

## Abstract

Aqueous fluids are one of the principal agents for transporting material in the Earth’s interior. Electrical conductivity measurements can be used to reveal the distribution of water in the Earth’s mantle because the presence of a water-rich fluid in a rock can significantly enhance the bulk conductivity. To better constrain the fluid distribution and fraction in mantle wedges, we measured the electrical conductivity of olivine containing minor amounts of a salt-bearing aqueous fluid at high pressure and temperature. The three-dimensional microstructure of the interstitial pores visualized by the high-resolution synchrotron X-ray computed micro-tomography (CT) shows a change in fluid distribution from isolated pockets at a fluid fraction of 0.51 vol.% to interconnected networks at fluid fractions of 2.14 vol.% and above due to grain anisotropy and grain size differences, accounting for the nonlinear increase in electrical conductivity. The rapid increase in conductivity indicates that there is a threshold fluid fraction between 0.51 and 2.14 vol.% for forming interconnected fluid networks, which is consistent with the 3-D images. Our results provide direct evidence that the presence of >1.0 vol.% aqueous fluid with 5.0 wt.% NaCl is required to explain the high conductance anomalies above 0.01 S/m detected in deep fore-arc mantle wedges.

## Plain Language Summary

Aqueous fluids are one of the principal agents for chemical transport in Earth’s interior. Precise determination of fluid fractions is essential to understand bulk physical properties, such as rheology and permeability, and the geophysical state of the mantle. Laboratory-based electrical conductivity measurements are an effective method for estimating the fluid distribution and fraction in a fluid-bearing rock. In this study, the electrical conductivity of texturally equilibrated fluid-bearing forsterite aggregates was measured for the first time with various fluid fractions at a constant salinity of 5.0 wt.% NaCl at 1 GPa and 800°C. We found that the electrical conductivity nonlinearly increases with increasing fluid fraction, and the data can be well reproduced by the modified Archie’s law. The three-dimensional (3-D) microstructure of the interstitial pores visualized by the high-resolution synchrotron X-ray computed micro-tomography (CT) shows a change in fluid distribution from isolated pockets at a fluid fraction of 0.51 vol.% to interconnected networks at fluid fractions of 2.14 vol.% and above due to grain anisotropy and grain size differences, accounting for the nonlinear increase in electrical conductivity. The rapid increase in conductivity indicates that there is a threshold fluid fraction between 0.51 and 2.14 vol.% for forming interconnected fluid networks, which is consistent with the 3-D images. Our results provide direct evidence that the presence of >1.0 vol.% aqueous fluid with 5.0 wt.% NaCl is required to explain the high conductance anomalies above 0.01 S/m detected in deep fore-arc mantle wedges.

## 1. Introduction

In subduction zones, aqueous fluids released from the subducting slab percolate through the mantle wedge, controlling arc magma genesis, and element recycling (e.g., Hermann et al., 2006; Iwamori, 1998; Tatsumi, 1986; van Keken et al., 2011). Although the pore fluid preserved in the subducting oceanic lithosphere will be expelled by compaction at relatively shallow depth, hydrous minerals may effectively carry water deep into the mantle and release it through dehydration reactions upon progressive metamorphism. Because the presence of fluid significantly enhances the bulk electrical conductivity of rocks (e.g., X. Guo et al., 2015; Shimojuku et al., 2014, 2012; Sun et al., 2020), the distribution of fluids in subduction zones...
may be imaged by magnetotelluric (MT) observations. Recently, a growing number of MT observations have revealed high electrical conductivity anomalies (0.001–1 S/m) in the deep fore-arc crust and mantle wedge in some subduction zones (e.g., Araya Vargas et al., 2019; Pommer & Evans, 2017; Worzewski et al., 2011). Because the temperature is relatively low in fore-arc regions (<700–800°C, Syracuse et al., 2010), aqueous fluid rather than silicate melt has been assumed to be the highly conductive phase present in the lower crust and mantle wedge, supplied by the subducting slab through a permeable window in the fore-arc mantle wedge (Huang et al., 2019). Although conductive minerals such as sulfide (Ducea & Park, 2000), graphite (Frost et al., 1989; Glover, 1996), and magnetite (Manthilake et al., 2016) have also been suggested to be the cause of the observed high electrical conductivity, their volume fraction may not be sufficiently high to form interconnected networks in rocks.

Fluid fraction is a key parameter that controls not only the electrical conductivity, but also other physical properties of fluid-bearing rocks, such as rheology, permeability, and seismic wave velocities (Bloch et al., 2018; Rippe et al., 2013; von Bargen & Waff, 1986; Worzewski et al., 2011). The accurate quantitative estimation of the fluid fraction, therefore, is fundamentally important to better interpret geophysical anomalies and to understand the dynamics of material cycling in subduction zones. A number of models with homogeneous and simple fluid geometries have been proposed to describe the fluid fraction dependence of electrical conductivity in fluid-rock mixtures (Archie’s law, Archie, 1942; modified Archie’s law, Glover et al., 2000; Hashin-Shtrikman upper and lower bound model [HS model], Hashin & Shtrikman, 1962; tube model, Schmeling, 1986; cube model, Waff, 1974). In the cube and HS upper bound models, all grain boundaries are assumed to be completely wetted by a fluid phase. In the tube model, the fluid forms an interconnected tube network at grain edges. Archie’s law is empirical and has been utilized to describe fluid distribution in porous water-saturated sandstone, allowing changes in fluid connectivity with increasing fluid fraction (e.g., ten Grotenhuis et al., 2005).

At high pressure (P)-temperature (T) conditions, the equilibrium fluid distribution is primarily controlled by the dihedral angle (i.e., wetting angle) between the rock matrix and aqueous fluid. In an ideal system with isotropic interfacial energy and uniform grain size, the system has one true dihedral angle accompanied by curved solid-fluid interfaces with constant mean curvature, and the fluid distribution is homogeneous. The fluid forms an interconnected network at all grain edges, as in the tube model, for a dihedral angle below the critical value of 60°. For higher dihedral angles, the fluid fills isolated pockets and a relatively high fluid fraction is required to form interconnected fluid networks (Holness, 1992, 1993; Watson & Brenan, 1987). In reality, however, minerals have anisotropic interfacial energies; thus, the system has a range of true dihedral angles associated with crystallographically controlled faceted interfaces. Pores surrounded by faceted planes can result in isolated fluid pockets at grain corners even at dihedral angles smaller than 60° (e.g., Price et al., 2006), affecting the bulk fluid connectivity of the system. In addition, based on the grain size distribution in real rocks, fluid may concentrate in the domains of smaller grains (Wark & Watson, 2000). Therefore, the fluid distribution in a fluid-bearing, deep-seated rock may deviate from the ideal systems, preventing a precise evaluation of the fluid fraction from MT data using simple theoretical models. Experimental measurements of the electrical conductivity in texturally equilibrated fluid-bearing rocks are necessary to constrain the actual relationship between electrical conductivity and fluid fraction.

