1   **Revision 1**

2   **Phase relations and formation of K-bearing Al-10 Å phase in the MORB + H$_2$O system:**

3   **Implications for the H$_2$O- and K-cycle in subduction zone**

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21   **Abstract** The potassium (K) and water (H_2O) cycles in subduction zones are predominately

22   controlled by the stability of K- and H_2O- bearing minerals, such as K mica, lawsonite, and

23   dense hydrous magnesium silicates (DHMS). K micas (muscovite or phlogopite) are the

24   principal H_2O and K hosts in subduction zones and Earth's upper mantle and play a significant

25   role in the deep H_2O and K cycles. The Mg-10Å phase, normally appearing in hydrated

26   peridotite in high-pressure experiments, has been considered as an important water-carrier in

27   subducted hydrated peridotite. In this study, we found a K-bearing Al-10Å phase in the MORB +

28   H_2O system (hydrated basalt) at high-pressures according to X-Ray diffraction and stoichiometry.

29   We experimentally constrained its stability field at high pressure. By considering newly and

30   previously documented compositions of the 10Å phase and micas, we confirmed a continuous

31   solid solution or mixed layering between the 10Å phase and K-mica at the interlayer site,

32   suggesting that the K cycle and the H_2O cycle in subduction zones are coupled. From the

33   discussion of the effect of $f H_2O$ on stability of the Al-10Å phase, we conclude that a cold

34   subduction zone can host and carry more bulk H_2O and K into Earth's deep mantle than a hot

35   one. This work expands the stability regions of the 10Å phase from the ultramafic system (Mg-

36   10Å phase) to the mafic system (Al-10Å phase), and emphasizes the significance of the 10Å

37   phase for the deep H_2O and K cycle in subduction zone.

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39   **Keywords** H_2O- and K- cycle; Al-10 Å phase; K-mica; High pressure; Subduction zone

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Introduction

44   Subduction zone fluids play an important role in metamorphism of subducted slabs, the

45   slab-mantle interaction, and the generation of arc magmas (Peacock 1990; Schmidt and Poli

46   1998; Padrón-Navarta et al. 2010). Water can be carried into deep Earth by various hydrous

47   minerals (e.g. amphibole, lawsonite, mica, and DHMS) in a subduction zone and released from

48   the subducted slab via dehydration to trigger melting of the mantle wedge and the production of

49   arc magmas (Wyllie 1988; Tatsumi and Eggins 1995; Poli and Schmidt, 2002; Kawamoto 2006;

50   Hacker 2008). The oceanic basaltic crust, with a bulk composition equivalent to MORB or OIB,

51   is commonly altered to greenschist facies by hydrothermal activity and subsequent interaction

52   with sea water during residence on the oceanic floor (Miyashiro 1973; Alt 1995). The principal

53   hydrous phases in the altered oceanic crust are amphibole, chlorite, zoisite, and clay minerals

54   (Tatsumi and Eggins 1995; Alt 1995; Staudigel 2014). During subduction into Earth's mantle,

55   the dehydration of hydrous minerals in the subducted slab strongly depends on the stability of

56   water-bearing minerals, the subduction depth, and the thermal structure of the subduction zone

57   (Van Keken et al. 2011; Schmidt and Poli 2014). Altered oceanic crust may evolve from

58   greenschist through amphibolite to dry eclogite along a hot subduction path and the

59   accompanying phase relations and dehydration behavior have been widely studied and reviewed

60   (Maruyama and Okamoto 2007; Schmidt and Poli 2014 and reference therein). The isobaric

61   amphibole breakdown at ~ 2.5 GPa, traditionally defining the amphibolite-eclogite-transition,

62   has been held responsible for mass transfer triggering arc magmatism above hot subduction

63   zones (Wyllie 1988; Tatsumi and Eggins 1995). However, most subduction zones, especially in

64   the Phanerozoic ones, usually transforms from lawsonite blueschist to lawsonite eclogite along

65 cold subduction path (Brown 2006; Syracuse et al. 2010; Tsujimori and Ernst 2014). The phase

66 relations from lawsonite blueschist to lawsonite eclogite in cold subduction zone are still not

67 very clear.

68   Lawsonite, $CaAl_2Si_2O_7(OH)_2 \cdot H_2O$, a hydrous index mineral for low-temperature

69 metamorphism, normally occurs in hydrated basaltic rocks at lawsonite blueschist and lawsonite

70 eclogite facies (Maruyama et al. 1996; Poli and Schmidt 2002). Its stability, 3 to 10 GPa below ~

71 800 °C, has been experimentally determined in the MORB + H_2O system (Schmidt 1995;

72 Okamoto and Maruyama 1999). Until now, only about ten lawsonite eclogite occurrences have

73 been documented worldwide, in contrast to the vast number of Phanerozoic cold subduction

74 orogenic belts (Tsujimori et al. 2006). Lawsonite eclogites are rare because of the difficulty of

75 lawsonite preservation during exhumation (Clarke et al. 2006; Whitney and Davis 2006; Wei and

76 Clarke 2011). That is why little attention has been paid to the phase relations in lawsonite

77 eclogites.

78   The 10 Å phase, $Mg_3Si_4O_{10}(OH)_2 \cdot nH_2O$, was synthesized in a simple MgO-SiO_2-H_2O

79 (MSH) system by Bauer and Sclcar 1981 and in a hydrated peridotite system by Fumagalli and

80 Poli 2005. It can contain as much as 13.6 wt.% H_2O (Welch et al. 2006; Fumagalli and Stixrude

81 2007). Under water-saturation condition, talc can transform into the 10 Å phase at pressures

82 between 3 and 5 GPa through the reaction Talc + H_2O = 10 Å phase (Pawley and Wood 1995;

83 Chinnery et al. 1999). The 10Å phase is stable at pressures up to 9.5 GPa, above which it

84 transforms into another hydrous phase, the 3.65 Å phase, in the MgO-SiO_2-H_2O system (Pawley

85 et al., 2011). Due to its significance for the deep H_2O cycle, the physical and chemical properties

86 of the 10 Å phase at high pressure have been widely investigated theoretically (Wang et al. 2004;

87 Fumagalli and Stixrude 2007) and experimentally (Fumagalli et al. 2001; Comodi et al. 2005;

88   Comodi et al. 2006; Welch et al. 2006; Chollet et al. 2009; Pawley et al. 2010; Pawley et al.

89   2011). A phlogopite-type layer structure has been proposed for the 10 Å phase on the basis of

90   neutron powder diffraction on deuterated crystals (Pawley et al. 2004) and structure refinement

91   via X-ray diffraction (Fumagalli et al. 2001; Comodi et al. 2005). Fumagalli and Poli (2005)

92   reported a the 10 Å phase containing about 10 wt.% Al_2O_3 in experiments on hydrous K-free

93   peridotites, which was confirmed by Dvir et al. (2010). Complex interlayering between the 10 Å

94   phase and chlorite has been suggested to account for this peculiar mineral chemistry. Fumagalli

95   et al. (2009) experimentally studied alkalis in phlogopite in metasomatized, K-doped peridotites

96   at high pressure and a possible mixed layering or solid solution between the 10 Å phase and

97   phlogopite was suggested to account for K contents as low as 0.3 p.f.u. in phlogopite formed at

98   low-temperature and high-pressure conditions. Given that talc is a widespread low-temperature

99   and high-pressure mineral in hydrated oceanic crusts, especially in hydrous Mg-gabbro crust

100   (Liou and Zhang 1995; Massonne 2004; Bucher and Grapes 2009; Wei and Clarke 2011), it is

101   likely that talc would transform into the 10 Å phase in a hydrous basalt at water-saturation and

102   high pressure conditions. Our experimental study will focus on the formation of the 10 Å phase

103   in hydrated basalt in a cold subduction environment.

104   K micas (muscovite and phlogopite) are the principal H_2O and K hosts in subduction

105   zones and Earth's upper mantle, they are significant for studying the deep H_2O and K cycle. A

106   systematic increase of the K content of volcanic arc magmas with the depth of the Wadati-

107   Benioff-Zone below arc volcanoes is common. This observation is attributed to the stability of

108   K-bearing minerals (such as K mica) in the subducting slab (Dickinson and Hatherton 1967;

109   Ringwood 1974; Tamura et al. 2007). The understanding of the properties of K mica can reveal

110   the mechanisms transporting H_2O and K into Earth's mantle and generating K-bearing volcanic

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111 arc magmas in the subduction zones. For example, phengite is able to transport K and H_2O down

112 to 300 km depth and will transfer to K-hollandite above 10 GPa (Schmidt, 1996). Dehydration or

113 melting of phengite in the subduction zone could liberate K-rich fluids or melts - the

114 metasomatic agents for the generation of calc-alkaline arc magmas in the mantle wedge

115 (Domanik and Holloway 1996; Schmidt 1996; Tamura et al. 2007). When these K-rich fluids or

116 melts interact with ultramafics at the slab-mantle interface, some H_2O and K will be re-stored in

117 phlogopite in the metasomatized mantle (Wunder and Melzer 2003; Fumagalli et al., 2009;

118 Mallik et al. 2015). Proceeding within the mantle wedge and at the slabs-mantle interface, this

119 process is reflected by phlogopite-spinel peridotites, phlogopite-garnet peridotites, and orogenic

120 phlogopite peridotites in UHP terrains (e.g., Sulu garnet peridotite, China: Zhang et al. (2007);

121 Ulten peridotite: Rampone and Morten (2001); Bardane peridotite: van Roermund et al. (2002)).

122 Recently, Mallik et al. (2015) also experimentally confirmed that slab-derived melt reacts with

123 peridotite to form a phlogopite-bearing pyroxenitic residue. Other experiments have shown that

124 phlogopite can be stable up to 6-7 GPa at 1100 °C in lherzolite and its breakdown is controlled

125 by appearance of K-richterite (Konzett and Ulmer 1999), which will be replaced by Phase X (K_{2-}

126 $_xMg_2Si_2O_7H_x$ with x = 0-1), at approximately 12-13 GPa, 1200 °C (Konzett and Fei 2000). At

127 sub-solidus conditions, Fumagalli et al., (2009) inferred that a solid solution or mixed layering of

128 phlogopite and the 10 Å phase is an important H_2O and K host at relatively low temperatures and

129 high pressures in metasomatized peridotite. However, there has been no systematic study on the

130 stability of K- and H_2O-bearing phases in hydrous basalts at sub-solidus conditions.

