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Effect of dehydration on the electrical conductivity of phyllite at high temperatures and pressures

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Abstract The electrical conductivity of phyllite (measured in situ at 0.5-2.5 GPa and 773-1173 K) increases with increasing temperature, satisfying an Arrhenius relation. Dehydration of phyllite at 973-1173 K enormously enhances its electrical conductivity, and the activation enthalpy (0.64-0.81 eV) remains almost constant before and after dehydration. The inflection point of the relationship between electrical conductivity and temperature is used to determine the dehydration temperature (T_d) at each considered pressure (P), leading to the following relationship: $T_d = 1181 - 100P$. The derived relation implies that the dehydration depths of hot and cold subduction zones are ~70 and ~129 km respectively, which are both close to the depths of arc magma source regions, thereby indicating that the dehydration of pelite significantly influences the generation of melt in subduction zones.

Keywords High temperature and high pressure · Electrical conductivity · Phyllite · Dehydration · Subduction zone

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Introduction

Understanding the material composition and thermodynamic state of the Earth's interior requires in situ measurements of the electrical conductivity of minerals and rocks at high temperatures and pressures, and comparison with magnetotelluric (MT) and geomagnetic depth sounding (GDS) results. Previous works have measured the electrical conductivities of many minerals and rocks in the crust and upper mantle, and suggested interpretations of high-conductivity layers (HCLs) in the Earth's interior (Zhu et al. 2001; Huang et al. 2005; Yang 2012; Cherevatova et al. 2014; Dai and Karato 2014a; Hu et al. 2014; Rao et al. 2014). However, little research has considered the electrical properties of pelite, a clastic rock in subduction zones, meaning that previous studies on the physical and chemical properties of subduction zones remain incomplete. Therefore, previous interpretations of HCLs should be reconsidered.

Pelites come in different forms (e.g., mudstone, clay rock, shale, and slate) and can transform to phyllite during metamorphism. Phyllite contains abundant sericite and chlorite, which are important hydrous minerals in subduction zones (Tatsumi and Eggins 1995; Cabral et al. 2012; Micheuz et al. 2015). As the subducting slab is heated and compressed, the pelites undergo a series of metamorphic changes (Wyllie 1982). Previous experimental petrological studies have shown that the dehydration of pelite at high temperatures and pressures accompanies the formation of new mineralogical phases (Tatsumi and Eggins 1995; Zhao et al. 2015). The dehydration temperature of minerals and rocks is generally determined using high-pressure differential thermal analysis and the inflection point of the Arrhenius relation between electrical conductivity and temperature (Song et al. 1996; Li et al. 2005). Previous works have demonstrated that dehydration can greatly enhance the electrical conductivity of minerals and rocks at

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high temperatures and pressures (Song et al. 1996; Hicks and Secco 1997; Fuji-Ta et al. 2007a). Therefore, the dehydration temperature can be directly determined from the measurable increase of electrical conductivity. This temperature is a crucial parameter to determine the dehydration depth of pelite in a subduction zone.

This work reports the electrical conductivity of phyllite measured in situ at 0.5–2.5 GPa, 773–1173 K, and 10^{-1} – 10^{6} Hz. At each pressure, the inflection point of the Arrhenius relation between electrical conductivity and temperature was used to determine the dehydration temperature of the phyllite, and the conduction mechanisms at different stages were explored in detail. Furthermore, a relationship between dehydration temperature and pressure was successfully established, and the dehydration depths of phyllite in hot and cold subduction zones were determined according to the dehydration conditions and the geothermal gradient in each case. Because the dehydration depth range of pelite is similar to that of arc magma source regions, we suggest that the dehydration of pelite significantly influences the generation of melt in subduction zones. The high electrical conductivity observed after dehydration can also account for the HCLs of certain regions in subduction zones.

Experimental procedures

Sample preparation

Fresh, brown phyllite samples were collected from the Fangshan District, Beijing, China. Their mineralogical composition was determined using optical microscopy and scanning electron microscopy (SEM) at the State Key Laboratory of Ore Deposit Geochemistry, Institute of Geochemistry, Chinese Academy of Sciences (CAS), Guiyang, China. By volume, the main minerals in the phyllite were chlorite (50%), sericite (30%), and quartz (14%), with minor albite (6%).

