A procedure for stable electrical measurements on a rock sample against high contact resistance as a prerequisite for electrical tomography

Takeshi Suzuki (✉ suzuki.takeshi.38n@st.kyoto-u.ac.jp)
Graduate School of Science, Kyoto University  https://orcid.org/0000-0001-7886-7729

Ryokei Yoshimura
Disaster Prevention Research Institute, Kyoto University

Ken'ichi Yamazaki
Miyazaki Observatory, Research Center for Earthquake Prediction, Disaster Prevention Research Institute, Kyoto University

Naoto Oshiman
Disaster Prevention Research Institute, Kyoto University

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Abstract

An important geophysical issue is determining the resistivity of rocks under various conditions. To characterise the internal resistivity structure of a rock needs electrical data from many small electrodes attached around its surface. Effective measurement must overcome the high resistance of the rock itself and the high contact resistance on the rock's surface during measurement. Therefore, we developed a new method for stable, multi-point, electrical measurement on rock samples that is effective at high contact and sample resistance. The method employed conductive, adhesive epoxy electrodes, which strongly attached even to dry rock surfaces and provided high conductivity. Sustained current injection for long periods into high-resistance rocks was fulfilled using a constant direct current source with high internal resistance. Accurate voltage measurement across the high-resistance rock was accomplished by differential measurement using two high input resistance voltmeters. Measurements of high resistance also require a stable measurement environment: the temperature and humidity in the laboratory were controlled using an air conditioner, a humidifier, a dehumidifier, and a vinyl tent. Signal noise arising from human activities was eliminated by the remote operation of the measuring equipment and switching terminal. We assessed the precision and stability of our new method when applied to a dry granite sample at various levels of absolute humidity. The method recorded highly reproducible measurements under all absolute humidity conditions, thus indicating its validity. The observed changes of measured values with absolute humidity showed that atmospheric moisture greatly influences a sample's resistance and contact resistance, and indicate the importance of stabilising the temperature and humidity conditions in the laboratory when taking electrical measurements of dry rocks at room temperature and pressure. Additional electrical measurement of dry granite using a simple electrode array constituted the first step toward electrical tomography measurements. A 40-electrode array acquired the potential distribution on the granite's surface in response to injected current. Sample resistivity, which was sufficient to explain the measured potential distribution, was estimated by forward modelling. This demonstrates the robustness of our method, and indicates that it is potentially applicable to electrical tomography.

1. Introduction

Electrical resistivity estimated through geo-electromagnetic observations is crucial to understanding underground strata and their compositions. Its spatial variation qualitatively reflects the tectonic and geological setting. However, quantitative interpretation of the obtained resistivity and its spatial variation is not easy because subsurface resistivity is complexly affected by many factors. Its proper interpretation requires a good understanding of the rocks’ electrical properties. Therefore, electrical measurements of a variety of rock samples across a wide range of conditions and spatial scales are essential.

Previous studies have measured the resistivities of various rock samples in a variety of conditions. For example, Brace et al. (1965) measured the resistivity of granite saturated with salt and tap water at high pressure. Coster (1948) measured the resistivity of granite, gabbro, basalt, peridotite, gneiss, and eclogite at various temperatures up to 1,000 °C. Fuji-ta et al. (2004) studied the electrical resistivity of granulite at
1.0 GPa and 300–890 K, and Fuji-ta et al. (2007) investigated the electrical resistivity of gneiss at 1.0 GPa and up to 1000 K. However, most previous studies focused only on the bulk resistivity of rock samples. If estimating the internal resistivity structure of a rock sample were possible, heterogeneous structures such as fractures could show identifiable resistivity patterns at the laboratory scale. This information could be used to relate field-scale electromagnetic survey results with the composition and state of subsurface constituents. Therefore, not only measuring the bulk resistivity of a small sample, but also imaging its internal structure, is desirable for interpreting large-scale electrical resistivity structures.