In the last decade, many laboratory-based electrical conductivity measurements have been conducted in fluid-bearing systems (e.g., Guo & Keppler, 2019; X. Guo et al., 2015; Shimojuku et al., 2014, 2012; Sun et al., 2020). For example, Shimojuku et al. (2012, 2014) measured the electrical conductivity of a fluid-bearing quartzite as a function of fluid fraction and fluid salinity, and suggested that a high fluid fraction above 32.0 vol.% or a high salinity above 10.0 wt.% is required to account for high conductivity anomalies in the lower crust. However, these measurements were performed with short run durations at constant P-T; thus, the attainment of textural equilibrium was not guaranteed, similar to other previous experiments. Olivine is the dominant mineral in mantle wedges, while previous studies utilized quartz (Shimojuku et al., 2012, 2014), albite (Guo et al., 2015), and clinopyroxene (Sun et al., 2020) as the matrix mineral, which may affect the dihedral angle (i.e., fluid geometry) and fluid chemistry (i.e., charge carriers in the fluid) in the experimental systems. In addition, the lack of the three-dimensional (3-D) analyses of the fluid fraction and pore structures in previous experiments hampers the accurate interpretation of MT data due to an insufficient physical understanding of the fluid fraction dependence of electrical conductivity.
In this study, we measured the electrical conductivity in texturally equilibrated \( \text{H}_2\text{O}-\text{NaCl}\)-bearing forsterite (Mg-endmember of olivine) aggregates with various fluid fractions at a NaCl concentration of 5.0 wt.% at 800°C and 1 GPa. Combined with 3-D images of the fluid distribution determined by synchrotron X-ray computed micro-tomography (CT), the results are used to discuss the fluid fraction dependence of electrical conductivity and estimate the fluid fraction and distribution in deep fore-arc mantle wedges.

2. Experimental Procedures

2.1. Sample Preparation

To avoid the difficulties associated with electrical conductivity measurements in iron-bearing systems, synthetic forsterite was used as the starting material. Polycrystalline forsterite aggregates were synthesized from iron-free gel powder at 1200°C and 1.8 GPa in a piston cylinder apparatus with 1/2-inch diameter talc-pyrex assemblies at Bayerisches Geoinstitut, University of Bayreuth. Details of the sol-gel preparation are described in Ohuchi and Nakamura (2006). The gel powders were placed in an oven at 300°C for 12 h to remove moisture, and were then loaded in a platinum capsule (outer diameter: 5.0 mm) that was sealed by arc welding. The piston cylinder apparatus was first pressurized up to 90% of the target pressure; then, the remaining pressure was applied after heating to the target temperature. During the run, the temperature was monitored and controlled by an S-type thermocouple (Pt-Pt\textsubscript{90}Rh\textsubscript{10}) and temperature uncertainty is less than 20°C. The experiment duration was 24 h at the target temperature. The experiments were quenched to room temperature within 1 min, and the pressure was released slowly. The run products were extracted from the platinum capsules and checked under an optical microscope. The synthesized forsterite grains were then ground in an agate mortar and were sieved to obtain size fractions of 38–53 µm. Moreover, reagent-grade NaCl (Chem. Pure, 99.999% purity) was dissolved in deionized and distilled water under ambient conditions to obtain a salinity of 5.0 wt.%.

2.2. Electrical Conductivity Measurements

Electrical conductivity measurements were conducted at 800°C and 1.0 GPa in a piston cylinder apparatus with 3/4-diameter talc-pyrex assemblies at Bayerisches Geoinstitut, University of Bayreuth. The configuration of the cell assemblies was as described by Guo and Keppler (2019). A 1-mm diameter platinum rod and a Pt\textsubscript{95}Rh\textsubscript{5} capsule with an outer diameter of 5.0 mm and inner diameter of 4.4 mm were employed as the inner and outer electrodes, respectively (Figure 1). A ceramic disk with 1 mm thickness was placed at the bottom of the Pt\textsubscript{95}Rh\textsubscript{5} capsule. The forsterite powder and saline fluid of each experiment (initial fluid fractions: 0.5 wt.%, 2.5 wt.%, 5.0 wt.%, and 10.0 wt.%) were loaded into the capsule between two alumina disks with 1-mm-diameter holes containing the platinum rod inner electrode. A boron nitride disk at the end of the capsule provided the seal. A thin ceramic Al\textsubscript{2}O\textsubscript{3} tube tightly fitted around the Pt rod and the central hole of the Pt\textsubscript{95}Rh\textsubscript{5} lid was served as an insulator. Two Pt wires with a diameter of 0.35 mm were welded to the inner and outer electrodes and connected to a Solartron 1260 impedance analyzer for measuring the conductivity. The whole capsule was surrounded by another shell of boron nitride to improve sealing. The procedures for increasing temperature and pressure were the same as described above. The experiments were conducted at 800°C and 1 GPa for 8 days, and the impedance spectra were measured every day using an alternating current voltage of 500 mV sweeping from 10 MHz to 1 Hz.

The bulk conductivity \( \sigma_b \) of the whole cell was calculated from the resistance and the dimensions of the cell, using the equation for the cell constant in cylindrical geometry:

\[
\sigma_b = \frac{\ln(r_o/r_i)}{2\pi l R},
\]

where the cylindrical electrodes have an outer radius \( r_o \), and inner radius \( r_i \), \( l \) is the length of the sample, and the measured electrical resistance is \( R \). The electrical conductivities measured in this study were usually very high so that the background conductivity of assemblies was negligible for all investigated systems (Guo & Keppler, 2019; Ni et al., 2011). The two-electrode method employed in this study requires a correction
for the resistance of the electrode wires if the measured resistance of the sample is very low (Pommier et al., 2010). We utilized the short-circuit method introduced by Ni et al. (2011) to correct our measured data.