131   In order to understand the geochemical behavior of K and H_2O in hydrous basalt in a cold

132 subduction zone, we designed and carried out a series of high-pressure experiments on a natural

133 lawsonite blueschist (Fig. S1 and Table 1) along cold subduction path. In order to compare the

134   results with the stability of phlogopite in hydrous peridotite system, and to determine if the 10 Å

135   phase could be stable in hydrous basalt system, our experimental conditions overlap with those

136   used in previous studies in hydrous peridotite system (Fumagalli and Poli 2005; Fumagalli et al.,

137   2009).

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139   **Experimental procedures**

140   **Starting material**

141   In this study, a natural lawsonite blueschist (2S46) from the Early Paleozoic low-

142   temperature and high-pressure metamorphic belt in the North Qilian Mountains, NW China, was

143   selected as starting material (Song et al. 2005). Its bulk composition was obtained by X-ray

144   fluorescence (XRF) at the MOE Key Laboratory of Orogenic Belts and Crustal Evolution,

145   Peking University. Compared with the global average MORB compositions (McDonough 2014)

146   and starting materials previously used for experiments on lawsonite (Okamoto and Maruyama

147   1999) and phengite (Schmidt 1996) in the MORB + H_2O system, the starting material in this

148   study contains more Na_2O (6.81 wt.%, outside the range of altered MORB), less CaO (4.2 wt.%),

149   and similar K_2O (Table 1). The natural starting material contains ~ 5.6 wt.% bulk water (L.O. I.;

150   Table 1), consistent with the experimentally estimated water content of blueschists (5-6 wt.%) in

151   the MORB + H_2O system (Schmidt and Poli 1998; Okamoto and Maruyama 1999). No

152   additional water was added in the experiments. The modal proportions (volume %) of each

153   mineral in the lawsonite blueschist were visually estimated under the optical microscope by

154   Zhang, et al. (2009) and this study (Table 2): it predominantly consists of lawsonite, glaucophane,

155   chlorite, albite, and quartz, with minor amount of garnet (< 2%; Fig. S1). The starting material

156   contains more than 90 vol.% of water-bearing minerals (glaucophane, lawsonite and chlorite)

157   and about 48 vol. % of Na-rich minerals (Na-amphibole and albite). The CaO of the starting

158   material is almost entirely hosted by lawsonite. Revealed by thermodynamic calculations, the

159   lawsonite blueschist experienced a peak metamorphism at 335-355 °C and 0.8-0.95 GPa, P-T

160   conditions at the transition from the lawsonite blueschist to the epidote blueschist facies (Zhang

161   et al., 2009).

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Experimental conditions and apparatus

164   The starting material was finely ground (< 200 mesh) in an agate mortar under ethanol.

165   To simulate the natural situation in the subduction zone and enable the escape of any H_2O

166   liberated by dehydration, for each experiments, the fine starting material was sandwiched

167   between diamond powder (grain size: 30 - 40 μm, Alfa Aesar, A Johnson Matthey Company; ~

168   0.5 mm thick) in an unsealed platinum tube (outer diameter = 3 mm, length = ~ 2.5 mm) (Fig. 2).

169   Before the sample loading, the platinum tubes were boiled in diluted HCl for 30 min,

170   ultrasonically cleaned in ethanol for 10 min, and then stored in an oven at 110 °C. In order to

171   evaluate the influence of fluid migration on the stability and properties of hydrous minerals at

172   high pressure, we performed two additional experiments in sealed platinum capsules at 4 and 5.5

173   GPa. For the sealed-capsule experiments, the sample-loaded capsule was arc-welded. In both

174   kinds of capsules, the starting materials are in direct contact with the Pt capsule which was not

175   pre-saturated with Fe. Iron loss to the capsule may occur depending on the conditions of the

176   experiments (Johannes and Bode 1978).

177   All high-pressure experiments were performed at pressures from 3.5 GPa to 8 GPa and

178   temperatures of 600 °C to 900 °C at the High Pressure High Temperature Lab of Peking

179   University, using a CS-IV 6×14 MN cubic press. The press consists of six tungsten carbide (WC)

180   anvils, with 23.5 × 23.5 mm^2 sized truncated tips, which are simultaneously pushed by six

181   hydraulic rams. The press can produce stable pressures up to about 10 GPa with three well-

182   calibrated experimental assemblies (Liu et al. 2012a). For experimental runs below and above 6

183   GPa, the assemblies BJC-1 (Fig. 1) and BJC4-6were used, respectively. The cell pressures were

184   determined by using the phase transition points of Bi (I-II at 5.5 GPa, II and III at 2.69 GPa and

185   III-V at 7.7 GPa), Ba (I-II transition at 5.5 GPa) and ZnFe (band gap change at 6.6 GPa and I-II

186   transition at 8.9 GPa). Details of the cell assemblies and calibrations have been described in a

187   previous publication (Liu et al. 2012b). The pressure uncertainties, which largely depend on the

188   accuracy of the calibration reaction, were estimated to be less than 3%. The experimental

189   temperature was measured and controlled with a $Pt_{94}Rh_6$-$Pt_{70}Rh_{30}$ thermocouple (type B),

190   ignoring the pressure effect on its e.m.f.. The temperature gradients are less than 15 °C as

191   checked in a test run at 1200 ^{o}C by three thermocouples located on the top, middle and bottom of

192   the BN pressure medium in the graphite heater. Both pressures and temperatures were

193   automatically controlled during the entire experiments. Samples were first pressurized to the

194   target pressure, then heated at a rate of 100 °C/min, and finally held at the target temperature for

195   sufficient time to reach equilibrium (Table 2). Each experiment was quenched by shutting off the

196   power supply to the furnace, followed by automatic decompression to atmospheric pressure.

197   To identify phases and mineral assemblages of the run products, a piece of the recovered

198   sample was mounted in epoxy resin, and polished for analyses with scanning electronic

199   microscopy (SEM) and electron probe microanalysis (EPMA). The remaining sample materials

200   were ground in an agate mortar under ethanol before obtaining powder X-ray diffraction patterns

201   (XRD).

202   The powder XRD patterns were collected with an X'Pert Pro MPD X-ray diffractometer

203   operated at 40 kV and 40 mA with Cu K-alpha radiation. In order to obtain high-resolution

204   powder XRD pattern for analysis of the crystal structure of the new phase (the Al-10Å phase),

205   we used a collecting time of 60 mins. The XRD patterns were analyzed with the Jade 6.0

206   software and compared with standard X-ray PDF data (International Centre for Diffraction Data)

207   to identify phases. For the new phase-bearing experimental products (BE-3 and BE-4), we

208   refined the lattice parameter of the new phase using the GSAS software package (Larson and

209   Von Dreele 2004). The BSE images and EDS spectra were collected on the polished sections

210   with a high-resolution field emission Quanta^{TM} 650 FEG SEM operated at 15 kV, equipped with

211   an Oxford INCA X-MAX50 250+ EDS. The quantitative chemical compositions of the run

212   products were analyzed with a JEOL JXA-8800R electron microprobe operated at 15 kV

213   accelerating voltage and 20 nA beam current, with a beam diameter of 2 μm and counting time

214   of 20-30s. Synthetic silica (Si) and spessartine (Mn), and natural pyrope (Mg), andradite (Fe, Ca),

215   albite (Na, Al), rutile (Ti), and sanidine (K) were used as standards. Data were reduced with a

216   ZAF correction program. All the analyses were performed at the MOE Key Laboratory of

217   Orogenic Belt and Crustal Evolution, Peking University.

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219   **Experimental equilibrium**

220   The use of natural minerals as the starting material poses challenge for the achievement

221   of equilibrium, especially at low temperatures. At a temperature of 650 ^{o}C, we performed

222   experiments up to 200 hours (Table 2) to ensure equilibrium by sufficiently long experimental

223   duration. Reversals in such a complex system are particularly difficult if not impossible. For the

224   highest temperature runs (900 °C), 50 hours heating was sufficient to equilibrate the experiment,

225   in comparison to 24 hours heating used in a previous study in a similar chemical system (Kogiso

226   et al 1997). For experimental temperatures below 800 °C, we increased the heating duration to

227   more than one hundred hours according to the Arrhenius equation of temperature dependence of

228   reaction rates. The BSE images indicated that most high-temperature experiments ($\geq$ 800 °C)

229   have reached equilibrium, while some lower temperature ($\leq$ 700 °C) runs have indications of

230   local disequilibrium (Fig 2).

231   **Results**

232   **Phase assemblages**

233   Altogether, we conducted 12 high-pressure and high-temperature experiments to

234   determine the phase relations on a lawsonite blueschist from 3.5 GPa to 8 GPa at temperatures of

235   600 °C to 900 °C. Experimental conditions and run products are summarized in Table 2. The

236   phase proportions of run products were determined by a combination of visual estimates from

237   BSE images (e.g., Fig. 2) and mass balance calculations on the basis of bulk composition and

238   mineral compositions.

