Electrical conductivity measurement of granulite under mid- to lower crustal pressure–temperature conditions

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SUMMARY
We have developed a technique to measure electrical conductivity of crustal rocks with relatively low conductivity and complicated mineral components in order to compare with results given by magneto-telluric (MT) measurements. A granulite from Hidaka metamorphic belt (HMB) in Hokkaido, Japan at high temperature and pressure conditions was obtained. The granulite sample was ground and sintered under the conditions similar to those of mid- to lower crust. We have observed smooth and reversible change of conductivity with temperature up to about 900 K at 1 GPa. The results were consistent with the electrical conductivity structures suggested by the MT data analysis. Considering pore fluid conduction mechanism or the role of accessory minerals in the rock, the mechanisms of electrical conductivity paths in dry or basic rocks should be reconsidered.

Key words: electrical conductivity, granulite, magnetotelluric, pore.

1 INTRODUCTION
The electrical conductivity structures of the crust and the upper mantle have been extensively studied by the broad-band magneto-telluric (MT) method. The MT soundings are sensitive enough to detect layered structures and the collected data can be analyzed to profile the properties of lithosphere with precision. Such investigations have been carried out in many regions under continents (e.g. Jones 1987) and island arcs. Comparing with the traditional geomagnetic depth sounding (GDS), the MT sounding became more effective by improving observation and data analysis.

However, results of MT soundings are difficult to explain due to lack of knowledge on electrical conductivity of concerned rocks. Even if such knowledge is available, results of MT soundings do not always agree with those of laboratory experiments. The laboratory data on rocks thought to be derived from the lithosphere is necessary.

Olhoeft (1981) has shown that, although electrical properties are extremely useful because of the high sensitivity to subtle changes within the earth, they are sometimes complicated to interpret the conductivity change due to the great number of parameters.

Glover & Vine (1992) measured the electrical conductivity of both carbon-bearing and carbon-free granulites at high temperatures and pressures. Glover & Vine (1994) reported the electrical conductivity of rocks saturated with saline fluids. Shankland et al. (1997) measured the conductivity of rocks freshly cored from depth at the German continental scientific drilling site (KTB hole). This attempt provided information on the increase of conductivity and reconnection of solid conductors in the rocks. The authors showed an increase in conductivity due to reconnection of carbon conduction pathways with increasing pressure.

Although it was considered that fluids and accessory minerals play essential roles in electrical conduction in the mid- and lower crustal rocks, our results suggest that conductivities of basic mid- to lower crustal rocks can account for results from MT soundings. In addition, outcrop raw rocks sometimes have cracks inside. Cracks in rocks are difficult to close by pressure up to 1 GPa (e.g. Knoche et al. 1998). Presence of cracks should cause serious errors to measure electrical conductivity.

This paper will report on the electrical properties of sintered granulite under the conditions in the mid- to lower crust. In particular, rock sample obtained from well-known geological field site of Hidaka metamorphic belt (HMB) in Hokkaido in Japan was used for the electrical conductivity experiments. The Hidaka Belt is noted for its high-temperature metamorphic rocks. Osanai (1985) studied pressure and temperature conditions in this area, precisely. Due to the detailed geological surveys, we have much knowledge of granulite studied by many authors. In addition, good quality MT data were obtained in this area and can be compared. The purpose of the present study is to measure electrical conductivity of rocks with definite pressure and temperature conditions within the crust.

Finally, the most important goals in this measurement are to synthesize a homogenous crustal rock sample and to keep the sample at desired pressure of 1 GPa and temperatures up to ca 900 K. Clear correlation and agreement are found between the results of our

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conduction measurements and the data obtained from MT measurements.

