



# High-pressure phase transitions and subduction behavior of continental crust at pressure–temperature conditions up to the upper part of the lower mantle

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## ABSTRACT

We precisely determined detailed phase relations of upper continental crust (UCC) at 20–28 GPa and 1200–1800 °C across the 660-km discontinuity conditions with a high-pressure multi-anvil apparatus. We used multi-sample chambers packed with both of UCC and pressure marker, and they were kept simultaneously at the same high-pressure and high-temperature conditions in each run. The high-pressure experiments were carried out in pressure and temperature intervals of about 1 GPa and 200 °C, respectively. At 22–25 GPa and 1600–1800 °C, UCC transformed from the assemblage of CaAl<sub>4</sub>Si<sub>2</sub>O<sub>11</sub>-rich phase (CAS)+clinopyroxene+garnet+hollandite+stishovite to that of calcium ferrite+calcium perovskite+hollandite+stishovite via the assemblage of CAS+calcium ferrite+calcium perovskite+garnet+hollandite+stishovite. No CAS was observed at 1200 °C. The textures and grain sizes in the run products suggested that hollandite (II) (monoclinic symmetry) was stable above 24–25 GPa and transformed to hollandite (I) (tetragonal symmetry) during decompression. We calculated the density of UCC at high pressure and high temperature from the mineral proportions which were calculated from the mineral compositions. UCC has a higher density than PREM up to 23.5 GPa in the range of 1200–1800 °C. Above 24 GPa, the density of UCC is lower than that of PREM at 1600–1800 °C, but is almost equal to that at 1400 °C and higher than PREM at temperature below 1400 °C. Therefore, we suggest that the subducted UCC may penetrate the 660-km discontinuity into the lower mantle, when its temperature is lower than 1400 °C at around 660 km depth.

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## 1. Introduction

Subduction of slabs into the deep interior of the earth gives a variety of influences on composition, dynamics and evolution of the mantle. The slab is composed of basaltic crust, harzburgite and slightly depleted pyrolite (e.g. Ringwood, 1994). When the slab subducts into the mantle, it is suggested that continental crust and terrigenous and pelagic sediments may also be subducted into the deep mantle. The mineralogical and petrological studies on ultra-high pressure metamorphic (UHPM) rocks suggest that continental crust subducts to the depth deeper than 200 km, based on discoveries of coesite, clionopyroxene in garnet, and other index minerals derived from continental crust (e.g. Chopin, 1984; Ye et al., 2000). The geochemical studies also assume that the continental crust material subducted into the mantle as deep as the transition zone and the lower mantle and recycled, on the basis of discoveries of isotopic signatures (e.g. <sup>87</sup>Sr/<sup>86</sup>Sr, <sup>143</sup>Nd/<sup>144</sup>Nd, <sup>176</sup>Hf/<sup>177</sup>Hf) of

lavas returned to the earth surfaces at hotspots (e.g. Jackson et al., 2007; Chauvel et al., 2008).

However, it has been generally postulated that it is unlikely that continental crust subducts into the mantle, because it is less dense than pyrolite in the upper part of the mantle. Recently, some mechanisms of subduction of continental crust (tectonic erosion, sediment subduction and arc subduction) to the depth deeper than 300 km have been suggested (Yamamoto et al., 2009a, b; Stern and Scholl, 2010). Results of high-pressure experiments indicated that subducted continental crust has a higher density than the average mantle in the depth range of about 300–660 km by transformations of constituent minerals to denser phases (e.g. coesite to stishovite, K-feldspar to hollandite) (Irifune et al., 1994; Dobrzynetskaia and Green, 2007; Wu et al., 2009). The subduction of continental crust component could play an important role in thermal evolution of earth, because it has significant amounts of long-lived radiogenic elements (<sup>40</sup>K, <sup>232</sup>Th, <sup>235</sup>U and <sup>238</sup>U).