The phyllite samples were ultrasonically cleaned in a mixture of acetone and ethanol, and ground to powder (<200 mesh) in an agate mortar before being dried in a furnace at 473 K for 3 h. The powder was loaded into a copper capsule with a 0.025-mm thick Ni-foil liner, which prevented interdiffusion between the sample and the copper capsule during hot-press sintering. The height of the copper capsule was 18 mm, and its inner and outer diameters were 8.0 mm and 8.5 mm, respectively. To avoid the influence of pores and micro-cracks on the electrical conductivity results, the powdered samples were sintered for 3 h in a multi-anvil highpressure apparatus at 2.0 GPa and 673 K, similar to the approach of Li et al. (2005). Then, the hydrothermally annealed samples were cut and polished into cylinders with diameter and height of 6 mm. Finally, the samples were baked at 473 K for 8 h to eliminate any absorbed water that might have affected the electrical conductivity measurements.

Impedance measurements

Measurements were carried out using a YJ-3000 t multi-anvil press and a Solartron-1260 Impedance/Gain-phase analyzer at the Key Laboratory of High-Temperature and High-Pressure Study of the Earth's Interior, Institute of Geochemistry, Chinese Academy of Sciences (CAS), Guiyang, China. Details of the measurement procedures are given by Dai et al. (2009) and Hu et al. (2013). The multi-anvil press can achieve pressures of up to 6.0 GPa, and six cubic WC anvils were assembled and compressed. Temperatures of up to 2273 K can be achieved with a lanthanum chromate heater, but the temperature range considered here was attained using a stainless steel heater (~298-1573 K). Errors in the electrical conductivity measurements were estimated to be less than 5%, and the principal sources of error were dimensional changes in the sample (<8%), experimental temperature error (<10 K), and experimental pressure error (<0.1 GPa).

The experimental assemblage for the electrical conductivity measurements is shown in Fig. 1. Pyrophyllite cubes of 32.5 mm edge length were used as the pressure medium, and three-layer stainless steel sheets (total thickness 0.5 mm) arranged around the Al₂O₃ insulator were applied as the heater. To shield against external electromagnetic and spurious signal interference, a layer of Ni foil (thickness 0.025 mm) was installed between the Al₂O₃ insulation and the MgO sleeve. The sample was loaded into the MgO tube, and two Ni disks (diameter 6 mm, thickness 0.5 mm) on the top and bottom of sample comprised the parallel-plate electrode. To prevent free water from influencing the impedance spectra measurements, all components of the apparatus (pyrophyllite, ceramic tubes, Al₂O₃, and MgO sleeves) were heated at 1073 K for 8 h in a muffle furnace. Temperatures were measured with a NiCr-NiAl thermocouple, and the pressure and temperature errors were ± 0.1 GPa and ± 10 K, respectively.

During the experiments, pressure was slowly increased at a rate of 1.0 GPa/h to the desired value, then temperature increased at 100 K/h to the designated values. Once the temperature reached a stable value, the impedance spectra were measured with an applied voltage of 1 V using a frequency range of 10^{-1} to 10^{6} Hz. Moisture can potentially influence the validity and precision of electrical conductivity measurements for minerals and rocks at high temperatures and pressures (Manthilake et al. 2009; Shatskiy et al. 2009; Yoshino et al. 2010; Dai et al. 2015; Hu et al. 2015), and its effects were mitigated here by maintaining the experimental assembly for at least 3 h at high temperature (773 K) and high pressure (0.5, 1.5, and 2.5 GPa) during the impedance spectroscopy measurements. When two successive impedance spectra were almost completely overlapping, we started to collect each data

measurements



point at intervals of 50 K. This ensured that all the conductivity results were obtained at a steady state condition. Furthermore, two heating and cooling cycles were conducted to confirm further the elimination of any moisture effect during the measurements. Two representative heating and cooling cycles at 1.5 GPa are displayed in Fig. 2. Overall, the influence of moisture on the electrical conductivity of phyllite was negligible. Impedance spectra of the phyllite samples were collected at 0.5-2.5 GPa and 773-1173 K.