239   The lowest experimental pressure applied in this study is 3.5 GPa at 650 °C (BE-9).

240   Compared with the starting material, the major changes are disappearance of chlorite and albite,

241   and appearance of more garnet and heterogeneous Na-clinopyroxene (Fig 2a). A small amount of

242   epidote was observed in the high-temperature part of the capsule. The main mineral assemblage

243   changed to glaucophane (30%), lawsonite (28%), garnet (15%), Na-clinopyroxene (25%), and

244   epidote (2%). Because the total amount of garnet and Na-clinopyroxene in this experimental

245   product is still lower than 50%, and lawsonite and glaucophane are still stable, the mineral

246   assemblage in this run product is still attributed to the lawsonite blueschist facies.

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247   At 4 GPa, we carried out two experiments (BE-1-1 and BE-1-2) at 650 °C with different

248   capsules: BE-1-2 in a sealed platinum capsule and BE-1-1 in a diamond powder-sandwiched

249   platinum tube. Both yielded the same mineral assemblages consisting of garnet, Na-

250   clinopyroxene, lawsonite, and a new water- and K-bearing phase: the Al-10Å phase (details are

251   discussed later in the mineral chemistry part), but with different mineral proportions (Table 2).

252   Both experimental products contain more than 75% (garnet + Na - clinopyroxene), ~ 10%

253   lawsonite, and 5-15% of the Al-10Å phase, yet no glaucophane was observed. The observed

254   assemblage represents the lawsonite eclogite facies. The compositions of Na-clinopyroxenes are

255   also heterogeneous in both experiments due to low experimental temperature. The closed system

256   experiment produced more and bigger crystals of the Al-10Å phase than that in open system,

257   indicating that H_2O plays a critical role in stabilizing the Al-10Å phase. The lower amount of the

258   Al-10Å phase and lawsonite in the open system coincides with higher amounts of garnet and Na-

259   clinopyroxene. The comparison of the experimental results at 3.5 GPa and 4 GPa at the same

260   temperature of 650 °C implies that the boundary between lawsonite blueschist and lawsonite

261   eclogite is located at a pressure between 3.5 and 4 GPa. When the experimental temperature is

262   increased to 750 °C at 4 GPa, all pre-existing hydrous minerals are decomposed within 120

263   hours. The mineral assemblage mainly consists of garnet, Na-clinopyroxene, and coesite; only

264   very little the Al-10Å phase was observed in the central part of the sample near the platinum tube,

265   where a local "closed" system persists in the open capsule. The Na-clinopyroxene from this run

266   is almost homogeneous, in contrast to the heterogeneity at low temperature. Lawsonite

267   disappeared completely. The experimental conditions approach the stability field of dry eclogite,

268   and the stability limit of the Al-10Å phase should be close to 750 °C at 4 GPa.

269   At 5 GPa, we performed one experiment at 650 °C, which yielded the same mineral

270   assemblage as the 4 GPa, 650 °C run - garnet, Na-clinopyroxene, the Al-10Å phase, and minor

271   lawsonite (5%). At a temperature of 650 °C, the amount of the Al-10Å phase increased with

272   pressure, from 5% at 4 GPa to 15% at 5 GPa.

273   At 5.5 GPa, we also carried out two experiments (BE-3-1 and BE-3-2) at 700 °C in

274   different capsules (Fig. 3). The same mineral assemblage, consisting of garnet, heterogeneous

275   Na-clinopyroxenes (Fig. 2c), the Al-10Å phase, and minor lawsonite, were observed in both

276   sealed and unsealed capsules. The experiment with sealed capsule produced more and bigger

277   crystals of the Al-10Å phase than with unsealed capsule (Fig. 3), similar to the observation of the

278   experiments at 4 GPa and 650 °C. Higher water content in the system facilitated the formation of

279   more and bigger the Al-10Å phase. When the temperature increased to 800 °C, lawsonite and

280   most of the Al-10Å phase disappear with appearance of minor coesite, which is comparable to

281   the result of the experiment at 4 GPa and 750 °C. At 900 °C, the Al-10Å phase completely

282   disappears and the amount of coesite increases to 10 %. The observed mineral assemblage is

283   consistent with dry eclogite, consisting of garnet, Na-clinopyroxene, and coesite.

284   At 7 GPa and 700 °C, the mineral assemblage consists predominantly of garnet and Na-

285   clinopyroxene, with minor lawsonite and Al-10Å phase (Fig. 2e). When the temperature

286   increased to 750 °C, lawsonite and most of the Al-10Å phase disappear with appearance of

287   coesite. The Na-clinopyroxenes in the run product are homogeneous. The mineral assemblage at

288   8 GPa and 700 °C is the same as that at 7 GPa and 750 °C, indicating that dry eclogite is stable

289   (Fig. 2f).

290

Mineral Chemistry

291

292   The grain size of major minerals (garnet, Na-clinopyroxene, glaucophane, lawsonite and

293   the Al-10 Å phase) in the run products is above 10 μm, big enough to analyze their chemical

294   composition with the electron microprobe. The mineral formulas were calculated from the

295   microprobe data according to charge balance rules with the AX program

296   (http://www.esc.cam.ac.uk/research/research-groups/holland/ax). A mica structure was chosen to

297   calculate the mineral formula of the Al-10Å phase given that the similar structure was refined by

298   XRD data (see below).

Garnet

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300   Garnet can be represented by four end-members: grossular (Grs), almandine (Alm),

301   pyrope (Pyp), and spessartine (Sps). The garnets formed at low temperatures ($\leq 800\ ^{o}$C) show

302   weak compositional zoning (Table S1 and Fig. 2). Their cores have nearly the same

303   compositions as the garnet in the starting material, which plot in the middle of the Grs - Sps -

304   (Pyp + Alm) triangle diagram (Fig. 4a). In comparison to the rim, the cores are characterized by

305   higher Mn content (Fig. 4a). The Ca content of garnet decreases with increasing pressure, while

306   the sum of its Mg content increases with temperature (Fig. 4b). The charge balance calculation

307   showed garnets in the run products contains some Fe^{3+} contents, from 0 to 0.17 p.f.u.; most of

308   them containing 0.10 of Fe3+ p.f.u. The Fe^{3+} content of garnet may be a consequence of Fe

309   alloyed with the Pt capsule causing oxidation of the sample (Merrill and Wyllie 1973; Johannes

310   and Bode 1978).

Na-clinopyroxene

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312   The experiments at low temperatures (≤ 700 ^{o}C) locally contains Na-clinopyroxenes with

313   heterogeneous compositions (Table S2, Fig. 2b, c, e and Fig. 4c). The compositional

314   heterogeneity may be attributed to local disequilibrium at low temperature. The jadeite content of

315   Na-clinopyroxene in this study is much higher than that in normal MORB composition at the

316   same PT condition because of higher Na_2O content in the starting material. The run products

317   show considerable aegirine - augite (Fe^{3+}) content in Na-clinopyroxenes and andradite content in

318   garnet, likely caused by alloyed Fe with Pt capsule. The experiments at high temperatures (≥ 800

319   ^{o}C) always show homogeneous Na-clinopyroxene (Fig. 2d).

Amphibole

321   Amphiboles only occur in the starting material and in the experiment at 3.5 GPa and

322   650 °C. All are Na-amphiboles (Fig. 4d). In the starting material, they show obvious

323   compositional zoning, from glaucophane cores to crossite rims. The composition of Na-

324   amphibole in the experimental run at 3.5 GPa and 650 °C is the same as the core composition of

325   the starting material (Table S3 and Fig. 4d). Compared to the compositionally zoned Na-

326   amphibole in the starting material, the Na-amphiboles in the experimental run are more

327   homogenous and their compositions plot in the glaucophane region (Fig. 4d).

Lawsonite and epidote

329   Lawsonites in all experimental products are homogenous and the composition is close to

330   the endmember mineral formula [$CaAl_2Si_2O_7(OH)_2.H_2O$]. Only minor Ca (< 0.14 p.f.u.) and Al

331   (< 0.11 p.f.u.) are substituted by (Fe, Mg) and Si, respectively. A small amount of epidote

332   formed in the experimental run at 3.5 GPa and 650 °C. In comparison to the end-member mineral

333   formula of epidote [$Ca_2FeAl_2[SiO_4][Si_2O_7]O(OH)$], the $Fe^{3+}/(Fe^{3+}+Al)$ ratio of epidote varies

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334   from 0.15 to 0.34. Some Ca in epidote is substituted by Fe^{2+}, similar to the substitution in

335   lawsonite (Table S4).

336

The Al-10 Å phase

338   At pressures above 4 GPa, glaucophane completely disappeared and a new hydrous phase

339   formed. The XRD data of the recovered sample from experiment (BE-3) at 5.5 GPa and 700 °C

340   show a characteristic peak, which is similar to the phengite or the (Al-bearing) 10Å phase

341   (Fumagalli et al. 2001; Fumagalli and Poli 2005) (Fig. S2). The BSE images of this new phase

342   also appear like a typical phyllosilicate (Fig. 3b), showing the layered structure of phengite or the

343   10Å phase. However, the chemical composition of the new phase is different from neither those

344   of phengite nor the 10Å phase. The new phase has high Al, K, and Si contents and low Mg

345   content, compared to the composition of the 10Å phase (Table 3). The EMP analyses show

346   rather low totals (81 - 94 wt.%), likely reflecting variable H_2O contents (6-19 wt.%). We used

347   the phengite structure parameters (Smyth et al., 2000) to refine the lattice parameters of the new

348   phase. The lattice parameters of the new phase (a = 5.1955Å, b = 9.0705Å, c = 20.1544Å) were

349   determined by Rietveld refinement of the XRD data of BE-3 (Fig. 5) using the GSAS software

350   package (Larson and Von Dreele 2004). They are similar to the lattice parameters of hexagonal

351   phengite (a = 5.32046Å, b = 9.0368Å, c = 19.8864Å; Smyth et al., 2000). Compared to the

352   structure parameters of the 10Å phase (a = 5.3231Å, b = 9.2031Å, c = 10.2161Å; Comodi et al.

