



Research paper

Structural changes of potassium-saturated smectite at high pressures and high temperatures: Application for subduction zones



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ABSTRACT

In a subduction zone, water-rich pelagic sediments overlying the subducting oceanic plate are accumulated, forming an accretionary wedge. A portion of these sediments, primarily smectites, can subduct and lose the water present in their pores, but the mechanism of the clay structural transformation and its composition are not well-defined. Smectite clay minerals can remain stable, carrying water at pressures corresponding to the upper mantle and thus contributing to the occurrence of melting and metasomatic processes in the mantle. In this work, we performed several high-pressure and high-temperature experiments to verify the structural behavior of a potassium-saturated smectite under various pressure and temperature conditions: (1) atmospheric pressure at two different temperatures (100 °C to 700 °C); (2) room temperature (25 °C) and pressure up to 11.5 GPa – diamond anvil cell (DAC) experiments; (3) under pressures of 2.5 GPa and 4.0 GPa and temperatures from 200 °C to 700 °C. Scanning electron microscopy (SEM), X-ray diffraction (XRD), transmission electron microscopy (TEM) and infrared spectroscopy (FTIR) analysis on all samples studied suggest that, under elevated pressures (2.5 GPa, which corresponds to a depth of approximately 75 km, and 4.0 GPa, equivalent to a depth of approximately 120 km), smectite remains stable until 200 °C. With increasing temperature, the initial structure partially loses its interlayer H₂O and transforms into a mixed layer of illite/smectite, which is maintained until 400 °C under 2.5 GPa and until 300 °C under 4.0 GPa. Beyond these conditions, with increasing temperature, the smectite loses all of its interlayer H₂O and transforms into muscovite. Additionally, at room temperature, smectite is stable under nearly 12 GPa. These results contribute significantly to the understanding of how pelagic sediment dehydration and transformation occur in the subduction process, as well as the behavior of smectite under the influence of increasing pressure and temperature.

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1. Introduction

In a subduction zone, the presence of water in the mantle is essential for initiating a partial melting of the overlying mantle wedge. Water can be loaded up to these depths through the subducting oceanic crust, where the amphibolite-eclogite transformation releases H₂O at a depth of ca. 60 km (Maruyama and Okamoto, 2007). Furthermore, the transport of hydrous phases to depths is another important way to trigger partial melting (Harlow and Davies, 2004). Hemipelagic sediments of the oceanic crust can be subducted to great depths, where they

dehydrate and decrease the melting point of the surrounding rocks. Thus, hemipelagic sediments play an important role in the occurrence of partial melting of the overlying mantle wedge.

Smectite clay minerals, one of the most common types of minerals present in the hemipelagic sediments of a subducting oceanic crust, carry water to great depths in a subduction zone if the geothermal gradient is low enough, as can be observed in the subduction zones formed in an old-to-moderately-old plate (50–100 Ma) (Peacock, 1991). If the temperature is above 650 °C at depths of 30–90 km, as in a young oceanic plate, the subducting oceanic crust reaches the solidus temperature condition, and the released water dissolves in the melt. Although a high convergent rate is recognized in some young slabs, the age of the subducting slabs dominantly controls the thermal structure of a subduction zone (Maruyama and Okamoto, 2007). Therefore, the age of the subducting slabs also controls the seismogenic zone, partitioned between the smectite composition (aseismic zone) and the illite

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composition (seismic zone – 100–150 °C/5–15 km in depth). Subduction zone faults generate earthquakes, and the transformation from smectite to illite controls the seismogenic zone (Vrolijk, 1990; Hyndman et al., 1997).

In this context, smectite stands out as one of the most important minerals responsible for enriching the lithospheric mantle with water and incompatible elements such as potassium, rubidium and strontium, when its structure is destabilized (Meunier, 2005). By increasing the pressure and the temperature, smectite loses its interlayer water and at the same time transforms into a mixed-layer illite–smectite. In the first stage of this transformation, the illite forms randomly ordered mixed layers with the smectite, but with the progress of the reaction, the stacking of these sequences becomes ordered, and the illite content increases (Inoue et al., 1988). The reaction consumes potassium, which implies an input of this element beyond the insufficient amount present in the original smectite composition. This element may be derived from the dissolution of a detrital mineral (K-feldspar or mica) or from surrounding saline solutions. With continuous increasing of the pressure and the temperature (above 210 °C and 8–10 kbar of pressure), the ordered illite clay mineral transforms into muscovite mica (Meunier, 2005). A further increase in the pressure and the temperature could allow melting of these rocks and sediments and an increase in the incompatible (e.g., Sr, K, Rb, Ba and Th) element content in the mantle wedge, related to the subduction context. Although all of these reactions are known in the subduction-related zone, the mechanism of clay structural transformation and its evolving composition is not well established.

In this work, we studied the stability of a potassic smectite clay mineral, obtained by cation exchange from a primary calcium smectite, through high-pressure and high-temperature experiments at 1 atm, 2.5 GPa and 4.0 GPa pressures under various temperatures. We also studied the stability of the same smectite at room temperature and under pressures up to nearly 12 GPa.