Fig. 2 Logarithm of electrical conductivity versus reciprocal temperature for the sample during different heating/cooling cycles at 1.5 GPa

Results

Typical Nyquist and Bode diagrams measured at 1.5 GPa and 773-1173 K are shown in Figs. 3 and 4, respectively. The real part (Z'), imaginary part (Z"), modulus (|Z|), and phase angle (θ) of the impedance spectra follow the relationships $Z' = |Z|\cos\theta$ and $Z'' = |Z|\sin\theta$ (Nover et al. 1992; Huebner and Dillenburg 1995). The impedance spectra in Fig. 3 each include an integrated semicircle and a straight line at 773-973 K, but only show a complete semicircle at 1023-1173 K. Generally, the semicircle occurring at high frequency represents the impedance of the grain interior, and the tail at low frequency represents the impedance of the grain boundary or the sample-electrode interface. Furthermore, the complete arc representing grain-boundary conduction occurs at the intermediate frequency range of 0.01-200 Hz (Roberts and Tyburczy 1991), whereas the electrode response can appear separately or as a straight line, which is characteristic of diffusion at the sample-electrode interface (MacDonald 1992; Hu et al. 2015). Therefore, the additional linear section observed here reflects the effect of the electrodes, and the bulk sample resistance can be determined by fitting the first semicircular arc. The equivalent circuit shown in Fig. 3 has resistor (R) and constant-phase element (CPE) components for the bulk impedance for the sample (R_s and CPE_s, respectively) and for the sample-electrode interface (R_E and CPE_E , respectively).

The fitting error is less than 5%. The electrical conductivity of the sample was calculated as follows:

$$\sigma = L/SR,\tag{1}$$

Fig. 3 Nyquist plot of the complex impedance of phyllite at 1.5 GPa and 773–1173 K



where *L* is the height of the sample (m), *S* is the cross-sectional area of the electrodes (m²), *R* is the fitting resistance (Ω), and σ is the electrical conductivity of the sample (S/m).

The logarithmic electrical conductivity of phyllite is plotted against the reciprocal temperature in Fig. 5 for pressures of 0.5-2.5 GPa and temperatures of 773-1173 K. The

Fig. 4 Bode plot of phyllite at 1.5 GPa and 773–1173 K





Fig. 5 Relationship between electrical conductivity of phyllite and reciprocal temperature at 0.5–2.5 GPa and 773–1173 K

relationship between electrical conductivity and temperature before dehydration fits the Arrhenius formula:

$$\sigma = \sigma_0 \exp(-\Delta H/kT) \tag{2}$$

where σ_0 is a pre-exponential factor (K·S/m), ΔH is the activation enthalpy (eV), *k* is the Boltzmann constant (eV/K), and *T* is absolute temperature (K). The fitting parameters for the bulk sample conductivity are listed in Table 1.

The progression in electrical conductivity with time during dehydration is shown in Fig. 6 for a sample held at 2.5 GPa and 973 K. The electrical conductivity increases sharply in the initial 40 min and then gradually levels off, reaching a steady state after 180 min. The attainment of constant electrical conductivity indicates the completion of dehydration. The mineral associations of the phyllite sample before and after dehydration were accurately determined, and the results of optical microscopy and SEM are shown in Figs. 7 and 8 and listed in Table 2. At 0.5 GPa, the dehydration temperature of the sample was 1173 K, and partial melting occurred during dehydration. After dehydration, the electrical conductivity showed good repeatability during cooling and heating. At 1.5 and 2.5 GPa, the dehydration temperatures were 1023

 Table 1
 Fitted parameters for the electrical conductivity of phyllite

 before dehydration
 Fitted parameters

Run no.	P (GPa)	$T(\mathbf{K})$	$\text{Log } \sigma_0 (\text{S/m})$	$\Delta H ({\rm eV})$	R ²
DS2	0.5	773–1073	-0.21 ± 0.01	0.69 ± 0.01	0.9969
DS3	1.5	773–973	$\textbf{-0.10} \pm 0.01$	0.64 ± 0.01	0.9991
DS4	2.5	773–923	1.35 ± 0.04	0.81 ± 0.01	0.9989



Fig. 6 Relationship between electrical conductivity of phyllite and time at 2.5 GPa and 973 K

and 973 K, respectively. New minerals (garnet, biotite, and kyanite) appeared in the experimental products during dehydration, and no melt appeared (Figs. 7 and 8). The electrical conductivity slowly increased upon heating after dehydration, and showed good repeatability during cooling and heating. While the electrical conductivity of the sample increased with increasing pressure, the effect was relatively weak before dehydration. However, the electrical conductivity of the sample at 0.5 GPa was more than two orders of magnitude lower than that at 1.5 or 2.5 GPa after dehydration.