353   2005), the c lattice parameter is almost doubled.

354   We calculated the mineral formulas of the new phase using the AX program assuming

355   mica structure (Table 3). In comparison to phengite [$K(Mg,Fe)Al(Si_4O_{10})(OH)_2$], the new phase

356   has lower K contents (0.03 - 0.44 p.f.u.) and contains more H_2O (6 - 19 wt.%). The high H_2O

357   content would be more consistent with the (Al-bearing) 10Å phase [$Mg_3(Si_4O_{10})(OH)_2 \cdot nH_2O$]

358   (Fumagalli et al 2001; Fumagalli and Poli 2005). However, the (Fe^{2+} + Mg) content of the new

359   phase (< 0.87 p.f.u.) is much lower than those known for the 10Å phase (= 3 p.f.u.; Fumagalli et

360   al 2001), while its Al content is distinctly higher (= 1.32 - 2.02 p.f.u.). Noticeably, the new phase

361   always contains significant K, between 0.03 and 0.44 p.f.u. Given that the new phase has a

362   similar structure to phengite (K-mica) and the 10Å phase and that solid solution or mixed

363   layering between phlogopite (K-mica) and the 10Å phase is possible for low-K phlogopite

364   (Fumagalli et al. 2009), we suggest the new phase, which is also a solid solutions or mixed

365   layering between K-mica and the 10Å phase. However, the new phase discovered here is not the

366   conventional 10Å phase, which is considered as the Mg-endmember of a solid solutions series.

367   Here, we propose a substitution of 3(Mg + Fe^{2+}) by 2Al on the octahedral site. Accordingly, the

368   chemical composition of the new phase is closer to the composition of an Al-endmember (Table

369   3). We will therefore refer the new phase to "Al-10Å phase" throughout the text and, for clear

370   distinction, we will use "Mg-10Å phase" for the conventional 10Å phase hereafter. It is known

371   that the Mg-10Å phase can form in the talc, $Mg_3[Si_4O_{10}](OH)_2$, and H_2O system at 3-5 GPa

372   through the reaction Talc + H_2O = 10Å phase (Pawley and Wood 1995; Chinnery et al., 1999).

373   Accordingly, the Al-10Å phase proposed in this study may be a high pressure phase in the

374   pyrophyllite, $Al_2[Si_4O_{10}](OH)_2$, and H_2O system. Further experiments are needed to verified this

375   hypothesis.

376   **Discussion**

377   **Formation and stability of the Al-10Å phase in the MORB + H_2O system**

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378   All previous studies on the stability and the physical and chemical properties of the Mg-

379   10Å phase concerned the hydrated peridotite model system with emphasis on the MSH (MgO-

380   SiO$_2$-H$_2$O) system. There is a limited understanding of the effect of CaO, Al$_2$O$_3$, FeO, Na$_2$O, and

381   K$_2$O on the properties of the Mg-10Å phase (Fumagalli et al. 2001; Comodi et al. 2005; Comodi

382   et al. 2006; Welch et al. 2006; Chollet et al. 2009; Pawley et al. 2010; Pawley et al. 2011). So far,

383   the Mg-10Å phase has been thought to be present only in the hydrated ultra-mafic system at high

384   pressure (Kawamoto 2006). In this study, we report a new (Al, K)-bearing 10Å phase (Al-10Å

385   phase) formed in the hydrous mafic (MORB + H$_2$O) system. Given that the Mg-10 Å phase can

386   be synthesized from talc in a water-saturated system at high pressure (Pawley and Wood 1995;

387   Chinnery et al. 1999) and that talc is a common mineral in hydrous, low-temperature and high-

388   pressure metamorphic mafic rocks (especially of Mg-metagabbro) (Liou and Zhang 1995;

389   Massonne, 2004; Bucher and Grapes 2009; Wei and Clarke 2011), talc could transform into the

390   Mg-10Å phase in water-saturated meta-mafic rocks at higher pressure. The synthesis of the Al-

391   10Å phase in natural lawsonite blueschist in this study supports this hypothesis. The Al-10Å

392   phase synthesized in this study has distinctly high Al$_2$O$_3$ content compared to the traditional Mg-

393   10Å phase. Fumagalli and Poli (2005) found notable Al$_2$O$_3$-content (about 10 wt. %) in an Mg-

394   10Å phase in hydrous K-free peridotites (Na$_2$O-CaO-MgO-FeO-Al$_2$O$_3$-SiO$_2$ system); they

395   suggested that complex interlayering between the Mg-10Å phase and chlorite accounts for this

396   peculiar mineral chemistry. The chemical composition of the starting material in this study has a

397   much higher Al$_2$O$_3$ and lower (FeO + MgO) content (Table 1) than that of normal peridotite. The

398   observed Al-10Å phase is close to an Al-endmember. We expect a continuous solid solution on

399   the octahedral site between the Mg-10Å phase and the Al-10Å phase. The Al$_2$O$_3$-bearing 10Å

400   phase reported in previous studies (Fumagalli and Poli 2005; Dvir et al 2010) is likely an Al-

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401 bearing Mg-10Å phase, while the Al-10Å phase synthesized in this study is a Mg-bearing Al-

402 10Å phase.

403 　　　To understand the formation of the Al-10Å phase in the MORB + H_2O system, a ternary

404 A[Al_2O_3] - C[CaO] - F[(Mg+FeO)] diagram (with SiO_2 and H_2O in excess) is utilized (Fig. 6). It

405 illustrates typical protoliths of a wide range of metamorphic rocks (e.g. basaltic and ultrabasaltic

406 rocks) and possible metamorphic minerals (e.g. lawsonite and 10Å phase). Located on the side

407 between the Al_2O_3 and (Mg + FeO) corners in the ACF diagram, the composition of the 10Å

408 phase varies from the Al-10Å phase to the Mg-10Å phase (Fig. 6). The chemical composition of

409 the starting material of this study (lawsonite blueschist) plots in the ultrabasaltic rock field

410 instead of the basaltic rock field, although its mineral assemblage and bulk composition were

411 assigned to altered MORB. This is because starting material in this study contains much less

412 CaO (4.2 wt.%) and more Na_2O than normal altered basalts. In this study, the higher Na_2O

413 content of the starting material can stabilize Na-amphibole to higher pressure (Pirard and

414 Hermann 2015). The starting materials in this study are more consistent with ultrabasaltic than

415 basaltic rocks if the effect of Na_2O on phase relations of Na_2O-free minerals in the experimental

416 products can be ignored. Because the Mg-10Å phase is common in hydrous Mg-rich ultrabasaltic

417 rocks at high pressure and low temperature conditions, it is consequential that we synthesized the

418 Al-10Å phase in a hydrous, Al-rich but CaO-poor basaltic composition. Based on the

419 experimental results in this study, Na_2O-rich amphibole (glaucophane) and lawsonite can be

420 stable and are capable to carry K and H_2O to pressures up to 4 GPa. K and H_2O released by the

421 decomposition of lawsonite and Na_2O-rich amphibole with increasing pressure is re-stored in the

422 new K-bearing Al-10Å phase. A possible reaction for the formation of the Al-10Å phase can be

423 simply determined by Schreinemakers rules in the ternary ACF diagram: Lawsonite +

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424   Amphibole$_{ss}$ = Pyroxene$_{ss}$ + The 10Å phase. The occurrence of this reaction is supported by local

425   texture in our experimental runs showing the formation of the Al-10Å phase around relic

426   lawsonite (cf., Fig. 2c). The exact formation reaction of the Al-10Å phase need further

427   themodynamical calculation after we get enough data for the new phase.

428   The results of our high-pressure experiments defined the stability field of the Al-10Å

429   phase in the MORB + H$_2$O system (Fig. 7). The Al-10Å phase appears in run products from 4

430   GPa, 650 °C via 5.5 GPa, 800 °C to 7 GPa, 750 °C (Table 2). The stability field of the Al-10Å

431   phase is distinctly broader than that of the Mg-10Å phase in hydrated peridotite (Fumagalli and

432   Poli 2005; Pawley et al 2011) (Fig. 7). This work expands the stability region of the 10Å phase

433   from the ultramafic system to the mafic system, and highlights the significance of the 10Å phase

434   in deep H$_2$O and K cycles in subduction zones.

435   **Possible solid solution/mixed layering between K-Mica and the 10Å phase on the interlayer**

436   **site**

437   Numerous studies have focused on the structure, stability, and dehydration of the Mg-

438   10Å phase (Fumagalli et al. 2001; Fumagalli et al. 2005; Comodi et al. 2006; Welch et al. 2006;

439   Fumagalli and Stixrude 2007; Chollet et al. 2009; Pawley et al. 2010; Pawley et al. 2011). Both

440   neutron powder diffraction on deuterated Mg-10Å phase (Pawley et al 2004) and X-ray powder

441   diffraction (Fumagalli et al 2001; Comodi et al. 2005) indicate that the Mg-10Å phase has a

442   phlogopite-type stacking structure. A possible mixed layering or solid solution between the Mg-

443   10Å phase and phlogopite has been used to explain the low K content in phlogopite that was

444   synthezied in a K-doped peridotite at low temperature and high pressure conditions (Fumagalli et

445   al., 2009). We suggest that the Al-10Å phase and phengite (K-mica) can also coalesce by

446   continuous solid solution or mixed layering on the interlayer site.