2 EXPERIMENTAL PROCEDURE

2.1 Sample preparation

A granulite sample was obtained from Hidaka Metamorphic Belt (HMB) in Hokkaido, Japan as indicated in Fig. 1. The HMB is located in the south of Hokkaido and is noted for its high-temperature metamorphism. The HMB consists of the main zone and the western zone, which are bounded by the Hidaka main thrust. The main zone is composed of various kinds of metamorphic rocks (e.g. Komatsu et al. 1983). The granulite-facies metamorphic rocks were formed at the lower crust, and are distributed extensively in the lowermost part of the main zone of Hidaka belt. The pressure and temperature (P–T) condition are considered to be less than 1 GPa and ca 700–800 °C, respectively, beneath the Hidaka metamorphic belt. In addition, the metamorphic grade of granulite facies in this region is evident (Osanai 1985).

The sample was homogeneously ground down to less than grain size of 100 μm, and examined by x-ray diffraction. To obtain a homogenous rock sample, we have sintered at definite pressure and temperature. The granulite powder sample was sintered at 890 K and 1 GPa, which are considered to be less than equilibrium conditions of this granulite (Osanai 1985), in a sealed platinum capsule to avoid contamination. The assembly for sample sintering is shown schematically in Fig. 2. The capsule was surrounded by NaCl in order to avoid formation of cracks during decompression. The sintering time was about 1 hr.

A large number of authors have showed that the influence of oxygen fugacity during the sintering process and electrical conductivity measurements, cannot be neglected and interpretations from laboratory measurements require some caution (e.g. Olhoeft 1981; Berckhemer et al. 1982). In this study, oxygen fugacity must be controlled during the sintering, which could not be controlled completely. However, we sealed the sample by the Pt to avoid migration of oxygen, and minimized the sintering duration. Inside the capsule, the effects of oxygen fugacity are less than those of mantle materials because the sintering temperature is relatively low (e.g. Olhoeft 1981). Kariya & Shankland (1983) pointed out that the major effect of oxygen fugacity on conductivity measurements involves the oxidation state of iron. We conducted qualitative and quantitative evaluations using electron probe microanalysis (EPMA) to detect the reduction of Fe ions. Consequently, no major change of Fe ions can be observed. This analysis can prove that variation of oxidization and/or deoxidization states are only minor during the sintering process. Further, there are graphite and boron nitride out of the capsule, which will cause reduced conditions in the high-pressure cell.

From these reasons, we consider that oxidation of the sample was minimized in this study. In addition, we paid attention to make pore-free sample during the synthesis process (Katsura et al. 2001) and conducted qualitative and quantitative evaluations using EPMA to detect the reduction of Fe ions before and after conductivity measurements.

2.2 High-pressure experiments

High pressures are generated using a DIA-type cubic anvil press with maximum load of 2000 ton (UHP-2000/20, Sumitomo Heavy Industries, Ltd) installed at the Institute for study of the Earth’s Interior, Okayama University. The edge length of the anvil truncation is 15.0 mm. A pyrophyllite gasket of 3.0 mm in thickness is used. Generated pressures were calibrated at ambient temperature by detecting phase transitions of Bi (2.55 GPa), Tl (4.4 GPa) and Ba (5.5 GPa).

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Figure 3. Schematic cross-section showing details of the cubic pressure cell for electrical conductivity measurement. The cylindrical nichrome heater ensures the accurate temperature gradient in the sample region. The molybdenum discs are used as electrodes and are also utilized to keep the oxidation state of the sample.

Schematic drawing of the high-pressure cell for electrical conductivity measurement is shown in Fig. 3. The cell assembly mainly consists of MgO and BN. The cube shaped pressure medium and other parts of MgO are heated at ca 1000 °C for 2 hr to exclude the contained water and moisture before the measurement. The sample with the dimensions of 2.6 mm in diameter and 1.5 mm in thickness was surrounded by BN to avoid cracking because of its softness. Temperature was raised by a nichrome foil heater with thickness of 20 µm. Two pairs of chromelalumel thermocouples, 0.20 mm in diameter, were used and connected by molybdenum discs. These thermocouples were also used for electrical conductivity measurement. Alumina sleeves are used for insulation between the side anvils and thermocouples. The linearity between applied electric power and the temperature was checked from room temperature up to about 1300 K. In this temperature range, the thermocouple was not amputated and insulated from the nichrome foil heater.