Discussion

Comparison with published data

The volumetrically abundant chlorite and sericite appear to dominate the electrical conductivity of the phyllite samples. Conductivities were $\sim 10^{-3}$ S/m before dehydration. At 0.5 GPa, partial melting occurred during dehydration, with the melt occupying a volume fraction of about 0.5%. At 1.5 and 2.5 GPa, new minerals (garnet, biotite, and kyanite) appeared with no melt. Partial melting generally enhances the electrical conductivity of silicate minerals and rocks (e.g., Schilling et al. 1997; Maumus et al. 2005), but here the electrical conductivity of phyllite after partial melting at 0.5 GPa was 2–2.5 orders of magnitude lower than that with no melt at 1.5 or 2.5 GPa. This result might reflect the occurrence of abundant biotite (20%) in the dehydration products at higher pressures. Li et al. (2016) confirmed that the electrical conductivity of phlogopite is much higher than that of peridotite, serpentine,



Fig. 7 Photomicrographs of (a) the original phyllite sample and the experimental products at (b) 0.5 GPa, (c) 1.5 GPa, and (d) 2.5 GPa. Qtz = quartz, Chl = chlorite, Ser = sericite, Bt = biotite, Alm = almandine, Ky = kyanite, Ab = albite

talc, or pyrophyllite; therefore, a small quantity of phlogopite can significantly enhance the electrical conductivity of silicate rocks. Given that biotite and phlogopite belong to the same group of mica minerals, having similar crystal textures and



Fig. 8 Electron backscattered images of the recovered samples after EC measurements at (a) 0.5 GPa, (b) 1.5 GPa, and (c) 2.5 GPa. Qtz = quartz, Ser = sericite, Bt = biotite, Alm = almandine

Table 2 Mineralogical assemblage of original sample and experimental products		P (GPa)	<i>T</i> (K)	No.	Mineral associations
and experimental products	Before dehydration	/	/	DS1	Chl (50%) + Ser (30%) + Qtz (14%) + Ab (6%)
		0.5	773–1173	DS2	Qtz (70%) + Ser (25%) + Ab (4.5%) + Melt (0.5%)
	After dehydration	1.5	773–1173	DS3	Qtz (40%) + Alm (20%) + Bt (20%) + Ser (10%) + Ab (6%) + Ky (4%)
		2.5	773–1123	DS4	Qtz (40%) + Alm (20%) + Bt (20%) + Ser (10%) + Ab (6%) + Ky (4%)

chemical components, their electrical conductivities might be similar. The biotite observed here might therefore have a more significant effect on the electrical conductivity of the sample than the partial melting observed at lower pressure. The abundant biotite (20%) in the dehydration products show good interconnectivity (Fig. 8). Comparison with the results of Manthilake et al. (2015) shows that aqueous fluid is less conductive than the dehydration products of phyllite. The interconnected biotites were thus possibly responsible for the high electrical conductivity of the dehydration products of phyllite at 1.5 and 2.5 GPa. On the other hand, SEM analyses revealed dehydration-induced partial melting in the phyllite at 0.5 GPa, which can reasonably explain the about one order of magnitude enhancement of electrical conductivity seen at 1173 K. However, the volume fraction of melt was relatively low, only $\sim 0.5\%$, and therefore the electrical conductivity of phyllite at 0.5 GPa was lower than that at 1.5 or 2.5 GPa. The presence of sericite in the dehydration products made the partial dehydration of phyllite obvious. We also estimated the mass fraction of fluid released to the sample during dehydration at each of the three tested pressures to be ~ 4.0 wt%.