The high-pressure experiments of the upper continental crust (UCC) materials by Irifune et al. (1994) and Wu et al. (2009) have been made in the pressure range from the upper mantle to the

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transition zone using multi-anvil apparatus. They suggested that UCC could be subducted down to around 660-km discontinuity and could stagnate there. However, the phase relations at depth around the 660-km discontinuity are inconsistent between both the studies, and high-pressure experiments have not yet been made at pressures corresponding to deeper part than the 660-km depth. Also, temperature dependence of the phase relations in UCC has not yet been examined. Komabayashi et al. (2009) calculated high-pressure phase relations and zero-pressure density of tonalite–trondhjemite–granite (TTG) system (albite: quartz=1:7 in molar ratio) as a simplified continental crust composition, using the results available from the previous high-pressure experiments. They speculated that the density of subducted TTG may be higher than that of pyrolite down to the core–mantle boundary. However, effects of other minerals, e.g. CaSiO<sub>3</sub>–perovskite and KAlSi<sub>3</sub>O<sub>8</sub>–rich hollandite, stable in UCC in the deep mantle conditions were ignored in their study. In this study, we have examined detailed phase relations in upper continental crust (UCC) composition up to the pressure–temperature conditions of about 800 km depth and have calculated the density of UCC as a function of pressure and temperature to compare with that of Preliminary Reference Earth Model (PREM) (Dziewonski and Anderson, 1981). Based on the results, we have discussed possible subduction of the continental crust material into the lower mantle.

## 2. Experimental methods

### 2.1. Starting materials

Reagent-grade chemicals were used to prepare all the starting materials. Composition of upper continental crust (UCC) by Taylor and McLennan (1985) shown in Table 1 was basically adopted after excluding very small amounts of MnO and P<sub>2</sub>O<sub>5</sub>. The starting material of UCC was prepared by mixing the following oxides and silicates: SiO<sub>2</sub> (68.0), TiO<sub>2</sub> (0.6), Mg<sub>2</sub>SiO<sub>4</sub> (2.6), Fe<sub>2</sub>SiO<sub>4</sub> (3.0), NaAlSiO<sub>4</sub> (11.9), CaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub> (4.8), KAlSiO<sub>4</sub> (6.8) and CaSiO<sub>3</sub> (2.3), where numbers in parentheses are contents in mol%. Fine-grained SiO<sub>2</sub> prepared by heating silicic acid (SiO<sub>2</sub>·12.3 wt% H<sub>2</sub>O) at 1000 °C for 3 h and amorphous TiO<sub>2</sub> were used to promote reaction at high pressure and high temperature. Mg<sub>2</sub>SiO<sub>4</sub> forsterite was synthesized from a mixture of MgO and SiO<sub>2</sub> with a 2:1 M ratio by heating at 1500 °C for 75 h. Fe<sub>2</sub>SiO<sub>4</sub> fayalite was synthesized from

a mixture of Fe<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> with a 1:1 M ratio by heating at 1180 °C for 24 h in a controlled oxygen fugacity using a mixture of H<sub>2</sub>, CO<sub>2</sub> and Ar with volume ratios of 1:1:2. NaAlSiO<sub>4</sub> carnegieite was synthesized from a mixture of Na<sub>2</sub>CO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> with a 1.05:1:2 M ratios by heating at 1300 °C for 34 h. The addition of 5 mol% Na<sub>2</sub>CO<sub>3</sub> was based on taking into account Na-loss during the heating process (Kojitani et al., 2011). CaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub> anorthite was synthesized from a mixture of CaCO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> with 1:1:2 M ratios by heating at 1450 °C for 128 h. KAlSiO<sub>4</sub> kalsilite was synthesized from a mixture of K<sub>2</sub>CO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> with a 1.05:1:2 M ratio by heating at 1050 °C for 40 h. The addition of 5 mol% K<sub>2</sub>CO<sub>3</sub> was by the same reason as in the synthesis of NaAlSiO<sub>4</sub>. CaSiO<sub>3</sub> pseudowollastonite was synthesized from a mixture of CaCO<sub>3</sub> and SiO<sub>2</sub> with a 1:1 M ratio by heating at 1450 °C for 131 h. NaAlSi<sub>2</sub>O<sub>6</sub> jadeite was also used as a starting material in some runs. It was synthesized from a mixture of Na<sub>2</sub>CO<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> with 1.05:1:4 M ratios. This mixture was heated at 600 °C for 6 h, and NaAlSi<sub>2</sub>O<sub>6</sub> jadeite was synthesized from the heated mixture at 12 GPa and 1600 °C for 1 h. Identification of these synthetic compounds and confirmation of single-phase materials were made using a powder X-ray diffractometer and a scanning electron microscope with an energy-dispersive X-ray spectrometer (SEM-EDS). The oxides and silicates thus synthesized were thoroughly mixed in an alumina mortar, and used as the starting material of UCC. The composition of the starting material analyzed by the SEM-EDS is shown in Table 1 in comparison with UCC of Taylor and McLennan (1985).