Pressure for measurement is 1 GPa, which represents that of the lower crust. The temperature range is from ca 300 to 890 K. As mentioned later, the conductivity measurement was strongly influenced by the presence of water in and around the cell assembly and sample, so that cyclic heating and cooling was necessary beyond 500 K.

2.3 Measurements

The electrical circuits for measurement are shown in Figs 4(a) and (b). The sample is connected to a function generator and reference resistance of 1 MΩ in a series. A sinusoidal signal with amplitude of 1 Vp-p was applied to the circuit. The voltage applied on the reference, Vref as monitored by a digital multimeter (HP-3458A, Hewlett–Packard Development Company). In the case that the sample resistance is comparative or smaller than the reference resistance Rref, the voltage applied on the sample Vsample was monitored by another HP-3458A multimeter as shown in Fig. 4(a). If the resistance of the sample is very high, however, electrical noise is too serious to measure the voltage on the sample. Therefore, in this case, the voltage generated by the function generator was monitored, which is considered to be the same as those applied on the sample (Fig. 4b).

To obtain high quality and stable data, background noise was always monitored during the data acquisitions and, to avoid bias in the data, several data acquisitions were repeated. One data set contains two cycles of the signal and the number of data points in one data set is 4096.

The sample impedance Zsample is calculated as follows:

\[ Z_{sample} = R_{ref} \left( \frac{V_{ref}}{V_{sample}} \right) \]

The resistance of the sample Rsample is estimated by assuming that a capacitance C is connected to the sample in parallel, that is,

\[ 1/R_{sample} = 1/Z_{sample} + j\omega C \]

where \( \omega \) is angular frequency of the sinusoidal signal.

This low frequency was chosen as 10 mHz in order to avoid significant noise at frequencies higher than 60 Hz from the commercial power supply. In addition, as the electrical circuit for measurement itself has a large capacitance, low frequencies must be used for the precise electrical conductivity measurements. The capacitance was estimated less than a few nF at room temperature. However, because of small argument, the estimated values of capacitance should have significant errors and, therefore, these capacitance values are meaningless.

The electrical conductivity of the sample was calculated from the resistance and dimensions of the sample. The sample dimensions were measured after decompression, and not corrected to high P–T conditions.
2.4 Insulation test

As mentioned later, the sample resistance measured in this study is very high, that is, from 4.1 × 10^9 to 4.8 × 10^4 Ω. Hence we have to check carefully the insulation of the measurement system. First, we directly connected a reference capacitor that has a capacitance of 10 nF and resistance higher than 10^14 Ω. However, we obtained resistance of 1.4 × 10^10 Ω for this reference, which is the upper bound of the resistance measurement in the present system.

Next, we put a BN disc as a sample in the high P–T assembly to examine insulation of the assembly against temperature. The results of this insulation test are shown in Fig. 5. Before heating, the insulation resistance is in the order of 4.5 × 10^7 Ω. By heating to about 500 K, the insulation resistance is once increased and decreased up to about 1200 K in the third cycle, the resistance by three orders of magnitude decreases, that is, to about 2.9 × 10^6 Ω. Even after the sample is cooled to ambient temperature, the high insulation resistance can be kept. Therefore, the relatively low insulation resistance before heating is considered to be due to adsorbed water in the assembly.

The high insulation resistance can be kept up to 1300 K. However, the insulation resistance largely decreases above 1300 K. The large decrease of insulation resistance from 1000 to 1300 K is considered to be attributed to change of conduction mechanism of BN around the dummy sample.

3 RESULTS

Before and after the electrical conductivity measurements, sample characteristics should be examined. Through the qualitative and quantitative evaluations using electron probe microanalysis (EPMA), microstructures of the sintered sample were inspected. Minerals, cracks or fractures of the sample sintered up to about 900 K are observed by backscattered electron image (BEI). In Fig. 6, all grains are relatively uniform and no major fractures can be seen. Grain size of each mineral is less than 100 μm in diameter. As Osanai (1985) reported that granulite consisted of garnet, cordierite, biotite, quartz and plagioclase with minor pyrrhotite. Identified elements and mineral compositions of the sintered sample correspond to those of powder granulite sample obtained from raw rock.