The maximum electrical conductivity of chlorite is 10^{-4} S/ m before dehydration (Manthilake et al. 2016), slightly lower than that of the phyllite considered here. This discrepancy might be due to the influence of sericite in the phyllite. Similar to other silicate minerals and rocks, the electrical conductivity of phyllite shows a weak pressure dependence (Dai et al. 2008, 2012, 2013, 2014, 2015; Yang and McCammon 2012; Manthilake et al. 2016), increasing slightly under compression before dehydration, like chlorite (Manthilake et al. 2016). After dehydration, the electrical conductivity of phyllite increases by two orders of magnitude, thereby confirming the significant influence of the dehydration of hydrous minerals and rocks (Zhu et al. 1999; Fuji-ta et al. 2007a; Manthilake et al. 2016). Although the dehydration temperature of chlorite is similar to that of phyllite, the dehydrated mineral assemblages of chlorite and phyllite are different. Our phyllite was dominated by garnet, biotite, and kyanite after dehydration (Table 2); however, the hydrous chlorite reported by Manthilake et al. (2016) comprised mainly magnetite, olivine, and pyrope after dehydration.

Phyllite is a representative low-grade metamorphic rock in regional metamorphic belts, and its metamorphic grade increases with increasing pressure and temperature to become gneiss and granulite. Comprehensive research into the electrical properties of regional metamorphic belts would require the electrical conductivities of all three of these metamorphic rocks to be compared. Fuji-ta et al. (2007b) studied gneiss in the Higo metamorphic zone in Japan and found that its electrical conductivity in the direction parallel to the foliation is 0.5–1.5 orders of magnitude higher than that perpendicular to the foliation. Fuji-ta et al. (2004) studied the electrical conductivity of granulite that formed in the lower crust, in the zone of metamorphism, and reported that its electrical conductivities at high and low temperatures were similar to those reported by Satoh et al. (1998) and Ogawa et al. (1994), respectively. Compared with gneiss and granulite, the electrical conductivity of phyllite after dehydration at 1.5 GPa is similar to that of granulite and slightly lower than that of gneiss. These differences might arise from the samples' different mineral compositions and rock textures. Serpentinite is an important rock in subduction zones, and previous studies have indicated that its dehydration might account for the HCLs in such settings (Robertson et al. 2015). Phyllite is less electrically conductive than serpentinite both before and after dehydration, and the dehydration temperature of serpentinite is 100 K lower than that of phyllite at a given pressure (Zhu et al. 1999; Xie et al. 2002). The dehydration products of phyllite, gneiss, and granulite show similar slopes on a log σ versus 1/T diagram, which might indicate that the three metamorphic rocks have the same conduction mechanism (Fig. 9).

Conduction mechanism

The electrical conductivity of the phyllite samples before dehydration follows an Arrhenius relation with respect to temperature, indicating the presence of only one conduction mechanism. Fitting to the Arrhenius relation gives an activation enthalpy range of 0.69–0.81 eV (Table 1). The slope of the straight line after dehydration is similar to that before dehydration (Fig. 5).

Small polarons are considered to be the main charge carriers in Fe-bearing dry, nominally anhydrous minerals (e.g., Dai and Karato 2009a, b; Yang et al. 2012; Yang and McCammon 2012; Dai et al. 2016) and in some Fe-bearing nominally hydrous silicate minerals and rocks such as

Fig. 9 Comparison of the electrical conductivity of phyllite measured in this study and previous studies



chlorite, pyrophyllite, and serpentinite (Zhu et al. 1999, 2001; Reynard et al. 2011; Manthilake et al. 2016). Chlorite is the main mineral component of the phyllite samples, and its conduction mechanism dominates the conduction in the bulk rock. The activation enthalpy range for chlorite before dehydration is 0.55-0.56 eV, and small polaron conduction is proposed to be the dominant conduction mechanism in the chlorite at high pressures and temperatures (Manthilake et al. 2016). The activation enthalpy for chlorite is slightly lower than that for the phyllite samples, which might be linked to the phyllite's complex crystallographic structure and mineralogical composition. In addition, the calculated activation enthalpies for phyllite before and after dehydration are similar (Fig. 5). Therefore, small polaron conduction is suggested to be the dominant conduction mechanism, with the following conduction process:

$$Fe_{Mg}^{\times} + h^{\cdot} \rightleftarrows Fe_{Mg}^{\cdot},$$
 (3)

Where Fe_{Mg}^{\times} and Fe_{Mg}^{\cdot} are divalent and trivalent Fe ions at Mg lattice sites, respectively, and h[·] is an electron hole, which migrates along the same direction as the small polaron (Fe_{Mg}) in an electrical field.