2.3. Scanning Electron Microscope (SEM) Observations

The experiments were immediately quenched to room temperature at the end of experimental run within 1 min by turning off the heating power after the last electrical conductivity measurement. The recov-
ered capsules were cut open using a diamond wire saw to expose the run products. The run products were then impregnated with epoxy resin in a vacuum box and polished down to 1.0 μm surface roughness with alumina powder and subsequently to 0.06 μm by colloidal silica suspension. The polished cross sections were observed using a field emission-type SEM (FE-SEM; JEOL JSM-7100F) with an accelerating voltage of 15 keV. The dimensions of the samples (i.e., outer radius \( r_o \), inner radius \( r_i \), and length \( l \)) were carefully measured in backscattered electron (BSE) images and the average values of these parameters were used for the conductivity calculation. This procedure should minimize possible errors due to sample deformation during the experiment.

2.4. Synchrotron X-Ray Microtomography

In order to determine the fluid distribution and fraction, the recovered samples were prepared for X-ray CT analysis with synchrotron radiation. Microtomography of run products was conducted at the undulator beam line BL20XU at the SPring-8 facility, Hyogo, Japan. The cross-section of the monochromatic X-ray beam was approximately 2 × 1 mm at 240 m from the light source (around the sample position). The highly collimated undulator radiation from the low emittance storage ring used in this study is very suitable for high spatial resolution tomography. The undulator gap (K-value) was properly tuned at each energy to obtain a relatively large field of view in the vertical direction; that is, the selected energy was slightly lower than the odd-order peaks of the undulator radiation. Therefore, the flux density of the monochromatic beam was approximately 5 × 10^{12} photons s^{-1} mm^{-2} (Uesugi et al., 2001) at the sample position. The contrast resolution of the X-ray CT system in SPring-8 has been reported by Uesugi et al. (1999). For each recovered sample, around 1,800 projections were taken by X-ray CT, and a considerable sub-volume of the run products was extracted from these images, avoiding any cracks, to precisely evaluate the fluid fraction.

2.5. Determination of Water Content

The water content in the forsterite aggregate of one recovered sample (Sample No. ECM-3) was measured using a Thermo Scientific Nicolet iN10 FT-IR Microscope (FTIR) at Tohoku University. A doubly polished section of the sample, with a thickness of ∼300 μm, was prepared for the FT-IR measurement. The unpolaredized infrared spectra of the sample were obtained from 17 sampling points. At several points measurements were carried out with either increasing aperture size (from 50 × 50 to 400 × 400 μm) or scanning time (from 3 to 25 s), to evaluate the influence of aperture size or scanning time on measured content. Then 10 sampling points were measured with a constant aperture size of 200 × 200 μm and scanning time of 12 s. The water content in forsterite lattice was calculated from the integral of the measured absorbance at 3,500–3,750 cm\(^{-1}\) after subtracting a broad background associated with free water and fluid in forsterite aggregate. In this calculation, the method and integrated extinction coefficients of Bell et al. (2003) was used along with an orientation factor of 1/3. The bulk water content in forsterite aggregate including the water in fluid inclusions, grain boundaries, microcracks, and lattice defects in forsterite was calculated from the integral of the measured absorbance at 3,100–3,750 cm\(^{-1}\) based on Equation 5 reported by Paterson (1982).

3. Results

3.1. Micro-Textures and Fluid Fractions of the Recovered Sample

The run products are composed of sintered aggregates of forsterite and interstitial pores, which were occupied by saline aqueous fluid (Figure 1). Textural maturation was well established within the run duration of 8 days. The presence of many forsterite triple junctions with angles of ∼120° (Figure 1d) indicates the attainment of the balance of interfacial tensions at triple junctions (Huang et al., 2020; Liu et al., 2018). The grain size distribution determined from BSE images by Image-J shows a narrow grain size distribution with a peak around the mean grain size (Figure 1f), in agreement with textural equilibrium (Paul, 1997; ten Grotenhuis et al., 2005). A thin reaction layer with a thickness of less than 100 μm was observed between the forsterite and the Al\(_2\)O\(_3\) disk. Although this layer contains interstitial pyroxene along with relict olivine, its influence on the bulk electrical conductivity should be negligible, as all these phases are Fe-free and therefore poor conductors (e.g., Dai & Karato, 2009).
The fluid fractions of the experimental products were precisely determined using the 3-D images that were obtained from synchrotron X-ray CT of the run products. For comparison, we also calculated the initial fluid fraction in the sample based on the weight of forsterite powder and of the NaCl solution loaded into the capsule. The fluid fractions of the samples before and after electrical conductivity measurement are shown in Table 1. Representative 3-D images of the sub-volumes of the recovered samples are shown in Figure S1. The fluid fraction in the recovered samples is always smaller than the initial fluid fraction, with the difference being up to 12.37 vol.%. Although the initial fluid fraction was precisely controlled, the fluid loss at high P-T conditions was inevitable because of the initially imperfect sealing of the experimental setup, especially during the first day. After this period, the fluid fraction likely remained constant, as discussed below, based on time-series electrical conductivity data (Figure 2). The fluid fractions estimated from the recovered samples represent the fluid fractions during the last measurements of electrical conductivity.

### Table 1

**Summary of Experimental Conditions and Results**

| Run No. | T (°C) | P (GPa) | wt.% of fluid (before EC) | vol.% of fluid (before EC) | vol.% of fluid (after EC) | Salinity (wt.%) | σ (s/m) | logσ (S/m) |
|---------|--------|--------|--------------------------|----------------------------|--------------------------|----------------|---------|-----------|
| ECM-6   | 800    | 1      | 0.50                     | 1.7(0.1)                   | 0.51(0.05)               | 5.00           | 1.65E−03| −2.78     |
| ECM-4   | 800    | 1      | 2.50                     | 8.0(0.1)                   | 2.14(0.05)               | 5.00           | 3.56E−02| −1.45     |
| ECM-3   | 800    | 1      | 5.00                     | 15.2(0.1)                  | 8.14(0.05)              | 5.00           | 2.41E−01| −0.62     |
| ECM-5   | 800    | 1      | 10.00                    | 27.4(0.1)                  | 15.05(0.05)             | 5.00           | 4.80E−01| −0.32     |

Note. The volume fractions of fluid after the experiments were determined from the recovered samples by using synchrotron X-ray CT. The temperature uncertainty is ±20°C. The electrical conductivity measurements are highly reproducible (see Figure S2), but systematic errors due to sample deformation of up to 5% are possible.