447   In order to test the existence of solid solution or mixed layering between mica and the

448   10Å phase at the twelve-fold coordinated site, we plotted previously reported compositions of

449   natural and synthetic micas and (Al-bearing) 10Å phases in a (Ca + Na) - K - H_2O triangular

450   diagram (Fig. 8). The triangular diagram shows continuous composition between the 10Å phase

451   and K-Mica, but a distinct gap between Na-Mica and K-Mica, consistent with the previously

452   reported coexistence of paragonite and phengite in blueschist and eclogite (Ahn et al., 1985). The

453   limited data available do not permit conclusions on the compositional variation between Na-mica

454   and the 10Å phase. Previous studies also have paid some attentions on non-stoichiometric K on

455   the twelve-fold coordinated site of micas (as low as 0.7 p.f.u.), especially for those formed at low

456   temperatures and high pressures (Wang and Banno 1987; Torre et al 1996; Lü et al 2008). The

457   illite/talc substitution [$K^{XII} + Al^{IV} = \square^{XII} + Si^{IV}$] has been proposed to explain non-stoichiometric

458   K on the twelve-fold coordinated site of micas, based on study of the miscellaneous isomorphous

459   substitution in K white micas (Wang and Banno 1987; Guidotti and Sassi 1998; Guidotti and

460   Sassi 2002; Fumagalli et al. 2009). However, the model of vacancies on the twelve-fold

461   coordinated site cannot explain "excess" water and low K content in some K-micas from typical

462   metamorphic rocks (Hervig and Peacock 1989) and in the Al-10Å phase in this study. Fumagalli

463   et al (2009) suggested a possible 10Å phase substitution ($K^{XII} + Al^{IV} = H_2O^{XII} + Si^{IV}$) to explain

464   the high water and low (K + Na) content in phlogopite from metasomatized peridotites. We

465   plotted previously reported composition of synthetic or natural micas and 10Å phases in a

466   (Na+K)/(Na+K+H_2O)-Si diagram (Fig. 9). The three endmembers (red filled circles) of this

467   diagram are mica, phengite, and 10Å phase. There are two evolutional trends during increasing

468   pressure, the substitution $Al^{VI} + Al^{IV} = Mg^{VI} + Si^{IV}$ at relatively high temperature, and the

469   substitution $K^{XII} + Al^{IV} = H_2O^{XII} + Si^{IV}$ at relatively low temperature. Both can cause the

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470   increase of the Si content in mica with pressure. However, molecular H_2O can be brought into

471   the mica structure only at relatively low temperatures and high pressures (Fig. 9). The

472   substitution $K^{XII} + Al^{IV} = H_2O^{XII} + Si^{IV}$ can also explain why the K_2O content in phengite

473   decreases with increasing bulk H_2O content at subsolidus conditions (Rosenthal and Frost 2014).

474   The observation supports that the mica-10Å phase solid solution or mixed layering at the twelve-

475   fold coordinated site forms a continuous series and its composition shifts to the 10Å phase

476   endmember at high pressures, especially at low temperatures. Further detailed structure studies

477   (XRD and TEM) are needed to better understand the structure evolution between K-mica and the

478   10Å phase.

479

480   **The effect of fH$_2$O on the Al-10Å phase**

481   We conducted experiments with sealed and unsealed capsules to understand the effect of

482   the water fugacity (fH$_2$O) on the formation and composition of the Al-10Å phase. The sealed

483   platinum capsule represents a relatively closed system, while the unsealed platinum tube with

484   diamond powder represents a relatively open system. From 4 GPa to 5.5 GPa, the amount of

485   lawsonite decreased, while the amount of the Al-10Å phase increased. At 4 GPa and 650 °C, the

486   Al-10Å phase forms relatively small grains (less than 10 μm) in the run products of both open

487   and closed systems. However, the amount of the Al-10Å phase formed in the open system is

488   about 5%, much lower than that in the in closed system (15%). At 5.5 GPa and 700 °C, the

489   amount of the Al-10Å phase increases to 15 % in the open system and to 25 % in the closed

490   system.

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491   At 5.5 GPa and 700 °C, the grain sizes of the Al-10Å phase are larger in the closed than

492   in the open system. For example, grains of the Al-10Å phase in the closed system grow to 50 μm

493   across. The large Al-10Å phase crystals always contain small Na-clinopyroxene and garnet

494   inclusions (Fig. 3a and b). However, in the open system, the Al-10Å phase is characterized by

495   euhedral sheets of about 15-μm length and about 5-μm width (Fig. 3c and d). The composition of

496   the Al-10Å phase also differs between the open and closed systems, with a higher K but lower

497   H_2O content in the open system (Table 3). It is expected that the fH_2O is higher in the closed

498   system than in the open system. High fH_2O enhances the formation of the Al-10Å phase and

499   crystal growth. It also leads to high H_2O/K ratio on the twelve-fold coordinated site.

500   **Coupled K and H_2O cycle in subduction zones**

501   K micas (muscovite and phlogopite) are the principal H_2O and K hosts in the crust. They

502   play a key role in deep H_2O and K cycles in subduction zones and Earth's upper mantle. As

503   discussed, it is evident that the properties of the solid solution/mixed layering between K mica

504   and the 10Å phase are controlled by the H_2O/K ratio, pressure and temperature conditions. From

505   the subduction zone perspective, the K cycle in the subduction zone should be coupled with the

506   H_2O cycle, especially in cold subduction zones. The starting material in this study contains 0.16

507   wt.% K_2O, representing the normal oceanic crust composition. All K_2O is hosted by glaucophane

508   in the starting material. Along cold subduction path, glaucophane will disintegrate completely

509   and liberate the bulk K_2O and significant H_2O. Meanwhile, the subducted basaltic rock will

510   transform into lawsonite and/or Al-10Å phase eclogite, and most K and H_2O released by

511   glaucophane decomposition will be re-stored in the solid solution/mixed layering of K-Mica and

512   10Å phase. This differs from the previous conclusion that descending oceanic crust releases all

513   its K when amphibole breaks down during relatively hot subduction (Tatsumi 1989; Tatsumi and

514   Eggins 1995).

515   We plotted typical PT-paths of cold, warm, and hot subduction on a phase diagram for

516   the MORB + H_2O system (Okamoto and Maruyama 1999) together with the stability regions of

517   phengite (Schmidt 1996) and the Al-10Å phase (this study) to discuss the coupled K and H_2O

518   cycles in different subduction scenarios (Fig. 10). Before the onset of subduction, the bulk K in

519   an altered MORB is stored in amphibole or mica. Along the hot subduction path, all K will be

520   released from the down-going slab due to dehydration melting of amphibole or mica at depths

521   less than 50 km. In this scenario, K cannot be transported into Earth's mantle beyond 50 km. On

522   the other hand, in a cold subduction zone, K in micas will remain in phengite, while K in

523   amphibole will be released by amphibole decomposition at depths of 80-100 km and be restored

524   in the solid solution/mixed layering of phengite and the 10Å phase. The solid solution/mixed

525   layering of phengite and the Al-10Å phase will be stable up to 8 GPa and then decompose into

526   K-hollandite and a K-rich fluid. Some K will be released from the subducted crust dissolved in

527   fluid, while other K will be incorporated into K-bearing omphacite through the reaction phengite

528   = $KAlSi_2O_6$-clinopyroxene + enstatite + coesite + K-rich fluid and transported into deeper mantle

529   (Schmidt 1996). The K and H_2O cycles in subduction regimes along hot or cold path should be

530   between both scenarios. For the example of a warm slab (Fig. 10), most of the K in amphibole

531   will be released into the fluid with amphibole decomposition at a depth of about 100 km, while

532   the release of the K in phengite always depends on the intersection of the subduction path and

533   the K-bearing MORB solidus. Of course, K can partly be restored into K-omphacite and

534   transported into Earth's deeper mantle. The amount of K stored in K-omphacite is controlled by

535   the pressure and the amount of omphacite in the subduction zone (Schmidt 1996; Han et al.,

536   2015).

537   According to the discussion on the effect of fH$_2$O on the Al-10Å phase, the H$_2$O/K ratio

538   can also play a significant role in the coupled H$_2$O and K cycle in the subduction zone. The

539   higher the H$_2$O content in the system, the more Al-10Å phase will be produced, leading to more

540   bulk K and H$_2$O into deeper mantle by the K-bearing Al-10Å phase. If the H$_2$O content of the

541   system is low, all of its bulk K could be stored in phengite and released by decomposition or

542   melting of phengite with increasing pressure and temperature. According to previous

543   thermodynamic calculations and high-pressure experimental constraints of phase relations

544   (Maruyama and Okamoto 2007; Wei and Clarke 2011), the subducted basaltic rocks can host and

545   bring much more H$_2$O into Earth's mantle in cold subduction zones than in hot ones. The high

546   fH$_2$O in a cold subduction zone will generally enhance the bulk K transportation to deep mantle

547   through the Al-10Å phase.

Implication

549   In this study, we found K-bearing Al-10Å phase in a hydrated basaltic rock at the high

550   pressures of the lawsonite eclogite facies and constrained its stability in the MORB + H$_2$O

551   system. The K-bearing Al-10Å phase has a similar and much smaller stable field than lawsonite

552   and phengite, respectively. This work expanded the knowledge of the 10Å phase from the

553   ultramafic system (previous studies) to the mafic system and emphasized the significance of the

554   10Å phase for the deep water and K cycle in subduction zones. Based on discussion of coupling

555   of the K and H$_2$O cycle in subduction zones and the effect of fH$_2$O on the Al-10Å phase, we

556   conclude that cold subduction zones can host and carry more H$_2$O and K deep into Earth's

557   mantle than hot ones.

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Figure captions

 Fig. 1 Experimental assembly (BJC-1) with the cubic press CS-IV 6 × 14 MN installed at the High-pressure Laboratory, Peking University. The starting materials are sandwiched between diamond powder in a Pt tube.