In order to investigate the internal resistivity structure of a rock sample, the potential distribution on its surface must be measured with a sufficient spatial resolution by using multiple electrodes attached to it. For this purpose, electrodes must be small and the sample must be large in order to allow a measurement array to be applied without the electrodes overlapping. The small size of the electrodes and the large size of the sample inevitably increase the contact resistance and sample resistance, respectively, hampering stable current injection and voltage measurement. Therefore, electrical tomography is generally difficult. Some previous studies of electrical tomography (e.g., Borsic et al. 2005, Stacey 2006) measured only high porosity samples under high water saturation, and thus avoided the major difficulties of high contact and sample resistance. Stacey (2006) reported that electrical tomography can only be performed with sufficient water saturation to provide proper connectivity between the rock surface and electrodes. Establishing a reliable procedure for resistivity measurements involving high sample and contact resistances would increase the information available about the electrical properties of rocks by allowing electrical tomography of various rocks under various conditions.

There are some general requirements for the stable electrical measurement of rocks. First, the intensity of current injection must be kept constant. Second, the voltage must be measured precisely. Satisfying these two requirements is not easy, especially for high sample and contact resistances; therefore, a specific, optimised, experimental set-up is required. Third, the measurement environment must be precisely controlled. Electrical measurements of rocks are possibly affected by atmospheric humidity when conducted at ambient temperature and pressure; indeed, Okuyama (1972) and Alvarez (1973) reported that the resistance and resistivity of dry rock change greatly with the surrounding moisture conditions. In addition, the measurement system must be free from any mechanical or electrical disturbances, because only a subtle signal noise can seriously alter high-resistance electrical measurements using microcurrents. Possible disturbances include the movements of operators, including their use of instruments, and electrical noise from power sockets. These effects related to the measurement environment should be treated carefully.

This study aimed to develop a reliable procedure for stable multi-point electrical measurements on a rock sample with high contact resistance. To make the procedure compatible with as wide a range of measurement conditions as possible, the designed method was applied to an extreme experimental condition; that is measuring dry rock at ambient temperature (about 300 K) and pressure (about 100 kPa), under which the contact and sample resistances are particularly high.
The remainder of this paper is organised as follows. Section 2 enumerates the requirements for stable multi-point electrical measurements on a rock sample with high contact resistance. Section 3 proposes a measurement procedure that fulfils these requirements. Section 4 describes the experimental set-up to test the method’s performance. Section 5 assesses the stability of the measured values and demonstrates the efficiency of the proposed procedure. Section 6 examines electrical measurements of a dry granite sample with many electrodes in a simple configuration as a first step toward electrical tomography. Section 7 explains future works. Section 8 presents the conclusions.

2. Measurement Requirements

Stable electrical measurements with small electrodes on rock samples against high sample and contact resistance have the following requirements regarding the electrodes, current sources, voltmeters, noise reduction, and measurement environment.

The measurement electrodes should be strongly attached with high conductivity, even when electrodes of arbitrary shape are used on the surfaces of dry rocks. Previous studies have employed metal plate electrodes of silver, molybdenum, or brass (e.g., Collett 1959; Fuji-ta et al. 2004; Borsic et al. 2005). However, metal plates do not contact the sample unless the sample surface is sufficiently wet or under confining pressure. Even in experiments with wet samples, a porous filter paper is often placed between the wet sample and electrode to connect them with the fluid. It is necessary for electrodes to be attachable to dry rocks at room temperature and pressure.

Electrical current must be injected into a high-resistance rock for a long period during measurement. Previous studies have employed function generators for current injection (e.g., Fuji-ta et al. 2004). However, common function generators are incompatible with very-high-resistance samples due to difficulties in applying high voltage and controlling the micro-current.

The voltage across a dry, high-resistance rock should be measured accurately. The conventional multimeters used in previous studies are not suitable for measuring voltage at high resistance (over about 10 GΩ) due to their low internal resistance. The voltmeter must have an internal resistance far greater than the target resistance.

