBSD 730 pattern was used disregarding the band width. Actinolite and hornblende could not be 731 distinguished solely by crystal structure. The hornblende reference structure was used for indexing hornblende single point data, whereas for mapping, both amphibole phases were 732 733 indexed using the actinolite reference structure. Resulting orientations from using either 734 amphibole reference structures are identical within the angular resolution of Hough transform 735 based EBSD indexing (Ram et al., 2015).

736 Two crystallographic orientation maps were collected at camera settings of 4x4 737 binning and 64.5 milliseconds exposure time. Hough settings were at a binned pattern size of 140x140 pixels, a theta step size of 0.5°, a Rho-fraction of 91%, a minimum peak distance of 738 9 pixels and a 9x9 pixels convolution mask. The number of bands used for indexing ranged 739 740 from 3 to 15. Map data were collected in hexagonal grid mode at 40 nm step size. In order to 741 increase the fraction of data points with a confidence index value above 0.1, the dataset was 742 cleaned by the confidence index standardization routine and the neighbor orientation 743 correlation method (level 1, i.e. the orientation of data points is changed according to 5 744 identically oriented next-neighbour points). This cleaning procedure leads to 98.8% (scan01) 745or 90.6 % (scan02) of the data points with a CI value > 0.1 compared to 91.4% (scan01) and 746 62.5% (scan02) of the unprocessed datasets.

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### Universal Stage Measurements

749 A universal stage was used for determining the shape orientations of the Fe-Ti oxide 750 inclusions and amphibole lamellae with respect to the lattice of the clinopyroxene host, the 751 crystallographic orientation of which had previously been determined using EBSD. For measuring the angular relations between "X"- and "Z"- type inclusions the thin section was 752 753 positioned so that the (010) plane of the clinopyroxene host was oriented perpendicular to the optical axis of the microscope. In this position the angle between the "X"- and "Z"- type 754 micro-inclusions could be measured directly, because they both lie in the (010) plane of the 755 756 clinopyroxene host. If only one of the inclusion types was in the field of view, the angle between the long axis of the inclusion and the traces of the (100)<sub>cpx</sub> parting planes, which in 757 758 this section are oriented parallel to the microscope optical axis, was determined. The 759 reproducibility of universal stage measurements depended on the size of the inclusions and on 760 the inclination of the (010) plane of the pyroxene host with respect to the thin section surface. Repeated measurements varied from  $\pm 0.5^{\circ}$  to  $\pm 2^{\circ}$ . A total of 50 measurements were used to 761

obtain the mean and standard deviation of the angle between the elongation directions of theX- and Z-type inclusions.

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### Electron probe microanalysis (EPMA)

766 Electron probe microanalyses were performed at the Department of Lithospheric 767 Research, University of Vienna using a Cameca SXFyve electron probe micro analyzer 768 equipped with 5 wavelength- and one energy-dispersive spectrometer. Well-characterized 769 homogeneous natural and synthetic minerals were used as standards. Analyses were done using a focused beam at 15 kV accelerating voltage and 20 nA beam current. Counting times 770 771 were 20 seconds on the peak position and 10 seconds on each background position. To 772 minimize loss of Na due to vaporization, the beam was defocused to a diameter of 5 microns 773 and 10 seconds counting time on the peak position were used for the analysis of feldspar. The 774PAP method (Pouchou and Pichoir, 1991) was used for matrix corrections. The relative error 775 of the laboratory internal standard is below 1%.