### 2.2. High-pressure experiments with the multi-sample cell technique

High-pressure experiments were made at 20.6–28.0 GPa and 1200–1800 °C with a Kawai-type 6–8 multianvil high-pressure apparatus at Gakushuin University. We adopted the multi-sample cell technique described in our previous work (Ishii et al., 2011). The high-pressure cell assembly with the sample cells used in this study is shown in Fig. S-1 (Supplementary material). Tungsten carbide anvils of truncated edge length of 2.5 mm were used in combination with a Cr<sub>2</sub>O<sub>3</sub>-doped semi-sintered MgO octahedron of 7 mm on edge as the pressure medium. The magnesia octahedron was dried at 100 °C over-night in a furnace before putting a Re capsule in it. The starting material of UCC and the pressure marker (one of Mg<sub>2</sub>SiO<sub>4</sub>, MgSiO<sub>3</sub> and MgO+Al<sub>2</sub>O<sub>3</sub>) were packed separately into two holes of 0.4- and 0.2-mm in diameter, respectively, of the Re capsule which was 1.0 mm in diameter and 0.7 mm in thickness. In a few runs, we also used the Re capsule with three holes (0.2-mm in diameter) to pack UCC, NaAlSi<sub>2</sub>O<sub>6</sub> jadeite and MgSiO<sub>3</sub>. Two Re discs of 1.0 mm in diameter and 0.1 mm in thickness were put on both sides of the Re capsule. A tubular rhenium furnace of 1.5 mm in diameter and 5.7 mm in length was put in the central part of the magnesia octahedron. The Re capsule containing the sample and the pressure marker was placed in the central part of the heater. A boron nitride sleeve was inserted between the Re capsule+discs and the tubular rhenium heater for electrical insulation. A LaCrO<sub>3</sub> sleeve was placed outside of the rhenium furnace for thermal insulation. Also, LaCrO<sub>3</sub> end-plugs were put at both ends of the heater. The run temperature was measured at the central part of outer surface of the furnace with a Pt/Pt–13%Rh thermocouple of 0.1 mm in diameter. No correction was made on pressure effect of the thermocouple emf. In each run, pressure was raised to a targeted pressure at almost constant rate during about 2–4 h, and then temperature was raised to 1200–1800 °C at a rate of about 100 °C/min. The sample assembly was kept for 2–3 h at the pressure–temperature conditions, then quenched, slowly decompressed, and recovered to ambient conditions. The recovered Re capsule holding the sample and the pressure marker was mounted on a slide glass plate with epoxy resin, and polished

**Table 1**  
Chemical compositions of starting materials for high-pressure experiments of upper continental crust.

Oxide(wt%)	This study <sup>a</sup>	Irifune et al. (1994)	Wu et al. (2009)	UCC <sup>b</sup>
SiO <sub>2</sub>	65.34(0.63)	66.20	69.03	66.00
TiO <sub>2</sub>	0.45(0.15)	0.60	0.64	0.50
Al <sub>2</sub> O <sub>3</sub>	17.39(0.33)	15.90	14.81	15.20
FeO <sup>c</sup>	3.99(0.27)	4.60 <sup>d</sup>	3.26	4.50
MnO	–	–	0.08	0.08
MgO	1.96(0.17)	1.90	1.08	2.20
CaO	3.77(0.17)	4.80	3.16	4.20
Na <sub>2</sub> O	3.46(0.18)	2.70	4.68	3.90
K <sub>2</sub> O	3.53(0.14)	3.30	2.11	3.40
P <sub>2</sub> O <sub>5</sub>	–	–	0.14	0.16
Total	99.89	100.00	98.99	100.14

<sup>a</sup> Composition of starting material analyzed with SEM-EDS and used in this study. Values in parentheses represent two standard deviations of the mean.

<sup>b</sup> Upper continental crust (UCC) composition from Taylor and McLennan (1985).

<sup>c</sup> Total Fe was calculated as FeO.

<sup>d</sup> FeO was replaced by CoO in the study by Irifune et al. (1994).

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