The measurement procedure should be designed to reduce expected noise in measurements at high resistance. This includes noise from the power supply, because even minute variations in the current have a large effect, owing to the injected current being very small in a high-resistance sample. Current leakage from insulation in the measurement circuit needs to be prevented. In high-resistance measurements, if the insulation and samples have similar resistance, then injected current would flow through both.

High-resistance measurement also requires a stable measurement environment with little variation in temperature, humidity, and other environmental conditions. As the current flow is very low, changes in the measurement environment can significantly affect measurements. While previous studies have focused
on the effects of only temperature (e.g., Kariya and Shankland 1983), at ambient conditions factors such as atmospheric moisture would also influence results.

3. Proposed Measurement Procedure

The current-injection and voltage-measurement techniques described here address the problems enumerated in the previous section regarding stable multi-point electrical measurements against high resistance.

Favourable properties in the measuring electrodes are achieved through using a conductive adhesive comprising uniformly dispersed conductive particles (e.g., silver) in an epoxy resin organic binder. The conductive adhesive allows arbitrary arrangements and shapes of the electrodes. Furthermore, it provides stable attachment for all surface types.

To achieve current injection at high resistance, the system for current injection and voltage measurement is designed as shown in Fig. 1; it is compared alongside a conventional measuring circuit. Our method uses a constant direct current source with high internal resistance in the same manner as Yamashita et al. (2014). There are two ways of finding resistance: measuring current under constant applied voltage, and measuring voltage under constant current. For very-high resistance samples, the constant-current method generates large voltage signals that provide more-accurate results than the very small currents recorded under constant voltage. This study uses direct current, because there are few alternating current sources that work with high resistance.

The proposed method ensures accurate voltage measurement by differential measurement using two voltmeters with high input resistance. In the resistance measurement of dry rock, insulation resistance between the negative terminal and chassis ground may be smaller than the sum of the sample and contact resistance, allowing current to leak into the negative terminal of the voltmeter. Even using a voltmeter with high input resistance (as used by Yamashita et al. 2014), current can leak with the conventional four-terminal circuit shown in Fig. 1(A), because the insulation resistance is usually much smaller than the input resistance. This problem is solved by shorting the negative terminal of the high input resistance voltmeter to the negative terminal of the current source. However, this short circuit causes the measured voltage to include a voltage drop by the contact resistance of the negative current electrode and potential fluctuation at the signal ground of the current source. This study uses the differential measurement shown in Fig. 1(B) to eliminate the above effects. The difference between the outputs of the two voltmeters yields the potential difference, while the signals common to both voltmeters cancel.

The differential measurement circuit has the voltage common to the negative terminals separated from that of the chassis ground to remove noise from the power supply. The guarding described by Tektronix (2016) is adopted to reduce current leakage from the measurement cables. It is applied to the wiring from the measurement instrument to the switch unit, and sets the shield of the coaxial cable at the same
potential as the inner conductor. When the shield is at the same potential as the inner conductor, there is no current flow between them. Therefore, this technique greatly reduces current leakage in the cable.

All measurements are performed with temperature and humidity kept as constant as possible using a humidifier, dehumidifier, and an air conditioner to reduce changes of sample resistance due to fluctuations in atmospheric moisture.

Human disturbance (e.g., vibration of the measurement circuit when attaching/detaching terminals and handling samples, and changes in temperature and humidity when staff enter or leave the laboratory) can cause strong signal noise; therefore, the proposed method employs remote operation of the instruments and a switching terminal to reduce signal noise arising from human activities.

4. Example Of The Experimental Set-up

The performance of the proposed method was assessed in a test measurement using the sample, tools, and instruments described here.

The cylindrical granite measurement target (52 mm diameter, 100 mm length; white granite from China) used in the test is shown in Fig. 2(A). Its ends were ground parallel, and the surface was not polished. This study used granite as a measurement target, because it is a typical rock component of the upper crust. As we hope to apply our measuring procedure to electrical tomography using dozens of electrodes, our procedure should be evaluated using a relatively large sample capable of hosting many electrodes without overlap. Before the test measurement, the granite sample was left in the laboratory at ambient temperature and pressure for at least one year.