Fig. 2 Selected backscattered electron (BSE) images of representative experimental products, with experimental conditions indicated on the figures. **(a)** Run BE-9: Coarse-grained glaucophane in a fine-grained matrix of garnet, Na-clinopyroxene, and small amount of lawsonite. **(b)** Run BE-1: Fine-grained Al-10Å phase in a lawsonite - bearing garnet - Na-clinopyroxene assemblage. **(c)** Run BE-3: Medium-grained Al-10Å phase in a lawsonite - bearing garnet - Na-clinopyroxene assemblage. **(d)** Run BE-5: Coesite in a garnet - Na-clinopyroxene assemblage. **(e)** Run BE-13: Fine-grained Al-10Å phase and lawsonite in a garnet - Na-clinopyroxene assemblage. **(f)** Run BE-8: Homogeneous coesite-bearing garnet - omphacite assemblage. The mineral abbreviation follow Whitney and Evans (2009) in all figures.

814

Fig. 3 BSE images of run products at 5.5 GPa and 700 °C experiments in different capsules. **(a)** Run BE-3-2: Large Al-10Å phase crystals with inclusions of garnet and omphacite also building up the matrix. **(b)** Al-10Å phase with garnet and omphacite inclusions (enlarged view of a). **(c)** Run BE-3: Euhedral sheet-like Al-10Å phase in a matrix of garnet and omphacite. **(d)** sheet-like shaped Al-10Å phase (enlarged view of c).

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820

821   **Fig. 4** Compositional characteristics of garnet, Na-clinopyroxene, and amphibole in run products

822   in comparison to that in the starting material. **(a)** Obvious compositional differences between

823   garnet core and rim. **(b)** Substitution of Ca by (Mg + Fe + Mn) in garnet with increasing pressure.

824   **(c)** Ternary jadeite (Jd) - aegirine (Aeg) - diopside (Wo + En + Fs) - diagram after (Morimoto, et

825   al., 1988) showing compositions of Na-clinopyroxene in the run products. **(d)** Classification of

826   amphibole in run products and starting material in the $Mg/(Mg+Fe^{2+})$ - $Fe^{3+}/(Fe^{3+}+CAl)$ diagram

827   after (Leake, et al. 1997).

828   **Fig. 5** X-ray diffraction profile of run product (BE-3-1) at 5.5 GPa and 700 ^{o}C. The continuous

829   red line is the calculated profile obtained by Rietveld refinement using the software package

830   GSAS (Larson and Von Dreele 2004). Diffraction patterns of the phases present are: garnet;

831   clino-pyroxene and new phase (Al-10Å phase).

832   **Fig. 6** Ternary diagram in the system of A (Al_2O_3) - C(CaO) - F(FeO + MgO) with SiO_2 and

833   H_2O in excess. Typical protoliths of metamorphic rocks and possible metamorphic minerals in

834   this system (Bucher and Grapes 2011) are illustrated. The model chemical composition of

835   starting material in this study (lawsonite blueschist) was plotted as red star in ultrabasaltic rock.

836

837   **Fig. 7** Experimentally constrained stability region of the Al - 10Å phase in this study in

838   comparison to stability regions of the Mg - 10Å phase in the hydrated peridotite system in

839   previous studies. Metamorphic facies boundaries experimentally derived for the MORB + H_2O

840   system are taken from Okamoto and Maruyama (1999). Hydrous phases in the starting material

841   and run products are marked with different colors.

842

843 **Fig. 8** Triangular (Ca + Na) - K - H_2O diagram for the twelve-fold coordinated site of reported

844 natural or synthetic mica and 10Å phase. The H_2O content on the twelve-fold coordinated site is

845 calculated assuming (Ca + Na) + K + H_2O = 1. Natural mica from HP/LT metamorphic rocks has

846 higher H_2O content on the twelve-fold coordinated site than that from LP/HT ones.

847

848 **Fig. 9** (K + Na) / (K + Na + H_2O) on the twelve-fold coordinated site versus Si of previously

849 reported natural or synthetic mica and 10Å phase. There are two compositional evolution trends

850 for mica (phlogopite and biotite) at low pressure: Substitution ($Al^{VI} + Al^{IV} = Mg^{VI} + Si^{IV}$). **2.**

851 Substitution ($K^{XII} + Al^{IV} = H_2O^{XII} + Si^{IV}$). For synthetic micas, the experimental pressures are

852 marked in the icon.

853 **Fig. 10** Comparison of the stability regions of the Al-10Å phase (this study) and phengite

854 (Schmidt 1996) in the MORB + H_2O system. Metamorphic facies boundaries experimentally

855 derived for the MORB + H_2O system are taken from Okamoto and Maruyama (1999). The blue

856 line shows the stability region of the Al-10Å phase obtained in this study; the red line shows the

857 stability region of phengite. Typical hot, warm and cold subduction paths (light grey arrows) are

858 taken from (Okamoto and Maruyama 1999) and (Schmidt 1996). The water contents along

859 different subduction paths are marked in the black solid circle (Okamoto and Maruyama, 1999).

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Appendix

865   **Fig. S1** Thin section photomicrographs (plane-polarized light) of the lawsonite blueschist 2S46

866   from North Qilian, NW China, the starting material of this study.

867   **Fig. S2** Selected X-ray powder diffraction (XRD) pattern of the run BE-3-1 (5.5 GPa and

868   700 °C). The standard XRD patterns of all phase possibly present in the run products are shown

869   for comparison. Typical diffraction peaks of the major phases are marked.

870

Fig. 1

Pyrophyllite

T.C. in Al$_2$O$_3$ tube

Steel cap

Mo sheet

Dolomite

Pt Tube

Graphite

Diamond Powder

BN

Sample

5mm

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Legend:

- □ Phengite in synthetic HP/HT eclogite (Schmidt, 1996)
- ⬡ Muscovite in different natural HP schist & eclogite (Li et al, 2012)
- ☆ Paragonite and phengite in HP Franciscan eclogite (Ahn et al, 1985)
- △ Phengite and paragonite in S.W.Tianshan HP/LT Eclogite (Lü et al, 2008)
- ▽ Phengite and paragonite in N. Qilian HP/LT Eclogite (Song et al, 2007)
- ⬭ Muscovite from HP/LT shales from Diablo Range (Torre et al, 1996)
- ○ Al-bearing 10Å in synthetic HP/LT peridotite (Fumagalli & Poli, 2005)
- ⬠ Phlogopite in synthetic HP/LT peridotite (Fumagalli et al, 2009)
- ■ Al-10Å in synthetic HP/LT eclogite (This study)

Na-Mica
Na(+Ca)

H₂O
10Å phase

K
K-Mica

Fig. 9

Fig. 10

Table 1 Bulk composition of starting material in comparison with previous studies

Compond (wt. %)	This study	Error	Average MORB	Altered MORB	Okamoto & Maruyama, 1999
SiO_2	47.76	0.25	50.06	41.63 - 52.30	50.63
TiO_2	0.358	0.02	1.52	0.41 - 2.25	1.74
Al_2O_3	16.07	0.18	15.00	13.20 - 17.95	14.72
Fe_2O_3	12.26	0.16	10.36	1.45 - 5.09	11.09
FeO				3.92 - 11.03	
MnO	0.34	0.02	0.19	0.04 - 0.37	
MgO	5.73	0.12	7.71	5.80 - 13.23	7.61
CaO	4.2	0.10	11.46	2.78 - 13.30	11.13
Na_2O	6.81	0.13	2.52	0.57 - 4.48	2.91
K_2O	0.161	0.01	0.19	0.02 - 8.16	0.13
P_2O_5	0.358	0.02	0.16	0.03 - 0.27	
H_2O	5.6				

Major elements of starting material were obained by XRF analysis; Average major element chemist
from McDonough, 2014; Chemical compositons of altered MORB are taken from Kigiso et al.
reference therein.

Schmidt, 1996
50.59

18.96
1.81
8.24

6.93
10.08
2.9
0.49

try of MORB is
, 1997 and

Table 2 Experimntal conditions and ru

Run NO.	P (GPa)	T (°C)	Time (hours)	Capsule	
Staring material	~ 0.9	~ 350	-	Pt tube	Law(40%)+Gl(38
BE-9	3.5	650	200	Pt tube	law(28%)+C
BE-1-1	4	650	200	Pt tube	Grt(45%
BE-1-2	4	650	150	Sealed Pt	Grt(35%)
BE-11	4	750	120	Pt tube	Grt(47%
BE-7	5	650	150	Pt tube	Grt(35%
BE-3-1	5.5	700	120	Pt tube	Grt(38%
BE-3-2	5.5	700	120	Sealed Pt	Grt(32%
BE-10	5.5	800	80	Pt tube	Grt(45%
BE-5	5.5	900	50	Pt tube	Grt
BE-13	7	700	150	Pt tube	Grt(40%
BE-12	7	750	120	Pt tube	Grt(46%
BE-8	8	700	120	Pt tube	Gr

Note: Grt = garnet; Na-Cpx = Na-clinopyroxene; Law = lawsonite; 10Å = the Al - 10
Coe = coesite; Phase proportion in parentheses are visual determinations from

ın products

Run products (Vol. %)
3%)+Chl(10%)+Ab(10%)+Grt(2%)+ Qz (< 1%)
Gl(30%)+Ep(2%)+Na-Cpx(25%)+Gr(15%)
)+Na-Cpx(42%)+Law(8%)+10Å(5%)
+Na-Cpx(40%)+Law(10%)+10Å(15%)
)+Na-Cpx(47%)+Coe(5%)+10Å(< 1%)
)+Na-Cpx(45%)+10Å(15%)+Law(5%)
)+Na-Cpx(45%)+10Å(15%)+Law(2%)
)+Na-Cpx(40%)+10Å(25%)+Law(3%)
)+Na-Cpx(45%)+Coe(7%)+10Å(3%)
(45%)+Na-Cpx(45%)+Coe(10%)
)+Na-Cpx(50%)+Law(6%)+10Å(4%)
)+Na-Cpx(48%)+Coe(2%)+10Å(2%)
t(45%)+Na-Cpx(48%)+Coe(5%)