The amount of H₂O retained in the smectite interlayers (>20% H₂O) is much larger than that in micas (~4.7%), kaolinite (~13.8%) or other hydrous minerals (Huang et al., 1994).

The stability of this clay mineral and its transformation into muscovite mica under high pressure and high temperature in a subduction-related zone is important for understanding the role of water in such processes as metasomatism and/or partial melting events within the upper mantle.

2. Experimental methods

2.1. Starting material

The smectite samples were obtained by gravimetric separation of bentonite rocks from the Rio do Rasto Formation in the city of Melo, Uruguay. The bentonite rock is composed predominantly of calcium dioctahedral smectite along with traces of quartz and feldspar (Albarnaz et al., 2009). The smectite was separated from the bentonite in size fraction minerals <2 μm by the sedimentation method according to Stoke's Law. This method involves the direct measurement of the distribution of fractions as a function of time for these particles, decanted in a vertical cell (beaker) with a convenient liquid (distilled water). The chemical formula of the studied smectite was approximately calculated by Calarge et al. (2003) using ICP-MS analyses: [Si_{3.87}Al_{0.13}] O₁₀ (Al_{1.43}Fe³⁺_{0.08}Mg_{0.53}Ti_{0.01}) (OH)₂ K⁺_{0.01}Ca²⁺_{0.23}.

Following the separation of the <2 μm fraction, a KCl (1 mol/L) solution was added to replace calcium by potassium in the structure of the smectite. The sample was rinsed as many times as necessary with de-ionized water to remove chlorides, followed by a test with AgNO₃ to confirm the absence of chlorine (Greene-Kelly, 1953). All experiments in this work were performed on the potassic smectite that will be referred to in this paper as K-Sm.

2.2. High pressure experiments

2.2.1. Diamond anvil cell experiments

High-pressure experiments at room temperature were performed using a Piermarini-Block diamond anvil cell (DAC). This instrument uses two diamonds as two anvils, which are pressed against each other, generating very high pressures between them. A metallic gasket with a circular hole (diameter of 250 μm) and the sample are placed between the two anvils, along with a small ruby crystal. The ruby crystal is used to measure the applied pressure because its fluorescent spectrum changes linearly with pressure (Jayaraman, 1983). This technique has the advantage of allowing in-situ measurements, such as FTIR, because diamonds provide optical access to the sample. However, in our laboratory, high-pressure conditions cannot be applied with temperature, and therefore, all experiments were performed at room temperature.

2.2.2. Toroidal chambers experiments

High-pressure experiments under high-temperature conditions were performed in a hydraulic press of 1000 tons at the Instituto de Física, UFRGS. This press has toroidal profile chambers that allow a relatively homogeneous distribution of pressure inside the chamber and reach pressures up to 7.7 GPa. The experiments were performed at pressures of 2.5 and 4.0 GPa at various temperatures (up to 700 °C). The pressure calibration was performed with Yb (4.0 GPa) and Bi (2.5 GPa and 4.0 GPa) because there are phase transitions in these materials under those pressure conditions. The pressure was considered accurate to ± 0.5 GPa. A temperature calibration was performed with Pt-PtRh13% thermocouples.

The reaction cell consists of a graphite cylinder and two graphite disks, two calcined pyrophyllite disks and a capsule of hexagonal boron nitride (hBN). The sample is placed inside the hBN capsule, which is placed into the graphite cylinder and between the pyrophyllite disks. Two graphite disks are positioned at the extremes of the graphite cylinder. This entire configuration is placed into a gasket of ceramic material with a toroidal, or ring-shaped, profile.

Pressure is applied to the sample gradually until the desired pressure is reached. Subsequently, an elevated temperature was applied by an electric current. Tests of the run time were performed previously to ensure thermodynamic equilibrium. In runs conducted for up to 3 h, we observed results that were not reproducible and had no grain identification, which suggest a lack of equilibrium. However, in runs conducted for a longer time, such as for 8 h, we observed reproducible results and grain formation, which suggest that the time was sufficient for the necessary kinetic reactions to occur. Longer run times reproduced the same results as the 8 hour runs. In this way, all experiments were conducted during an ideal time of 8 h to ensure smectite equilibrium reactions. After this time, the electric current is turned off, which causes a quench in the sample, to preserve the conditions of the experiment. All experiments in the toroidal chamber were performed twice to provide enough sample material to be analyzed and also to verify the reproducibility of the experiments.

2.3. Analytical techniques

X-ray diffraction analyses (XRD) were performed on the starting material before and after cation exchange, as well as on all run products. The mineral phases of the samples were examined in powder form using a Siemens D-500 Diffractometer. Data were collected with CuKα radiation, an angular rate of 2° to 80° (2θ), a step of 0.05° and a counting time of 2 s per step for all samples. Furthermore, to check the existence of the illite/smectite (I/S) structure in certain samples, a saturation process with ethylene glycol (Bradley, 1945) and diffraction analysis of the results was performed.

Infrared spectra by transmission and reflectance were performed on the samples and were obtained on a FTIR spectrometer model MB Bomem 100 in the spectral range of 350 cm⁻¹ to 4000 cm⁻¹.

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