The electrodes were conductive epoxy adhesive (CW2400 Epoxy, Chemtronics, Mansfield, England). At any size it can be expected to have high adhesion and good conductivity, even on dry rock surfaces.

Before attaching the electrodes, the rock’s cylindrical surface was masked by insulating masking tape, leaving gaps defining the electrode attachment area (Fig. 2(B) and (C)). This masking allowed precise control of the electrodes’ positions. Copper wire was attached to the epoxy adhesive to connect the measurement instruments. Table 1 lists the physical properties of the conductive adhesive.

A small square electrode area of 100 mm$^2$ was used in this test of our new measurement method. As the procedure was designed for future use in electrical tomography, the performance needs to be assessed using small electrodes.

This study used two instruments to inject constant direct current depending on the experimental purposes. The first was an electrometer (Model 6514, Keithley, Cleveland, Ohio, US.). It was used in resistance measurement mode to inject direct constant current and measure the resulting voltage; resistance measurement is by two-terminal measurement. The measured resistance value is the sum of the sample resistance and contact resistance. The constant amount of injected direct current was set
given the resistance range of the measured object (Table 2). The electrometer's maximum measurable resistance was 210 GΩ. The experiments in Section 5 used an electrometer, because it was necessary to measure the two-terminal resistance, to separate out contact and sample resistance, and to evaluate the dependence of the measurement environment on the sample and contact resistance.

The other instrument to inject constant direct current was a direct-current voltage/current source monitor (Model 6243, ADC; Saitama, Japan), which was able to set the amount of injected direct current to an arbitrarily large value up to 2 A at up to 32 V. Its maximum applied voltage was 110 V, and the minimum resolution of the injected current was 1 nA. The experiments in Section 6 used this device, because they required the greatest injected current possible and increased signal-to-noise ratio in order to facilitate the measurement of potential distribution. The measurement of potential distribution required measurement of not only the potential near the current electrodes but also the low potential values far from them. Therefore, increased current was necessary to amplify the potential, and this required an instrument capable of setting the amount of injected current.

The amount of injected current was measured by the ammeter of a multimeter (Model 3458A, Keysight; Santa Rosa, California, USA) with 1 pA resolution. Voltage measurement was with an electrometer (Model 6514, Keithley, Cleveland, Ohio, USA) set to voltage measurement mode. This instrument was also used for differential measurements. Its negative terminal was shorted with the negative terminal of the current source. In voltage measurement mode, the maximum input resistance is 200 TΩ, and insulation resistance between the negative terminal and the chassis ground is 10 GΩ. We electrically isolated the shorted negative terminal from the chassis ground to prevent the influence of ground noise. Each measuring instrument was connected to the power supply through the transformer to prevent noise from the outlet. In all the resistance and voltage measurements, guarding reduced leakage current.

Figure 3 shows the layout of the laboratory. The temperature in the laboratory was kept at 30 °C by an air conditioner during all measurements. Relative humidity was controlled with a dehumidifier (Model DM-10; Nakatomi, Nagano, Japan) capable of setting humidity in the range 30%–90% in 5% steps and a humidifier (Model HD-152, Dainichi, Niigata, Japan) programmable to 60%, 70%, or 80% humidity. The humidity condition was maintained throughout the laboratory, except the very high humidity condition, which was maintained only inside the vinyl tent. Temperature and relative humidity were recorded hourly by a temperature and humidity logger (Model LR5001, Hioki, Ueda, Japan) placed near the granite sample. The granite sample, thermo-hygrometer, and switching unit were placed on the same desk, while the granite sample was placed on insulating rubber plates to prevent leakage current. The granite sample that was subjected to analyses is shown in Fig. 2(A).

Terminal switching was performed by the switch unit (HP34970A, Hewlett-Packard; Palo Alto, California, USA). All measurements were controlled by LabVIEW software (National Instruments, 15.0). Operators did not enter the laboratory during the measurement period.