Typical examples of raw data of electrical conductivity measurements are shown in Figs 7(a) and (b). Though a few percent of the noise level can be seen of the measurement at 275 K as shown in Fig. 7(a), the measurement result at 675 K shows completely noise-free data in Fig. 7(b). We have collected raw data at each temperature condition. It is seen that the raw data show stable values from mid to high-temperature ranges.

After conducting several preparatory experiments of conductivity measurements, stable data were obtained. Experimental results are plotted on a log conductivity versus reciprocal temperature plot as shown in Fig. 8. To exclude the moisture of the assembly, a large number of heating and cooling cycles were repeated. In the first cycle, the sample was heated up to about 500 K. Subsequently the sample was cooled to room temperature. In the second heating cycle, the sample was heated to 820 K. The conductivity seemed to stabilize between 670 and 820 K. The conductivity values were generally lower than those in the heating cycle, although they were almost identical at the highest and lowest temperatures. In the third heating cycle, the sample temperature was raised to 870 K. From 570 to 870 K the conductivity increased from 4.9 × 10^{-4} to 5.9 × 10^{-3} S m^{-1}. Below 500 K, the behaviour of electrical conductivity of the sample is unstable. However, it can be seen that there is a stabilized gradient of the electrical conductivity in the third cycle above 500 K. Especially, above 570 K (ca 300 °C), the relationship between conductivity and the temperature becomes stable as expected from the classical Arrhenius equation. In general, it is very difficult to have the same conductivity values in the heating and cooling stages. Lee et al. (1983) pre-baked a sample of unsaturated amphibolite gneiss to 750 °C in order to dehydrate the sample assembly. Then, they measured the electrical conductivity of the sample, and concluded that the adsorbed water or unstable mineral conduction paths were no longer present in the baked sample. Our experiments also succeed in the electrical conductivity measurement to have quite stable and reversible values up to 870 K after annealing to intermediate temperatures. These results suggest that the rock sample sintered in the mid- to lower crustal condition is homogeneous.

The conduction paths of the sample give rise to the temperature dependence of the conductivity and independence from pressure. As shown in Fig. 8, all data do not indicate the real conductivity values at temperatures below 500 K due to moisture in and around
Figure 7. (a) and (b). Typical examples of raw data at 275 K (a) and 675 K (b). Two curved lines denote volts and current values on the sample, respectively.

Figure 8. Electrical conductivity values of granulite sample are plotted. This diagram presents plots of the electrical conductivity versus reciprocal temperature (1000/T). Small triangles and diamonds represent the conductivity of the heating and cooling stage of first and second cycles respectively. Solid circles represent the electrical conductivity values in third cycle. They are stable reversible and reproducible above 500 K.

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the assembly. However, the values at temperature from 570 K to 870 K are comparable to compiled data of dry lower crustal rock examined by Kariya & Shankland (1983). This temperature range is important because these conditions correspond to those of mid- to lower crust.

The inspection of mineral composition and microstructures of recovered sample after the electrical conductivity measurement is also required. No major change of minerals revealed by Fig. 9, illustrates the sample itself is essentially unchanged. Results from EPMA showed that weight per cents of all of identified minerals are similar to those of sintered sample. The chemical compositions of recovered sample are listed in Table 1. The compositions of some minerals could not be measured because of small grain sizes for EPMA. However, we could measure cation ratios (e.g. Fe ions) of identified minerals, and these were computed and evaluated. Consequently, we verified that recovered sample was not affected by chemical interaction of minerals.

4 DISCUSSIONS

4.1 Behaviour of electrical conductivity in the heating cycles

The present sample contains several kinds of minerals. These minerals could be effectively annealed in the different temperatures, and could react at several temperature stages. Therefore, it is not easy to explain the features of the electrical conductivity values in the heating cycles.