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#### 778 **APPENDIX 2**

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### THE OPTIMAL PHASE BOUNDARY THEORY APPLICATION

780 The underlying rationale of the optimal phase boundary theory suggested in 781 (Bollmann and Nissen, 1968) is that phase boundaries between a solid phase precipitate and 782 its host preferentially attain orientations that minimize interfacial energy. According to 783 Bollmann and Nissen (1968) the interfacial energy is minimized for interface orientations 784 corresponding to the best geometrical fit between the two crystal lattices at the interface. In 785 their endeavor to explain the shape and crystallographic orientation relations between 786 pigeonite lamellae and an augite host Robinson, et al., (1971, 1977) and Jaffe et al., (1975) 787 showed that for two monoclinic lattices aligned along their common b-direction and having 788 similar "b" parameter perfect coherency between a set of two lattice planes of each of the 789 lattices exists for two specific crystallographic orientation relations, which are related to each other by a rotation about the common "b" axis. The interface with perfect coherency between 790 791 the two sets of two lattice planes was referred to as "perfect interface" by Robinson, et al. 792 (1971). It has an "irrational" though precisely defined orientation with respect to both lattices. 793 Due to the pressure- and temperature-dependence of the lattice parameters, the orientation of 794 the "perfect interface" changes with pressure and temperature, a property, which has been 795 calibrated as a geothermometer for clinopyroxene-clinopyroxene and clinoamphibole-796 clinoamphibole lamellar intergrowths (Robinson et al., 1971, 1977; Jaffe et al., 1975).

797 Fleet et al. (1980), Doukhan et al. (1990) and Feinberg et al. (2004) applied the 798 relations derived by Robsinon et al. (1971, 1977) to clinopyroxene hosted magnetite 799 inclusions. Fleet et al. (1980) and later Feinberg et al. (2004) suggested a non-primitive 800 monoclinic cell for magnetite, with the monoclinic diad axis parallel to the [110]-direction of 801 magnetite, and a modified monoclinic cell for clinopyroxene. A projection of the monoclinic 802 cells of magnetite and clinopyroxene down their common "b"-direction is shown in Figure 1 803 (Appendix 2). The figure is drawn for perfect alignment of  $(111)_{mt}$  and  $(100)_{cpx}$  in the X-type 804 inclusions and perfect alignment of  $(-1-11)_{mt}$  and  $(-101)_{cpx}$  in the Z-type inclusions as is 805 indicated by our EBSD data. These CORs correspond to a 7° rotation of the two magnetite 806 lattices with respect to one another around their common [110]-direction. It is seen that for Z-807 type inclusions perfect coherency between the (-1-11)<sub>mt</sub> and (-101)<sub>cpx</sub> planes exists along the 808 heavy solid lines that are slightly inclined relative to the clinopyroxene [001]-direction. Similarly, for the X-type inclusions perfect coherency between the (111)<sub>mt</sub> and (100)<sub>cpx</sub> planes 809 810 exists along the heavy solid lines that are slightly inclined relative to the clinopyroxene [100]-811 direction. It is interesting to note that the spacing between the possible coherent interfaces is 812 significantly lower for the Z-type orientation than for the X-type orientation of the magnetite. 813 The Z-type COR is thus more favorable for producing coherency between densely packed 814 oxygen layers at the inclusion-host interface than the X-type orientation. This may explain 815 that the Z-type inclusions are more commonly observed than the X-type inclusions, which is 816 the case in the present study and was documented in earlier studies (Fleet et al. 1980).

"Perfect boundaries" in the sense of Robinsons et al (1971) with coherency of both, the  $(111)_{mt} - (100)_{cpx}$  pair as well as the  $(-1-11)_{mt}$  and  $(-101)_{cpx}$  pairs at the magnetiteclinopyroxene interface would require counterclockwise rotation of the X-type magnetite lattice by about 1.8° and clockwise rotation of the Z-type magnetite lattice by about 0,4°. This would slightly change the orientation and the spacing of possible coherent interfaces but would not change the fact that the spacing between possible coherent interfaces is significantly smaller for Z-type than for the X-type inclusions.

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### 825 **LIST OF FIGURES**

Figure 1. (a) Transmitted light image of clinopyroxene-hosted X- and Z-type inclusions near a big magnetite grain (Mt). (b) Schematic drawing of a (010)<sub>cpx</sub> -oriented hornblende lamella comprising X- and Z-type inclusions and of a (100)<sub>cpx</sub> -oriented actinolite lamella with

ilmenite plates. The angle between the X- and Z-type inclusions that is used as a geothermometer is marked  $\beta^*$ ; Hbl – hornblende, Ac – actinolite, ilm – ilmenite

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Figure 2. Forward-scattered electron (FSE) images (a, b) and secondary electron (SE) image (c) showing X- and Z-type inclusions lying in the (010) plane of clinopyroxene (Cpx): (a) needle-shaped inclusion predominantly composed of magnetite; (b) inclusion represented by magnetite-ulvospinel intergrowth and hornblende (Hbl) lamellae oriented parallel to (010)<sub>cpx</sub>; (c) crack along the (100)<sub>cpx</sub> parting plane crosscutting an altered inclusion, which follows a hornblende lamella in clinopyroxene.