5. Stability Of Measurements
This section evaluates the stability of the proposed measurement procedure by considering the stability of resistances measured between the current electrodes, $R_{\text{measured}}$, the potential difference measured between the potential electrodes, $V_{P1} - V_{P2}$, and the measured current, $I$. The measurement method is correct in principle. However, inaccuracy in the set-up might lead to incorrect measurements that are expected to be unstable. Therefore, we can assess the validity of the measured values by checking their stability.

We checked the stability of measurement through six sequences of repeated measurements at six levels of relative humidity and constant temperature. In addition, we checked the adhesion performance through observations of the contact surface, because strong adhesion between electrodes and the sample is important in our experimental set-up.

5.1. Configuration of electrodes

Figure 4 shows the granite sample's cylindrical surface and the electrode arrangement with the measurement instruments. We observed the contact surface by micro-focus X-ray computed tomography (CT) to confirm the contact state of the electrodes. The CT results in Fig. 5 show that the electrodes were well attached, despite the roughness of the surface, thus demonstrating the strong adhesion achieved by the proposed method.

5.2. Stability evaluation procedures

5.2.1. Data sampling with humidity and temperature setting

$R_{\text{measured}}$, $I$, and $V_{P1} - V_{P2}$, were measured for 600 s in each measurement, which was repeated multiple times. Sampling was conducted every 1 s. To eliminate the charge between the current electrode and granite surface, all terminals were shorted after each 600 s measurement. The discharge time for each repeat measurement was set to 2 h during resistance measurements in the GΩ range. Resistance measurements in the MΩ range used a longer discharge time of 6 h due to the greater injected current amount (Table 2).

Six sequences of repeated measurements gathered data for $R_{\text{measured}}$, $I$, and $V_{P1} - V_{P2}$. One sequence is considered as a group of repeated measurement data performed at fixed humidity and temperature. The six sequences considered relative humidity at six set values (40%, 50%, 60%, 70%, 80%, and 90%) and constant temperature (30 °C).

5.2.2. Separation of sample and contact resistance from measured resistance

We used forward modelling to separate $R_{\text{measured}}$ into $R_{\text{sample}}$ and $R_{\text{contact}}$. This approach uses the potential difference between the potential electrodes, $V_{P1} - V_{P2}$, as the fitting parameter and the injected current, $I$, as the input parameter. Sample resistivity, $\rho_{\text{sample}}$, was estimated by grid searching with forward modelling, and it was converted to $R_{\text{sample}}$. 
The numerical calculation code developed by Suzuki et al. (2017) was used for forward modelling. This code is a transformation of the Dey and Morrison (1979) formulation into a cylindrical coordinate system. It calculates the static potential distribution in a cylindrical medium generated by a constant current. This study assumes the cylindrical calculation area to be a homogeneous structure with the same size as the granite target. Measured $I$ is used as the source constant current in the calculation.

At first, we estimated $\rho_{\text{sample}}$ as a fitting parameter by comparing the measured $V_{p1} - V_{p2}$ with values calculated using multiple $\rho_{\text{sample}}$ values for multiple potential distributions in cylindrical samples. Of the $V_{p1} - V_{p2}$ values subsequently extracted from these calculated potential distributions, the one most consistent with the measured value was selected. The corresponding $\rho_{\text{sample}}$ was selected as true.

Next, we determined $R_{\text{sample}}$ using Ohm's law. We extracted the potential difference between the current electrodes, $V_{C1} - V_{C2}$, from the potential distribution of the determined $\rho_{\text{sample}}$ substituted into Ohm's law. As the calculated potential distribution does not include the potential drop by $R_{\text{contact}}$, the value obtained by dividing the extracted $V_{C1} - V_{C2}$ by the measured $I$ is $R_{\text{sample}}$ not including $R_{\text{contact}}$.