As is described in the previous section, the conductivity values are generally lowered by repeating the heating cycles. This is probably because the sample was annealed in the heating cycles. By annealing, the defects in minerals decreased, and effective conduction mechanisms change from extrinsic ones to intrinsic ones. In the first heating, we cannot recognize any stable trend below 500 K. However, the stable data is seen in the temperature range from 500 to 870 K in the third cycle. The stable trend implies that the sample is well annealed and intrinsic conduction mechanism operates in such conditions.

It is also noted that the values are essentially the same in the first cooling and second heating, and in the second cooling and first heating, in high-temperature range. These features imply that the sample was effectively annealed at the highest temperature.

The sintering temperature was 890 K. As far as the measurement temperatures did not exceed the sintering temperature, the conductivity decreases if the sample temperature was decreased. However, once exceeding the sintering temperature, the conductivity is almost constant. It was confirmed by several preparatory experiments. Thus we carefully conducted electrical conductivity measurements of granulite sample below the sintering temperature.

4.2 Comparison with field data

As the stabilized trend of the temperature range from 570 to 870 K shows stable data and corresponds to that of mid- to lower crustal conditions, we can discuss the relation between laboratory data and electromagnetic (EM) field data. In application, the electrical conductivity of granulite sample measured in this study is compared with field data of Hidaka metamorphic belt (HMB), Hokkaido, Japan. This direct comparison may account for the properties of mid- to lower crust beneath this metamorphic belt.

Arita et al. (1998) successfully investigated the crustal structure around the HMB by the geological and geophysical methods. The integrated seismic refraction and magneto-telluric (MT) data indicate the collision tectonics and geometries of the crust. Especially, the origins of deep rocks are well interpreted and these results are consistent with geological patterns around the area where the granulite rock is sampled. Osanai (1985) suggested the pressure and temperature conditions in the lower crust of this area. In particular, the thermostatometric analyses of granulite-facies rocks indicate P–T conditions corresponding to those of lower crust. Thus we will be able to compare the results of electrical conductivity with that of MT observations.

Ogawa et al. (1994) conducted a wide-band MT survey across the northern part of metamorphic belt as shown in Fig. 1 (profile 1)
and modelled the 2-D crust-structure. Beneath the HMB, a low conductive block was found at depths from 5 to 50 km. This block may consist of the high-grade and high-temperature metamorphic rocks in the HMB. Satoh et al. (1998) carried out MT investigation around the southern part of HMB as shown in Fig. 1 (profile 2). Their 2-D results show that there are high- and low-conductive layers beneath the HMB. Compiled field MT data and results from averaged laboratory measurements data are summarized in Fig. 10.

Although the electrical conductivities of granulite were obtained at temperatures from the surface to the lower crust, the stable and reliable conductivities were obtained from 500 to 870 K. Therefore, our comparisons can be focused only on the conductivities from mid- to lower crust. Ogawa et al. (1994) found low \((3.3 \times 10^{-5} - 5.0 \times 10^{-4} \text{ S m}^{-1})\) conductive block beneath the HMB. The high-temperature metamorphic rock will not contain a hydrous fluid phase, so that the MT observation may show low conductivity in the mid- to lower crust.

The results of Satoh et al. (1998) accord with the conductivity of mid- to lower crustal temperature. We measured a conductivity of \(1.4 \times 10^{-7} \text{ S m}^{-1}\) at 670 K and a conductivity of \(5.9 \times 10^{-3} \text{ S m}^{-1}\) at 870 K in our reproducible experiments. These values are corresponding to the conductivity obtained from MT data, which is corresponding to the mid- to lower crustal temperature. Unfortunately, this MT sounding could not resolve deeper than 28 km depth because of lack of MT data in the low-frequency range. Nevertheless this accordance shows that there is some correlation between our experimental results under mid- to lower crustal condition and data from MT observations. Cooperative wide-band MT observations have been intensively carried out across the HMB in 2000 and 2001 (Mogi and Ogawa; personal communications). We hopefully await an opportunity to compare our data with the results from their MT analysis.