Å phase; Gl = glaucophane; Chl = chlorite; Ab = albite;
, the optical microscope images and SEM images

Table 3 Chemical composition of the Al-10Å pahse in the run products in co

	Al-10Å phase (This study)					
Sample	BE-1-1	BE-3-3	BE-1-2	BE-2	BE-3-2	BE-4
SiO_2	57.45(0.54)	55.79(0.44)	57.78(0.08)	56.84(0.38)	60.65(2.40)	58.98(3.21)
TiO_2	0.19(0.08)	0.96(0.16)	0.25(0.01)	0.23(0.02)	0.42(0.01)	0.30(0.03)
Al_2O_3	20.39(0.00)	17.27(0.22)	20.67(0.06)	20.96(1.27)	17.66(1.83)	19.97(2.21)
Cr_2O_3	0.33(0.06)	0.10(0.04)	0.31(0.23)	0.12(0.09)	0.10(0.06)	0.11(0.06)
FeO	4.34(0.63)	5.45(0.84)	4.75(1.13)	4.23(0.36)	7.17(0.03)	5.32(0.61)
MnO	0.02(0.00)	0.02(0.02)	0.04(0.05)	0.03(0.04)	0.06(0.01)	0.03(0.02)
MgO	5.46(0.52)	3.96(0.25)	5.29(0.59)	5.17(0.38)	4.06(0.40)	4.15(0.98)
CaO	0.62(0.79)	0.03(0.02)	0.09(0.04)	0.28(0.24)	0.35(0.23)	1.00(1.61)
Na_2O	0.06(0.01)	0.01(0.02)	0.06(0.02)	0.06(0.01)	0.28(0.35)	0.03(0.06)
K_2O	2.84(0.35)	1.33(0.85)	4.82(0.27)	3.83(1.81)	0.38(0.11)	0.52(0.12)
Totals	91.68(0.71)	84.92(0.36)	94.04(1.06)	91.72(0.58)	91.11(3.36)	90.40(1.62)
Oxygens	11	11	11	11	11	11
Si	3.81(0.03)	3.96(0.02)	3.78(0.01)	3.78(0.05)	4.01(0.01)	3.91(0.15)
Ti	0.01(0.00)	0.05(0.01）	0.01(0.00)	0.01(0.00)	0.02(0.00)	0.01(0.00)
Al	1.59(0.00)	1.44(0.02)	1.60(0.01)	1.64(0.09)	1.38(0.09)	1.56(0.19)
Cr	0.02(0.00)	0.01(0.00)	0.02(0.01)	0.01(0.00)	0.01(0.00)	0.01(0.00)
Fe^{2+}	0.24(0.04)	0.32(0.05)	0.26(0.06)	0.24(0.02)	0.40(0.01)	0.30(0.04)
Mn	0.01(0.00)	0.00(0.00)	0.00(0.00)	0.00(0.00)	0.00(0.00)	0.00(0.00)
Mg	0.54(0.05)	0.42(0.03)	0.52(0.06)	0.51(0.03)	0.40(0.06)	0.41(0.09)
Ca	0.04(0.06)	0.00(0.00)	0.01(0.00)	0.02(0.02)	0.03(0.02)	0.07(0.12)
Na	0.01(0.00)	0.00(0.00)	0.01(0.00)	0.01(0.00)	0.04(0.05)	0.01(0.00)
K	0.24(0.03)	0.12(0.08)	0.40(0.02)	0.33(0.16)	0.03(0.01)	0.04(0.01)
Sum	6.50(0.04)	6.33(0.05)	6.60(0.02)	6.55(0.08)	6.31(0.05)	6.32(0.06)

Notes: Compositons are average of 3-5 measurements on differ

...mparison with previous reported (Al$_2$O$_3$-bearing) 10Å phase

		Al$_2$O$_3$-bearing 10Å phase (Fumagalli et al., 2005)			
BE-7	BE-10	PX5	PX3	Lz5	Lz10
58.13(0.28)	51.91(0.64)	45.00(0.52)	46.29(1.37)	45.94(1.28)	45.72(1.48)
0.15(0.06)	0.65(0.21)	-	-	-	-
19.21(0.05)	22.97(0.48)	10.22(0.81)	10.53(0.68)	10.34(0.54)	9.01(0.79)
0.60(0.66)	0.21(0.04)	-	-	-	-
3.73(1.65)	0.77(0.19)	4.22(0.35)	4.67(0.53)	3.10(0.36)	3.56(0.58)
0.08(0.06)	0.02(0.01)	-	-	-	-
6.08(0.12)	1.76(0.30)	28.69(0.38)	26.68(1.18)	30.11(0.89)	31.28(1.36)
0.27(0.09)	0.23(0.15)	0.12(0.03)	0.72(0.35)	0.31(0.16)	0.20(0.19)
0.13(0.03)	0.15(0.20)	0.82(0.05)	0.34(0.26)	0.06(0.04)	0.07(0.02)
1.43(0.76)	2.1(0.72)	-	-	-	-
89.78(1.20)	80.81(1.18)	89.07	89.23	89.87	89.83
11	11	11	11	11	11
3.88(0.04)	3.79(0.02)	3.000(0.035)	3.120(0.061)	3.030(0.044)	3.010(0.090)
0.01(0.00)	0.04(0.01)	-	-	-	-
1.51(0.01)	1.98(0.05)	0.803(0.045)	0.836(0.052)	0.804(0.040)	0.699(0.061)
0.03(0.03)	0.01(0.00)	-	-	-	-
0.21(0.09)	0.05(0.01)	0.235(0.014)	0.263(0.030)	0.171(0.020)	0.196(0.031)
0.00(0.00)	0.00(0.00)	-	-	-	-
0.61(0.02)	0.19(0.03)	2.850(0.029)	2.680(0.069)	2.960(0.053)	3.070(0.118)
0.02(0.01)	0.02(0.01)	0.008(0.002)	0.052(0.025)	0.022(0.012)	0.014(0.014)
0.02(0.00)	0.02(0.03)	0.106(0.004)	0.044(0.034)	0.008(0.005)	0.008(0.003)
0.12(0.07)	0.20(0.07)	-	-	-	-
6.41(0.00)	6.29(0.03)	7.002	6.995	6.995	6.997

...ent grains with standard deviation in parentheses

Sample	Test-1	Test-1	Test-2	Test-2	BE-1	BE-1	BE-2	BE-2	BE-3
Min	grt-C	grt-R	grt-C	grt-R	grt	grt	grt-C	grt-R	grt-C
SiO_2	36.54	37.71	37.48	37.83	37.27	36.60	36.95	37.77	37.05
TiO_2	0.14	0.28	0.20	0.55	0.18	0.05	0.15	0.41	0.12
Al_2O_3	18.70	20.77	20.88	21.13	19.73	19.71	20.29	20.97	19.65
Cr_2O_3	1.71	0.08	0.08	0.14	0.51	0.44	0.46	0.10	0.37
Fe_2O_3	2.46	2.61	0.81	2.90	1.12	3.04	1.46	0.73	3.03
FeO	11.94	28.35	13.49	20.25	13.72	12.77	14.67	31.00	13.15
MnO	17.28	0.97	16.20	0.73	15.43	14.45	16.38	0.91	16.11
MgO	0.13	5.24	0.23	7.44	0.19	0.18	0.23	4.35	0.20
CaO	11.02	4.46	11.46	7.87	11.72	12.37	9.86	4.07	11.37
Na_2O	0.01	0.20	0.00	0.29	0.01	0.05	0.00	0.11	0.00
K_2O	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.04	0.00
Totals	99.93	100.68	100.83	99.15	99.88	99.66	100.45	100.47	101.06
Oxygens	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00
Si	2.97	2.96	2.98	2.94	3.00	2.96	2.97	2.99	2.96
Ti	0.01	0.02	0.01	0.03	0.01	0.00	0.01	0.02	0.01
Al	1.79	1.92	1.96	1.93	1.87	1.88	1.92	1.95	1.85
Cr	0.11	0.01	0.01	0.01	0.03	0.03	0.03	0.01	0.02
Fe^{3+}	0.15	0.15	0.05	0.17	0.07	0.19	0.09	0.04	0.18
Fe^{2+}	0.81	1.86	0.90	1.31	0.92	0.86	0.99	2.05	0.88
Mn	1.19	0.07	1.09	0.05	1.05	0.99	1.12	0.06	1.09
Mg	0.02	0.61	0.03	0.86	0.02	0.02	0.03	0.51	0.02
Ca	0.96	0.38	0.98	0.65	1.01	1.07	0.85	0.35	0.98
Na	0.00	0.03	0.00	0.04	0.00	0.01	0.00	0.02	0.00
K	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
Sum	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00