Geophysical implications

The various constituent minerals examined here, before and after dehydration, are consistent with the two main metamorphic reactions that occur in subduction zones (Tatsumi and Eggins 1995):

$$2(Mg, Fe)_{4.5} Al_3 Si_{2.5} O_{10} (OH)_8 + 4SiO_2$$

= 3(Mg, Fe)_3 Al_2 Si_3 O_{12} + 8H_2 O, (4)

$$\begin{split} &3(Mg,Fe)_5\;Al_2Si_3O_{10}(OH)_8+5KAI_3Si_3O_{10}(OH,\,F)_2=8Al_2SiO_5+\\ &5K(Mg,Fe)_3\;AlSi_3O_{10}(OH,\,F)_2+SiO_2+12H_2O \end{split}$$

(5)

Therefore, the dehydration depth of pelite in a subduction zone can be determined based on the geothermal gradient and the relationship between dehydration temperature and pressure.

The observed inflection points in the relationship between electrical conductivity and temperature indicate the dehydration temperatures at 0.5, 1.5, and 2.5 GPa to be 1173, 1023, and 973 K, respectively. The dehydration temperature (T_d) of the phyllite observed here shows a linear relationship with pressure (*P*): $T_d = 1181 - 100P$. Li et al. (2005) studied the pressure dependence of the dehydration temperature of pelite, finding a relationship of T = 1165-70.6P using high-pressure differential thermal analysis (HP-DTA). Although the mineral species of the two rocks are similar, they have different proportions of hydrous minerals; their different dehydration temperatures might also be caused by different measurement methods.

As shown in Fig. 10, the geothermal gradients at cold and hot subduction zones are 4 and 10 K/km, respectively (Ahrens 1989; Peacock 1990). The corresponding dehydration points for phyllite are 3.8 GPa and 801 K and 2.0 GPa and 981 K, respectively, which occur at depths of 129 and 70 km, respectively. Previous studies have confirmed a depth range of 90–150 km as the source of arc magma (Tatsumi 1989; Saunders et al. 1991; Spandler and Pirard 2013; Bebout and Penniston-Dorland 2016). Hence, we suggest that the dehydration of pelite contributes to the development of magma in subduction zones.

Previous studies have confirmed that HCLs can be caused by hydrogen in nominally anhydrous minerals, aqueous fluid, silicate melt, carbonate melt, and graphite layers (Duba and Shankland 1982; Hyndman et al. 1993; Maumus et al. 2005; Gaillard et al. 2008; Yang et al. 2011; Dai and Karato 2014a, b, c, d; Manthilake et al. 2015). This study provides further evidence to suggest that biotite in the dehydration products of phyllite can cause HCLs. HCLs in subduction zones are generally thought to be caused by the dehydration of serpentinite (Robertson et al. 2015; Zhang et al. 2015), and the influence of pelite is disregarded. The electrical conductivity of phyllite after dehydration was observed here to be 10^{-2} to 10^{-1} S/m, a range of values similar to those of the HCLs of the middle and lower crust in subduction zones (Robertson et al. 2015). Therefore, the dehydration product of pelite might be the cause of the HCLs in subduction zones, where pelite is potentially abundant and widespread.

Conclusions

Based on the newly acquired data as elaborated and discussed above, the following conclusions can be drawn:

- The electrical conductivity of phyllite significantly increases with increasing temperature, and slightly increases with increasing pressure.
- The dehydration temperature of phyllite ranges from 973 to 1173 K at 0.5–2.5 GPa, and dehydration dramatically enhances its electrical conductivity.
- 3) Interconnected biotite is proposed to be responsible for the high electrical conductivity of the dehydration products of phyllite at 1.5 and 2.5 GPa. Dehydration-induced partial melting is observed at 0.5 GPa; however, its limited volume fraction (~0.5%) does not raise the electrical conductivity of phyllite at 0.5 GPa above that at 1.5 or 2.5 GPa.
- Given the calculated activation enthalpy and the results of previous studies, small polaron conduction is suggested to be the conduction mechanism of phyllite at high temperatures and pressures.
- 5) The dehydration depths of phyllite are similar to the depths of magmatic source regions; consequently, we infer that the dehydration of pelite contributes to the generation of magma in subduction zones.



Fig. 10 Dehydration depths of pelite in hot and cold subduction zones

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