Figure 3. (a) Secondary electron (SE) images of a magnetite-ilmenite Z-type inclusion and an actinolite lamella with ilmenite (Ilm) inclusion; (b) optical reflected light image of ilmenite plates (Ilm) in an actinolite lamella (Ac), and adjoined ilmenite-magnetite Z-type inclusions in the clinopyroxene host. The transversal cracks are oriented sub-parallel to (001)<sub>cpx</sub>, and contain unidentified Fe-oxide or Fe-hydroxide phases, they probably represent altered pigeonite lamellae similar to those described in Robinson at al. (1971).



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Figure 4. (a) Stereographic projection (upper hemisphere) showing the poles of lattice planes 850 851 and lattice directions of the clinopyroxene host (blue circles) and magnetite of X-type 852 inclusions (red) and Z-type inclusions (black) as obtained from EBSD analysis; the 853 crystallographic correspondence between X- and Z-type inclusions and the clinopyroxene host is given in Table 1. (b) Crystallographic and shape orientation relations between Fe-Ti 854 855 oxide micro-inclusions and clinopyroxene host. Projection down the [010] direction of the 856 clinopyroxene host: the long axes of the X- and Z-type inclusions are subparallel to the a- and c- axes of clinopyroxene and corresponding CORs are shown. The angle between the X- and 857 Z-type inclusions that is used as a geothermometer is marked as  $\beta^*$ . 858



Figure 5. (a) EBSD orientation distribution map of clinopyroxene (gray) hosted composite magnetite (blue) – ilmenite (red) Z-type micro-inclusion and associated amphiboles (green), (010)-hornblende and (100)-actinolite lamellae, the crystallographic planes corresponding to the phase boundary orientations are indicated; (b) pole figures of EBSD orientation data with corresponding lattice elements indicated by open circles;



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Figure 6. (a) Orientation distribution map of ilmenite plates (red) in an actinolite (100)lamella (green) within clinopyroxene (gray); (b) corresponding pole figures. The deflection of the actinolite from the main orientation is caused by twinning (bright green).



Figure 7. (a) Composition variation (molar proportions, based on singe analyses) across the transition from  $Cpx_1$  via  $Cpx_2$  to titanian pargasite (Hbl). (b) Al+Ti+Mn+Na+K vs Si+Ca+Mg (molar proportions) in clinopyroxenes and amphibole of gabbro-41 according to our data (circles), and data previously published by Pertsev et al. (2015) (triangles); the clinopyroxenes Cpx<sub>1</sub> and Cpx<sub>2</sub> are marked by white and gray symbols, amphibole Hbl is marked by black symbols; (c) Element map of ulvospinel-magnetite micro-inclusion and associated Hbl lamellae (corresponding to those shown in Figure 2 b) in clinopyroxene.

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# 881 FIGURE (Appendix 2)



Figure 1. Schematic drawing of clinopyroxene lattice in terms of the alternative cell (grey 884 885 solid lines), superimposed are magnetite lattices in terms of the alternative monoclinic cell in 886 X-type and Z-type orientations (black solid lines), the heavy solid lines show interface orientations, where the  $(100)_{cpx}$  planes are perfectly coherent with the  $(111)_{mt}$  planes for the 887 888 X-type inclusions and the (-101)<sub>cpx</sub> planes are perfectly coherent with the (-1-11)<sub>mt</sub> planes for 889 the Z-type inclusions; the insert in the lower right shows the conventional magnetite cell 890 (double dashed lines) viewed down the [110]<sub>mt</sub> -direction, which is parallel to the "b"-891 direction of clinopyroxene.