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### 3.2. Electrical Conductivity Variation With Time

Figure 2 shows the variation in electrical conductivity during each experiment. Experiments were designed to run for 8 days to guarantee textural equilibration, and the electrical conductivity of the samples was measured each day during the run. The experiment with the lowest fluid fraction of 0.51 vol.%, however, was merely run for only 5 days because the thermocouple failed on the fifth day. In all experiments, the conductivity strongly decreased during the first day and then gradually stabilized over time. Most likely, the strong change in conductivity at the beginning of the experiment is related to fluid loss, while the more gradual change after the first day may be related to textural changes.

The conductivity in all systems was essentially stable after 72 h, which indicates that both fluid fraction and fluid geometry remained constant (e.g., Figure 1d). Therefore, we consider the electrical conductivity measured just before quenching as the electrical conductivity at textural equilibration.

### 3.3. Electrical Conductivity Under Textural Equilibrium Conditions

The impedance spectra obtained in the last electrical conductivity measurements of each experiment are shown in Figure 3. For the experiments with fluid fractions of 0.51, 2.14, and 8.14 vol.%, the electrical impedance spectra contain well-developed and complete semi-circle arcs at high frequencies. In these cases, the cell resistance can be obtained from the diameter of the semicircle. For the highest fluid fraction of 15.05 vol.%, the semi-arc disappeared completely and was replaced by a segment under the real axis (Z”), which was most likely caused by inductance of the Pt wires. In this case, the cell resistance was directly obtained from the intercept with the real axis (Z” = 0). The conductivity variation among five measurements during the last 30 min before quenching is shown in Figure S2 for each experiment. The measurements are highly reproducible. The accuracy of the Solartron 1260 impedance analyzer is specified by the manufacturer as 0.1% for the range of 10 Ω-100 kΩ at 10 kHz. However, minor systematic errors could have been introduced by changes in the sample geometry upon quenching. Due to the small fluid fractions and high fluid density under run conditions, these errors are unlikely to exceed 5%.
It is expected that the conductivity circuits in forsterite aggregates depend on the geometrical distribution of the present fluid (Roberts & Tyburczy, 1999; Shimojuku et al., 2012). The impedance spectra of the systems in this study are governed by a combination of resistor and capacitor circuits. From a combination of the measured impedance spectra (Figure 3) and visualized 3-D fluid distributions (Figure 4), the conduction circuits in this study can be determined. In low fluid fraction systems with isolated fluid pockets, the sample conductivity is mainly governed by the interior of the forsterite grains, and the electrical circuit is composed of the response of the grain and fluid in a series circuit. In high fluid fraction systems with an interconnected fluid network, the sample conductivity is mostly governed by the fluid tube, and the electrical circuit is composed of the response of the grain interior and fluid-filled grain edges in a parallel circuit.

3.4. 3-D Images of Fluid Distribution

The fluid distribution in the recovered samples was visualized using the software ParaView for the sub-volume 3-D CT images (Figure 4). The fluid distribution is relatively localized on smaller scales, and homogeneous on larger scales for all samples. For the system with the lowest fluid fraction of 0.51 vol.%, the aqueous fluid occurred mostly as isolated fluid pockets (Figure 4a1). In contrast, the aqueous fluid was locally interconnected via tubular pathways in the system with a fluid fraction of 2.14 vol.% (Figure 4b1). For the higher fluid fractions of 8.14 and 15.05 vol.%, the interstitial fluid was well interconnected (Figures 4c1 and 4d1), with the diameter of interconnected tubes increasing with increasing fluid fraction.

3.5. Water Content in Forsterite

The infrared absorption spectra in the range of from 3,100 to 3,800 cm$^{-1}$ collected on 10 sampling points with the same aperture size of 200 $\times$ 200 $\mu$m and scanning time of 12 s are shown in Figure 5. The infrared absorption spectra of H$_2$O-bearing forsterite are in good agreement with those of natural mantle olivines as compiled by Beran and Libowitzky (2006). The spectra show sharp absorption peaks derived from structurally bound water in forsterite between 3,500 and 3,750 cm$^{-1}$. Some of the spectral peaks between 3,600
and 3,750 cm$^{-1}$ may also be attributed to inclusions of hydrous phases, such as talc and serpentine (e.g., Beran & Libowitzky, 2006). However, the temperature of the experiments was significantly above the stability limit of these phases (Ulmer & Trommsdorf, 1995) and therefore, it is possible that the high-frequency bands in the infrared spectrum are still due to intrinsic lattice defects. Moreover, a band at 3,300 cm$^{-1}$ and a broad absorption background at 3,100–3,750 cm$^{-1}$ are also visible and most likely represent free water in fluid inclusions in the forsterite crystals and water in some microcracks, grain boundaries, and dislocations of the forsterite aggregate, respectively. The entire absorption range of 3,100–3,750 cm$^{-1}$ was integrated to calculate the bulk water content of the forsterite aggregate (Figure 5a) based on Equation 5 given in Pater (1982). In contrast, to obtain the water content in the forsterite lattice, a linear background was subtracted from the sharp bands at 3,500–3,750 cm$^{-1}$ (Figure 5a) and the remaining absorption was integrated and calculated based on the method reported by Bell et al. (2003), as shown in Figure 5b.