Note: -C respects the core

Table 3 Chemical compositions of garnets in the run products

BE-3	BE-4	BE-4	BE-5	BE-5	BE-6	BE-6	BE-7	BE-7	BE-8
grt-R	grt-C	grt-R	grt-C	grt-R	grt-C	grt-R	grt-C	grt-R	grt-C
36.90	36.92	37.40	36.42	37.29	36.66	37.93	37.05	37.46	37.77
1.35	0.19	0.55	0.19	0.45	0.22	0.60	0.24	0.03	0.15
20.39	19.75	19.50	19.67	20.15	19.38	20.59	19.62	19.40	20.39
0.20	1.00	0.21	0.37	0.28	0.39	0.18	0.76	0.23	0.06
2.14	2.96	2.92	2.89	2.85	3.25	1.04	2.37	5.56	0.95
27.49	13.12	27.41	12.79	24.12	12.37	25.21	13.73	14.90	13.50
0.81	14.03	2.31	15.52	3.41	16.92	1.75	14.47	0.26	16.53
5.16	0.21	3.74	0.18	4.19	0.21	5.14	0.24	0.17	0.24
5.61	12.89	6.54	11.60	6.84	11.00	7.15	12.24	22.85	11.23
0.14	0.02	0.09	0.00	0.25	0.00	0.13	0.00	0.01	0.04
0.00	0.00	0.00	0.01	0.01	0.02	0.00	0.00	0.00	0.02
100.19	101.09	100.68	99.64	99.84	100.42	99.71	100.72	100.88	100.88
12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00
2.91	2.94	2.97	2.95	2.96	2.96	2.98	2.97	2.93	3.01
0.08	0.01	0.03	0.01	0.03	0.01	0.04	0.02	0.00	0.01
1.90	1.86	1.82	1.88	1.88	1.84	1.91	1.85	1.79	1.91
0.01	0.06	0.01	0.02	0.02	0.03	0.01	0.05	0.01	0.00
0.13	0.18	0.17	0.18	0.17	0.20	0.06	0.14	0.33	0.06
1.81	0.87	1.82	0.87	1.60	0.83	1.66	0.92	0.98	0.90
0.05	0.95	0.16	1.07	0.23	1.16	0.12	0.98	0.02	1.12
0.61	0.03	0.44	0.02	0.50	0.03	0.60	0.03	0.02	0.03
0.47	1.10	0.56	1.01	0.58	0.95	0.60	1.05	1.92	0.96
0.02	0.00	0.01	0.00	0.04	0.00	0.02	0.00	0.00	0.01
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00

composition; -R respects the rim composition; The formulas of garnet are calculated on the basis of 12 oxyge

BE-8 grt-R	BE-9 grt	BE-10 grt	BE-11 grt-C	BE-11 grt-R	BE-12 grt	BE-12 grt	BE-13 grt-C	BE-13 grt-R
38.80	37.88	37.39	37.49	37.58	37.53	37.38	36.68	38.50
0.28	0.14	0.11	0.31	0.19	0.25	0.19	0.19	0.24
20.58	19.89	19.02	20.15	21.08	19.98	20.78	19.80	21.79
0.01	0.45	2.73	0.43	0.12	0.82	0.14	0.49	0.09
0.00	0.13	0.92	0.00	1.15	0.62	0.30	2.01	0.31
24.33	15.83	14.61	14.04	19.41	13.19	14.86	14.14	27.13
0.88	14.81	14.88	15.45	0.73	16.68	15.38	14.15	0.70
4.10	0.25	0.36	0.18	0.91	0.17	0.67	0.38	5.21
9.99	11.03	11.29	11.65	17.91	11.35	10.15	11.43	5.82
0.22	0.00	0.00	0.00	0.07	0.03	0.05	0.05	0.34
0.02	0.00	0.01	0.00	0.02	0.01	0.00	0.00	0.03
99.21	100.41	101.32	99.70	99.18	100.63	99.90	99.32	100.16
12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00	12.00
3.05	3.04	2.99	3.02	2.97	3.00	3.00	2.97	3.00
0.02	0.01	0.01	0.02	0.01	0.02	0.01	0.01	0.01
1.91	1.88	1.79	1.91	1.97	1.88	1.96	1.89	2.00
0.00	0.03	0.17	0.03	0.01	0.05	0.01	0.03	0.01
0.00	0.01	0.06	0.00	0.07	0.04	0.02	0.12	0.02
1.60	1.06	0.98	0.95	1.29	0.88	1.00	0.96	1.77
0.06	1.01	1.01	1.05	0.05	1.13	1.04	0.97	0.05
0.48	0.03	0.04	0.02	0.11	0.02	0.08	0.05	0.61
0.84	0.95	0.97	1.00	1.52	0.97	0.87	0.99	0.49
0.03	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.05
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00	8.00

:n and 8 cations

Table 2 Experimntal conditions and r

Run NO.	P (GPa)	T (°C)	Time (hours)	Capsule	
Staring material	~ 0.9	~ 350	-	Pt tube	Law(40%)+Gl(38
BE-9	3.5	650	200	Pt tube	law(28%)+C
BE-1-1	4	650	200	Pt tube	Grt(45%
BE-1-2	4	650	150	Sealed Pt	Grt(35%)
BE-11	4	750	120	Pt tube	Grt(47%
BE-7	5	650	150	Pt tube	Grt(35%
BE-3-1	5.5	700	120	Pt tube	Grt(38%
BE-3-2	5.5	700	120	Sealed Pt	Grt(32%
BE-10	5.5	800	80	Pt tube	Grt(45%
BE-5	5.5	900	50	Pt tube	Grt
BE-13	7	700	150	Pt tube	Grt(40%
BE-12	7	750	120	Pt tube	Grt(46%
BE-8	8	700	120	Pt tube	Gr

Note: Grt = garnet; Na-Cpx = Na-clinopyroxene; Law = lawsonite; 10Å = the Al - 10
Coe = coesite; Phase proportion in parentheses are visual determinations from

ın products

Run products (Vol. %)
%)+Chl(10%)+Ab(10%)+Grt(2%)+ Qz (< 1%)
Gl(30%)+Ep(2%)+Na-Cpx(25%)+Gr(15%)
)+Na-Cpx(42%)+Law(8%)+10Å(5%)
+Na-Cpx(40%)+Law(10%)+10Å(15%)
)+Na-Cpx(47%)+Coe(5%)+10Å(< 1%)
)+Na-Cpx(45%)+10Å(15%)+Law(5%)
)+Na-Cpx(45%)+10Å(15%)+Law(2%)
)+Na-Cpx(40%)+10Å(25%)+Law(3%)
)+Na-Cpx(45%)+Coe(7%)+10Å(3%)
(45%)+Na-Cpx(45%)+Coe(10%)
)+Na-Cpx(50%)+Law(6%)+10Å(4%)
)+Na-Cpx(48%)+Coe(2%)+10Å(2%)
t(45%)+Na-Cpx(48%)+Coe(5%)

Å phase; Gl = glaucophane; Chl = chlorite; Ab = albite;
the optical microscope images and SEM images

Table 5 Chemical composition of glaucophane in the run products

Sample	BE-9	BE-9	BE-9
Mineral	gl-C	gl-R	gl
SiO_2	55.9	55.26	55.77
TiO_2	0.15	0.16	0.03
Al_2O_3	9.58	11.3	9.44
Cr_2O_3	0.15	0.13	0.23
Fe_2O_3	0.34	0	0.55
FeO	17	16.48	17.77
MnO	0.46	0.45	0.32
MgO	6.21	5.64	5.98
CaO	0.75	0.85	0.69
Na_2O	7.88	8.15	8.06
K_2O	0	0.01	0.02
Total	98.42	98.44	98.87
Si	23	23	23
Ti	7.94	7.83	7.93
Al	0.02	0.02	0.00
Cr	1.61	1.89	1.58
Fe^{3+}	0.02	0.02	0.03
Fe^{2+}	0.04	0.00	0.06
Mn	2.02	1.95	2.11
Mg	0.06	0.05	0.04
Ca	1.32	1.19	1.27
Na	0.11	0.13	0.11
K	2.17	2.24	2.22
Sum	15.31	15.32	15.37

The formulas of amphibole are calculated on the basis of 23 oxygen

Table 6 Chemical compositions of lawsonite and epidote in the run products

Sample	BE-1-1	BE-1-2	BE-2	BE-3	BE-4	BE-7	BE-9	BE-12	BE-13
Mineral	Law	Law	Law	Law	Law	Law	Law	Law	Law
SiO_2	38.94	39.20	38.35	38.68	38.71	38.22	39.59	39.53	39.70
TiO_2	0.05	0.26	0.09	0.05	0.02	0.07	0.98	0.10	0.07
Al_2O_3	31.09	30.53	30.20	31.06	31.49	31.17	30.87	30.93	31.45
Cr_2O_3	0.14	0.00	0.10	0.05	0.15	0.10	0.03	0.01	0.04
Fe_2O_3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
FeO	0.74	1.18	1.19	0.78	0.76	1.13	0.93	1.41	0.71
MnO	0.01	0.23	0.05	0.02	0.04	0.03	0.03	0.07	0.03
MgO	0.00	0.07	0.05	0.00	0.00	0.00	0.04	0.25	0.04
CaO	17.53	17.00	16.30	17.05	16.78	17.63	17.06	15.31	15.76
Na_2O	0.04	0.05	0.02	0.02	0.01	0.01	0.00	0.09	0.00
K_2O	0.00	0.03	0.01	0.00	0.01	0.00	0.00	0.00	0.01
Total	88.55	88.57	86.36	87.73	87.97	88.38	89.54	87.70	87.90
Si	2.04	2.06	2.06	2.04	2.04	2.02	2.05	2.08	2.08
Ti	0.00	0.01	0.00	0.00	0.00	0.00	0.04	0.00	0.00
Al	1.92	1.89	1.91	1.93	1.95	1.94	1.89	1.92	1.94
Cr	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00
Fe^{3+}	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe^{2+}	0.03	0.05	0.05	0.03	0.03	0.05	0.04	0.06	0.03
Mn	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Mg	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.02	0.00
Ca	0.99	0.96	0.94	0.97	0.95	1.00	0.95	0.86	0.88
Na	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
K	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Sum	4.99	4.99	4.98	4.99	4.98	5.01	4.97	4.96	4.95

BE-9	BE-9
Ep	Ep
37.71	39.11
0.03	0.08
21.37	21.44
0.05	0.05
17.36	15.55
0.55	0.76
0.72	0.5
0	0.12
23.53	23.04
0.03	0.04
0.01	0
101.36	100.69
2.96	3.06
0.00	0.01
1.98	1.98
0.00	0.00
1.03	0.92
0.04	0.05
0.05	0.03
0.00	0.01
1.98	1.93
0.00	0.01
0.00	0.00
8.04	7.99