Our experimental results can account for the electrical properties of high-temperature metamorphic rocks in the mid- to lower crust of HMB in Hokkaido of Japan. However, our result of sintered granulite sample cannot explain the properties of global lower crust in the stable continent. If we know local tectonic setting and physical conditions around the sampling site of rocks, the data of laboratory measurements and the field MT data can be directly compared.

### 4.3 Comparison with other experiments

Electrical conductivity values of granulite are plotted in Fig. 8. As is shown in this figure, the conductivity of granulite of 570–870 K shows stability. We focus our attention on this temperature range because it corresponds to temperatures of the mid- to lower crust.

Olhoeft (1981) showed the electrical resistivity versus reciprocal temperature for dry granite and hornblende schist. Although the measurements on the dry westerly granite and hornblende schist indicate results similar to those of ours in the high-temperature range, the conductivities in the low-temperature range are scattered and unstable. The data most consistent with results on the lower crustal conditions can be seen in the compiled data of dry gabbro or basalt examined by Kariya & Shankland (1983). Mineral composition in gabbro or basalt is different from that of granulite, but both rocks surely represent the lower crustal rocks.

Glover & Vine (1992) ascribed the enhancement of conductivity to reconnection of carbon conduction pathways in the granulite sample. Furthermore, Duba et al. (1994) noted that some accessory minerals in the lower crust enhance the conductivity. We should carefully inspect the minerals which drastically change conductivity value of rocks. Though our results indicated the presence of small amount of conductive minerals by microprobe analysis, the total amount of the accessory minerals was low. Thus we can assume that the conduction paths of such a mineral in our sample are not established.

Glover & Vine (1992) and Duba et al. (1994) also addressed the problem of metamorphic rock sample collection from surface exposures. Although the granulite sample was collected on the surface of Hidaka metamorphic area in this study, it should be originated from the lower crust judging by its lithology. Results from geological investigation suggested that the granulite unit was metamorphosed at definite pressure and temperature. So we can use granulite sample because there is no distinct indication of chemical alteration or existence of saline fluids from the geological point of view.

There have been many studies on rocks saturated with fluid or containing conductive accessory minerals, which are intended to simulate the mid- to lower continental crust. However, in reality, the evaluation of laboratory measurement of relatively dry rock or homogeneous rock is still not sufficient. For instance, Wanna-maker (2000) pointed out the complex issues of dry lower crust and the role of natural fluid and mineral compositions in the lower crust concerned with individual tectonic setting. He emphasized distributions of large-scale conductive elements which are not represented in hand-specimen. Innumerable parameters can change the electrical conductivity of lower crustal rocks and, therefore, many conduction mechanisms must be considered. In the present study, we have sintered granulite rock sample and measured the electrical properties under the mid- to lower crustal P–T conditions. We need more measurements and data on unsaturated rock, saturated rock, and conductive materials in the rock, to understand the electrical conductivity.

### 5 CONCLUSIONS

The aim of the present laboratory experiments is to establish the techniques of electrical conductivity measurement of crustal rocks which show relatively low conductivity and have complicated mineral components. This approach can give evaluation of both the
Measurements of sintered granulite sample under mid- to lower crustal conditions. The most important result obtained in this experiment is the stable, reversible conductivity values up to 870 K at the fixed pressure of 1 GPa after conducting several experiments. In particular, above 500 K, our data are consistent with former experimental results of the exposed dry mid- to lower crustal rocks. It is also important to examine the quantitative elements analysis for evaluating the variation of oxidization and/or deoxidization states in the sintered sample.

Our experimental results can be compared directly with those of the present and former electro-magnetic soundings. The electrical conductivities of granulite under the mid- and lower crustal temperatures accord with the electrical conductivity structure suggested by MT data analysis.

In conclusion, before considering pore fluid conduction mechanism or the role of accessory minerals in the rock, the mechanism of dry or basic rocks should be examined and interpreted. Electrical conductivity of mid- to lower crust, derived from the laboratory data, is essential to the discussion of results of electro-magnetic soundings. The combining the results of electro-magnetic soundings and those of laboratory measurements will produce a clear and robust interpretation of the crust.

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