To evaluate the effect of the aperture size or scanning time on the bulk water content and forsterite lattice water content, up to 17 infrared absorption spectra were collected in the range of 3,100–3,800 cm$^{-1}$ with various aperture sizes or scanning times. Their respective influence on the water content is shown in Figures S3 and S4. For the 10 sampling points with the same aperture size of 200 × 200 µm and scanning time of 12 s, the bulk water content in the forsterite aggregates ranges from 1,026 to 1,216 ppm H$_2$O with an average of 1,100 ppm, while the water content in the forsterite lattice ranges from 137 to 163 ppm H$_2$O with an average of 149 ppm. The bulk water content showed a wider range compared to the water content in the forsterite lattice because of the inhomogeneity of the sample. The bulk water contents in the forst-
Figure 4. Three-dimensional (3-D) images of fluid-bearing forsterite aggregates after electrical conductivity measurements. (a–d) 3-D images of sub-volumes of recovered samples. (a1–d1) 3-D images of the fluid distributions extracted from (a) to (d). Blue areas represent the forsterite aggregates; red areas represent the interfaces between fluids and forsterite crystals; black areas represent the interior of fluid channels. The fluid distribution is relatively localized on smaller scales, and homogenous on larger scales for all samples. The spaces between the fluid are occupied by olivine crystals. The fluid in (a1) occurs mostly as isolated fluid pockets. The fluid in (b1) is locally interconnected. The fluids in (c1) and (d1) are well interconnected. In (c1) and (d1), there is an obvious bottleneck effect, i.e., the interconnected networks are composed of coarse channels (white double-headed arrows) and fine channels (white arrows). The fluid fraction for each sample is also shown. The cube size of all 3-D images is 100 × 100 × 100 µm.
terite aggregates are close to the value of 1,000 wt. ppm H$_2$O reported by Sommer et al. (2008) including the lattice water, fluid inclusions, water in grain boundaries and in microcracks. The water contents in the forsterite aggregate are much higher than the range of 205–527 wt. ppm H$_2$O reported by Ohuchi and Nakamura (2007), which may be due to the high initial fluid fraction.

**Figure 5.** Unpolarized FTIR absorption spectra of the recovered sample ECM-3. The spectra were collected from 10 sampling points with the same aperture size of 200 × 200 µm and scanning time of 12 s. The sample thickness is ∼300 µm. (a) Absorbance spectra in the range of 3,100–3,800 cm$^{-1}$. The total area of all absorption peaks between 3,100 and 3,750 cm$^{-1}$ was integrated to calculate the water content of the forsterite aggregates. The characteristic peaks of the absorption spectra were divided into three types: Spa-1, spa-2, and spa-3. Spa-1 at 3,300 cm$^{-1}$ is the absorption peak for aqueous fluids in the fluid inclusions of the forsterite crystals; spa-2 at 3,500–3,750 cm$^{-1}$ shows the absorption peaks for OH in the forsterite lattice; and spa-3 shows a broad absorption background at 3,100–3,750 cm$^{-1}$, which can most likely be attributed to water in some microcracks, grain boundaries, and dislocations, rather than structurally bound water. (b) Absorption spectra between 3,500 and 3,750 cm$^{-1}$ extracted after baseline correction. The area of these absorption peaks was integrated to calculate the water contents in the forsterite lattice.
4. Discussion

4.1. Fluid Fraction Dependence of Electrical Conductivity

In Figure 6a, the electrical conductivities measured in this study are plotted as a function of the fluid fraction as estimated from the CT data. The electrical conductivity steeply increased from 1.65 × 10^{-3} S/m at 0.51 vol.% to 3.56 × 10^{-2} S/m at 2.14 vol.%, but increased more slowly with increasing fluid fractions above 2.14 vol.%. At fluid fractions above 8.14 vol.%, the electrical conductivity increased gently. This fluid fraction dependence of the electrical conductivity may reflect the changes in the fluid distribution in the forsterite matrix. The 3-D images showed that the fluid was distributed as isolated pockets at 0.51 vol.% (Figure 4a1), while it formed an interconnected network at fluid fractions above 2.14 vol.% (Figure 4b1). This indicates that there is a threshold of fluid fraction between 0.51 and 2.14 vol.%. Once the fluid fraction exceeds this critical value, the fluid establishes an interconnected network, shifting the predominant conduction pathway from the forsterite interior to the saline aqueous fluid network. The drastic change in fluid connectivity, therefore, may exert a primary control on the fluid fraction dependence of electrical conductivity. At fluid fractions above 8.14 vol.%, the further increase in conductivity may be related to the enlargement of the average diameter in the fluid network.

4.2. Charge Carriers in the Aqueous Fluid

Because the conductivity in our study was primarily controlled by the interconnected aqueous fluids, considering the charge carriers in the fluid is important. For the fluids with 5 wt.% NaCl in our study, Na^+ and Cl^- ions produced by dissociation of NaCl are the dominant charge carriers. Manning (2013) reported that the dissociated fraction of NaCl in aqueous fluid at 1 GPa and 800°C is ~0.82 with a salinity lower than 5.8 wt.%, suggesting that most of the NaCl in the aqueous fluid was dissociated in our experiments. Although their contribution to the bulk electrical conductivity is likely limited, H^+ and OH^- produced by minor H_2O self-dissociation at elevated temperatures and pressures may also play a role as charge carriers (Manning, 2018). Elevated solubility of forsterite in a NaCl-bearing aqueous fluid was observed in previous experimental studies (Macris et al., 2020), suggesting that the solution in our study contained Mg^{2+} and Si^{4+}-bearing species. At a low concentration and nearly neutral pH, the predominant species of dissolved silica is monomeric orthosilicic acid (Manning, 2018), which does not contribute to electrical conduction. Although there may have been several ionic species in the fluid in our samples, the bulk electrical conductivity is most likely controlled by the motion of the dominant Na^+ and Cl^- ions within the interconnected fluid network in a saline aqueous fluid-bearing system (Guo & Keppler, 2019).

4.3. Comparison With the Electrical Conductivity of Fluid-Free Forsterite Aggregates and With Pure Saline Fluids

The bulk electrical conductivities measured in this study in aggregates of forsterite containing saline fluid should fall within the range of inherent conductivities in both end-member systems because no
significant charge carriers are released into the aqueous fluid from forsterite grains, as discussed in Section 4.2. Therefore, to verify the reliability of our measurements, we compared our results with the electrical conductivities of saline fluid and of fluid-free forsterite aggregates measured in previous studies (Figure 6a). For saline fluid, the electrical conductivity of aqueous fluid with 5.8 wt.% NaCl measured at 1 GPa and 800°C was adopted for comparison (Guo & Keppler, 2019). Its reported conductivity is 35 S/m, and significantly higher than the values measured in this study even at the highest fluid fraction of 15.05 vol.%.

Because there is no data for conductivities of hydrous iron-free forsterite aggregates, we compared our data with the conductivity of dry forsterite aggregates at 800°C and 1 GPa, as calculated with the equation given by Yoshino et al. (2017). Its conductivity of 2.46 × 10^-6 S/m is almost 3 orders of magnitude smaller than the conductivity measured at the lowest fluid fraction of 0.51 vol.% in this study. Water dissolved in forsterite is expected to reduce this difference because it strongly increases the conductivity. At 800°C, the electrical conductivities of hydrous iron-bearing olivine aggregates (3.5 × 10^-3 S/m with 455 ppm H2O, Gardès et al., 2014; 3.0–4.0 × 10^-3 S/m with 100–200 ppm H2O, Wang et al., 2006) and hydrous single olivine crystal (3.0 × 10^-4 S/m with 40 ppm H2O, Yang, 2012) with XFe = Fe/(Mg + Fe) = ~0.1 are 2 to 3 orders of magnitude higher than that of dry olivine (8.0 × 10^-6 S/m, Constable et al., 1992). Therefore, the conductivity of hydrous forsterite aggregates with 137–163 ppm H2O, as in this study, may be comparable to or slightly smaller than that of the fluid-bearing hydrous forsterite aggregates at the lowest fluid fraction of 0.51 vol.%.

These comparisons show that the conductivities measured in this study are between those in fluid-free forsterite aggregates and those in saline fluids reported in previous studies, demonstrating the reliability of our measurements.

4.4. Comparison With Models of Electrical Conductivity in the Fluid-Bearing Rocks

In this section, we compare the electrical conductivities measured in this study to those predicted using models with simple fluid geometries. The HS upper and lower bound model (Hashin & Shtrikman, 1962), the cube model (Waff, 1974), the tube model (Schmeling, 1986), and a modified Archie’s law (Glover et al., 2000) have been proposed to describe the effect of the fluid fraction on bulk electrical conductivity.

The HS model (Hashin & Shtrikman, 1962) provides the upper (HS⁺) and lower (HS⁻) bounds on the bulk conductivities of fluid–rock mixtures assuming extreme cases of fluid geometry, that is, completely interconnected fluid films along grain boundaries (dihedral angle = 0°) or isolated fluid pockets (dihedral angle > 60°). The two extremes are described by the following equations:

\[
HS^+ : \sigma_b = \sigma_f \left(1 - \frac{3(1 - \phi)(\sigma_f - \sigma_s)}{3\sigma_f - \phi(\sigma_f - \sigma_s)}\right) \quad (2)
\]

\[
HS^- : \sigma_b = \sigma_s \left(1 + \frac{3\phi(\sigma_f - \sigma_s)}{3\sigma_s + (1 - \phi)(\sigma_f - \sigma_s)}\right) \quad (3)
\]

where \(\sigma_b, \sigma_f,\) and \(\phi\) are the electrical conductivity of the fluid, the solid matrix, and the fluid fraction, respectively. The cube model (Waff, 1974) assumes that cubic grains with low conductivity and uniform grain size are surrounded by a high-conductivity fluid layer of uniform thickness. The result of this model is similar to that of the HS⁺ model. Its approximate bulk conductivity is described as follows:

\[
\sigma_b = \frac{2}{3} \sigma_f \phi + \sigma_s \left(1 - \phi\right) \quad (4)
\]

In the tube model (Schmeling, 1986), the fluid is assumed to be distributed as a rectangular tube network along the grain edges, but with unwetted grain boundaries:
Based on the above equations, it is obvious that the tube model gives a lower conductivity than the cube model.

Archie’s law is an empirical relation between the fluid fraction and bulk conductivity (Archie, 1942), which is commonly applied to describe the electrical conductivity of a porous sandstone. The conventional Archie’s law is designed for a conductive phase saturating a non-conductive matrix at shallow depths and low temperatures, where the conductivity of the matrix is negligible, whereas the conductivities of rock-forming minerals at high P-T conditions may significantly contribute to the bulk conductivity (Glover et al., 2000). To include the effect of solid conductivity, a modified form of the conventional Archie’s law (modified Archie’s law) was proposed by Glover et al. (2000) as follows:

\[
\sigma_b = \sigma_f \phi^m + \sigma_s (1 - \phi)^p.
\]

where the exponents \( m \) and \( p \) represent the connectivity of the fluid and solid phases, respectively. This model allows a change in fluid connectivity according to the fluid fraction.

In Figure 6a, our results are compared with those of the models discussed above. For the model calculations, we assumed \( \sigma_f = 35 \text{ S/m} \) for the conductivity of aqueous fluid with a salinity of 5.8 wt.% (Guo & Keppler, 2019). For \( \sigma_s \), we used the conductivity of the system with the lowest fluid fraction in this study (i.e., \( \sigma_s = 1.65 \times 10^{-3} \text{ S/m} \)) in which the fluid was not interconnected as revealed by 3-D images (Figure 4a1); thus, the effect of fluid on the conductivity could be minor. Our conductivities fall within the range of those of the HS models and thus do not violate the physical bounds. Because the dihedral angle defined by the curved olivine-fluid interfaces is smaller than 60° in the olivine + saline fluid system at 800°C and 1 GPa with a NaCl concentration of 5.0 wt.% (Huang et al., 2019), the fluid in our experiments is expected to be distributed along the triple junctions resulting in mostly the same conductivities as in the tube model. However, our conductivities are actually smaller than those of the tube model (Figure 6a). There are two plausible reasons for this discrepancy. First, this discrepancy may be attributed to the interfacial anisotropy of forsterite in our system. The interfacial anisotropy of forsterite results in the coexistence of both curved and faceted interfacial boundaries as observed in our samples (Figure 1) even when the system reaches the minimum surface energy (Price et al., 2006). The presence of faceted planes would lead to a range of true dihedral angles having the different wetting properties in different crystallographic orientations (Watson & Brenan, 1987). At low fluid fractions, the fluid pores surrounded by faceted planes have difficulty forming an interconnected network even at dihedral angles smaller than 60° (Price et al., 2006), while they can be interconnected at relatively high fluid fractions depending on the dihedral angle. This could result in the establishment of fluid interconnections at a range of fluid fractions, which is consistent with the change in fluid connectivity according to the fluid fractions confirmed by our CT images. Second, the normal grain growth driven by Ostwald ripening results in a heterogeneous grain size distribution ranging from 5 to 100 μm in our systems (Figure 1), which plays a significant role for the fluid distribution and thus the permeability (e.g., Cerpa et al., 2017; Wark & Watson, 2000). The fluid is expected to be concentrated in domains with finer grain sizes, assuming the presence of curved interfacial boundaries everywhere in a fluid-bearing, deep-seated rock system. Although the presence of faceted interfacial boundaries may decrease the degree of this fluid redistribution, the permeability in domains with finer grain sizes will locally match or exceed that in domains with coarser grain sizes (Wark & Watson, 2000). On the whole rock scale, however, such fluid localization increases the tortuosity of the fluid path, which might need a certain volume fraction of fluid to establish the interconnected fluid networks as seen in our CT images and hamper the enhancement of the bulk permeability and electrical conductivity. Furthermore, even for the high fluid fraction systems, in which the fluid was well interconnected, our conductivities were still smaller than those of the tube model. One plausible explanation for this is the variation in tube diameter along the tubes as modeled by Watanabe and Peach (2002), which is consistent with the bottleneck effect observed in the 3-D images of this study.
Although the modified Archie’s law is derived from the empirical Archie’s law, it considers both the conductivities of solid and fluid phases and describes the change in the degree of fluid connectivity with increasing fluid fraction, which could be an appropriate mixing model for determining the fluid fraction dependence of our conductivities, as discussed in Section 4.1. Therefore, we used the modified Archie’s law to fit our data with a variable cementation exponent \((m)\), and found that the \(m\) value of 1.9 best explains our data (Figure 6a).

5. Implication: Estimation of Fluid Fractions in Fore-Arc Mantle Wedges

An increasing number of MT studies have found the presence of high electrical conductivity anomalies (0.001–1 S/m) in the deep fore-arc regions of Cascadia (Jiracek et al., 1989; Soyer & Unsworth, 2006; Wannamaker et al., 2014), Mexico (Jödicke et al., 2006), Costa Rica (Worzewski et al., 2011), North Chile (Araya Vargas et al., 2019; Brasse, 2002; Schwalenberg et al., 2002), North Honshu (Mishina, 2009), Southern Kyushu (Ichiki et al., 2000), Ryukyu-Philippine (Shimakawa & Honkura, 1991), and New Zealand (Wannamaker et al., 2002, 2009). The high electrical conductivity anomalies in the Earth interior may be attributed to the presence of conductive mineral phases (sulfide, Duca & Park, 2000; graphite, Frost et al., 1989; Glover, 1996; magnetite, Manthilake et al., 2016), melt, or aqueous fluid. Conductive minerals are unlikely to be responsible for large-scale anomalies due to their small volume fractions in crust and mantle. Although fluid-induced partial melting widely occurs in mantle wedges, thermal models suggest that temperatures in the fore-arc regions are below 700–800 °C (Syracuse et al., 2010). Therefore, partial melting of the fore-arc mantle is unlikely because the solidus temperature of fluid-saturated depleted peridotite is higher than 920°C (Saha & Dasgupta, 2019). In addition, melts are much less conductive than aqueous fluid (e.g., Ni et al., 2011; Sinmyo & Keppler, 2017), which implies that to account for the same magnitude of conductivity at 800°C, the melt fraction would have to be about 100 times higher than the fraction of saline fluid. Such high melt fractions would significantly decrease longitudinal and shear wave velocities \((V_L\) and \(V_S\)) and increase \(V_L/V_S\) ratios (Takei, 2002). However, this is not always consistent with seismic data in the fore-arc mantle (Wiens et al., 2008).

The high electrical conductivity anomalies in the fore-arc mantle, therefore, are most likely due to the presence of slab-derived aqueous fluids. In hot subduction zones, such as Cascadia and Mexico, fluids liberated from the shallow subducting slabs at depths of 30–60 km can penetrate through the highly serpentinized mantle wedge and accumulate in the deep fore-arc region (Nakatani & Nakamura, 2016; Reynard et al., 2011). In intermediate and cold subduction zones, in contrast, saline fluid liberated at a depth of ~80 km can penetrate all the way through the fore-arc mantle wedge without mantle melting and serpentinization due to low dihedral angles (Huang et al., 2019). Therefore, our experimental results can be applied to MT data of intermediate and cold subduction zones where the slab-derived saline fluids can be present in the non-serpentinized fore-arc mantle. We assume that slab-derived fluid pervasively migrates through interconnected pores in the fore-arc mantle (Huang et al., 2019, 2020), causing a compaction of deformable solid matrix (McKenzie, 1984; McKenzie & Bickle, 1988). Although focused flow is another possible transport mechanism of aqueous fluid in the fore-arc mantle, it is likely less important in causing electrical conductivity anomalies on an MT grid scale. Remarkably high electrical conductivity anomalies of 0.1–1 S/m were often observed in crustal regions where the dominant minerals are not olivine. We thus focus on the high electrical conductivity anomalies of 0.001–0.1 S/m detected at mantle depths.

Our experimental results provide a realistic relationship between the electrical conductivity and fluid fractions in the forsterite-saline fluid system at 800°C and 1 GPa, with a fluid with a NaCl concentration of 5 wt.%, under conditions of textural equilibrium. In natural mantle settings, however, olivine contains iron, which enhances the electrical conductivity of the olivine matrix through polaron conduction (Fei et al., 2020; Yoshino & Katsura, 2013). To incorporate the effect of iron into the conductivity-fluid fraction relationship, we recalculated the bulk conductivity by using the conductivity of hydrous iron-bearing olivine aggregate \((3.0 \times 10^{-3} \text{ S/m}, \text{Wang et al., 2006})\) according to the modified Archie’s law with \(m = 1.9\) and the tube model (Figure 6b). The results showed that the difference in bulk conductivity between the iron-free and iron-bearing systems is negligible when the fluid forms an interconnected network in the models (i.e., the modified Archie’s law at fluid fractions above ~1 vol.% and the tube model). At fluid fractions below 1
vol.%, the bulk conductivity according to modified Archie's law is slightly higher in the iron-bearing system than in the iron-free system due to the contribution of the matrix conductivity.

In Figure 6b, the range of electrical conductivity anomalies (0.001–0.1 S/m) detected in fore-arc mantle wedges is shown. Based on the modified Archie's law calibrated by our experimental data, the presence of ~1.0–5.0 vol.% aqueous fluid with 5.0 wt.% NaCl is required to cause the high electrical conductivity anomalies of 0.01–0.1 S/m in fore-arc mantle wedges. This result does not depend on the iron concentration of olivine, and is almost consistent with a fluid fraction of ~2.0 vol.% for most mantle wedges (Iwamori, 1998). If the fluid completely wet all grain boundaries, compaction of solid grains can rapidly squeeze out the fluid from the rock. However, as our CT images demonstrate, the fluid rather forms some kind of interconnected channels along the grain edges. Draining of such fluid requires a change in grain shape that is limited by slow, thermally activated processes, such as recrystallization and deformation. Therefore, at a low-temperature fore-arc region, compaction may occur on geologic timescales, which could allow the presence of a few vol. % of fluid in the form of an interconnected network in the fore-arc mantle even on typical subduction timescales. The highest conductivity of ~0.1 S/m which corresponds to a high fluid fraction of ~5 vol% is generally limited at the upper part of the mantle wedge. This may indicate fluid accumulation due to the inhibition of compaction-driven fluid flow by the highly viscous mantle (Cerpa et al., 2017) and/or the sealing by the strong facets and higher dihedral angles of the lower crust minerals such as amphibole (e.g., Laporte & Watson, 1995; Laporte et al., 1997; Price et al., 2006). Alternatively, the highest conductivity may be attributed to fluid salinities higher than 5.0 wt% NaCl. Such salinities may be produced by the desiccation of the fluid during the ascent in the wedge (Bernini et al., 2013; Reynard, 2016; Reynard et al., 2011).

The anomalies of 0.003–0.01 S/m may represent less interconnected fluid pores with fluid fractions below 1.0 vol.% in the iron-bearing olivine matrix. The lower conductivities of 0.001–0.003 S/m may be attributed to iron-bearing olivine with a water content smaller than 100–200 ppm under fluid-undersaturated conditions. The conventional tube model would lead to lower fluid fractions of ~0–0.8 vol.% for the anomalies of 0.003–0.1 S/m. The large difference in the estimated fluid fractions between our model (i.e., ~1.0–5.0 vol.%) and the conventional tube model (i.e., ~0–0.8 vol.%) emphasizes the importance of our experimental data, which describe the realistic fluid distribution with interfacial anisotropy at mantle conditions.

The effects of temperature, pressure, and fluid compositions on the estimated electrical conductivity should be considered. Salinity significantly affects the conductivity because salt acts as an effective charge carrier in the form of dissociated ions (Guo & Keppler, 2019) and also increases the solubility of olivine, which increases the solute concentration in the fluids and affects the fluid geometry by changing the dihedral angle (Huang et al., 2019). The original salinity of the slab-derived fluid produced by dehydration reactions has been estimated to be 0.5–2.0 wt.% (Li & Hermann, 2015), but it can be enhanced during the fluid ascent in the fore-arc mantle wedge owing the preferential incorporation of water into the mantle minerals (Bernini et al., 2013; Reynard, 2016; Reynard et al., 2011). The salinity of 5.0 wt.% used in this study is plausible because comparable values were reported in upwelling slab-derived fluids (6.6 wt.%, Kusuda et al., 2014; Matsubaya et al., 1973) and fluid inclusions in a mantle xenolith (4.1–6.1 wt.%, Kawamoto et al., 2013). In addition, it does not deviate much from the salinity of seawater (3.5 wt.%), which is likely representative of pore fluids expelled by mechanical compaction in the shallow part of subduction zones. Carbon dioxide in the fluid may decrease the olivine-fluid dihedral angles at low-T and high-P conditions corresponding to fore-arc mantle wedges (Huang et al., 2020). The higher P-T conditions expected at greater depths enhance the conductivity of the fluid (Guo & Keppler, 2019) and the olivine solubility (Yoshino et al., 2007). The presence of pyroxenes may also increase the solubility of olivine (Tiraboschi et al., 2018) and decrease the olivine-fluid dihedral angle (Huang et al., 2020). If pyroxenes are enriched in Fe and H2O, they may effectively enhance the bulk conductivity (Yang & McCammon, 2012). However, because the silica is definitely needed to produce pyroxenite from peridotite protolith and the amount of silica transported by the slab-derived aqueous fluid/melt is limited, pyroxene formation by metasomatism in mantle fore-arc mantle wedge may be at most a local phenomenon which could not account for the high conductivity anomalies detected in the large scale. The factors mentioned above more or less increase the bulk conductivity to some extent, thereby lowering the estimated fluid fractions, although further experimental constraints are needed. Therefore, our data could provide the upper bound on the estimated fluid fraction in the fore-arc mantle. Nevertheless, the presence of significant amounts of aqueous fluid in the mantle suggests intense fluid circulation through the fore-arc mantle wedge in subduction zones.
6. Conclusion

In this study, we measured the electrical conductivity of fluid-bearing forsterite aggregates with various fluid fractions and a constant salinity of 5.0 wt.% NaCl in the fluid at 1 GPa and 800°C. Textural equilibrium was achieved owing to the long run durations of up to 8 days applied in this study. This was verified by the concentrated grain size distribution with a peak close to the mean grain size and the attainment of balance of interfacial tension at triple junctions. Our results enable us to establish a more realistic relationship between fluid fraction and bulk electrical conductivity in a non-ideal system, and to precisely estimate the fluid fraction in mantle wedges.

We found that the electrical conductivity increases nonlinearly with increasing fluid fractions, and that the modified Archie's law reproduces the experimental data very well. The 3-D microstructure of the interstitial pores determined by synchrotron X-ray CT shows a change in fluid distribution from isolated pockets to interconnected networks with increasing fluid fraction, accounting for the nonlinear increase in the electrical conductivity. Although the dihedral angle in the olivine-fluid system with a salinity of 5 wt.% is smaller than the critical value of 60° at 1 GPa and 800°C (Huang et al., 2019), the interfacial anisotropy of forsterite and the difference in grain size might localize the fluid distribution and further hamper the bulk electrical conductivity. These factors result in a deviation of the electrical conductivity obtained in our study from the idealized tube model. Moreover, a rapid increase in bulk conductivity indicates that a threshold of fluid fraction exists between 0.51 and 2.14 vol.%, beyond which interconnected fluid networks form, which is consistent with the visualized 3-D fluid distributions in the recovered samples. Our results provide direct evidence that the presence of >1.0 vol.% aqueous fluid with moderate salinity is required to explain the high conductivity anomalies above 0.01 S/m detected in fore-arc mantle wedges.

Data Availability Statement

The authors provide the datasets on figshare (https://doi.org/10.6084/m9.figshare.13245194.v1), including the electrical conductivity measurement, three-dimensional CT image, grain size distribution, and FTIR data, to support our research.

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