This estimation process of $R_{\text{sample}}$ is shown Fig. 5. The validity of the process was confirmed by additional experiments (Additional file 1) using a cylindrical plastic measurement target (52 mm diameter, 100 mm length; MC501CD R2, Mitsubishi Chemical Advanced Materials, Tokyo, Japan) (Additional file 1: Fig. S1). Its resistivity was $10^0$–$10^2$ Ω·m at 23 °C. The experiments confirmed that sample resistivity can be determined precisely by our estimation process regardless of the arrangement of the current and potential electrodes.

We also obtained $R_{\text{contact}}$ from $R_{\text{measured}}$ to determine $R_{\text{sample}}$. Assuming that $R_{\text{contact}}$ of the positive electrode is the same as that of the negative electrode in the two-terminal measurements, $R_{\text{contact}}$ is given by

$$ (1) $$

In this study, $R_{\text{contact}}$ is determined by Equation (1) using the obtained $R_{\text{measured}}$ and the estimated $R_{\text{sample}}$.

5.3. Results and Discussion

5.3.1. Inspection and processing of time-series data

The stability of the measured resistance, current, and potential difference was assessed using time series of the data of the type depicted in Fig. 7 for 40% relative humidity and 30 °C. The time-series data show transient phenomena. The current recorded for about 1 min after the start of measurement was larger than that specified by the resistance meter (0.9 nA; Table 2). This large current meant that it took several tens of seconds for the measured resistance and potential difference to stabilise.
The large current at the start of measurement was interpreted as an inrush current. The increase of observed resistance after the current had settled probably corresponds to charging. Both of these effects shifted the measured resistance to higher values than their actual values. Therefore, it is reasonable to adopt the minimum value observed in each 600 s of resistance data, which likely includes the smallest effects of charging and inrush current.

On the other hand, after the inrush current, the current and potential difference became almost constant in the time-series data, making the data points equivalent. For standardised selection, the current and potential difference at the time of minimum resistance were chosen, as indicated by the dashed line in Fig. 7.

5.3.2. Stability of repeated measurements

Table 3 shows the stability of temperature and relative humidity in the six sequences. In each case, temperature varied by at most approximately 0.5 °C, and humidity varied by at most approximately 3%. The measured temperature and humidity were far more stable than those of the outside air.

Figure 8 and Table 4 show the results of repeated measurements in the six sequences and their statistical comparison, respectively. Figure 8(A) confirms that the specified current of 0.9 nA was injected correctly without leakage current. Because the current flowing through the ammeter is the sum of the injected current and the noise current, the observed current is larger than the specified current of 0.9 nA. Fluctuations of measured currents in each sequence were limited to about ±0.1 nA in the GΩ range and about ±1 nA in the MΩ range. We defined the fluctuation at each sequence as the standard deviation of all measurements in each sequence. The measured potential differences and resistances in each sequence fluctuated within about ±20% of the mean, except at 80% humidity. The potential differences and resistances considerably decreased with increasing absolute humidity, with measured resistance being especially sensitive to absolute humidity even within each sequence.

Figure 8 indicates the high reproducibility of the current, potential difference, and resistance measurements. The performances of the two- and four-terminal measurements are respectively demonstrated by the stability of the resistance values and of the potential difference and current values. Dividing potential difference by current gives the sample resistance between potential electrodes $P_1$ and $P_2$ without considering the sample shape or electrode position. Kariya and Shankland (1983) found standard deviations of 0.73–0.98 for log electrical resistivity at temperatures of 500–1000 °C for dry rocks of granitic composition and texture. In comparison, our results in Table 4 have standard deviations of log resistance ranging from 0.01 to 0.25 in each sequence. The standard deviation of log resistance (i.e., the quotient of potential difference and current) with error propagation is 0.08–0.38 in each sequence, except at 80% humidity. These results indicate the high measurement accuracy achieved here compared with previous studies. Furthermore, the statistics were calculated for each sequence in this study, but the standard deviation would become smaller if the average and standard deviation were calculated not by sequence but by narrow bins of absolute humidity.
All the results in Table 4 show the greatest standard deviation for the 80% humidity sequence. This was attributed to the sample resistance being close to the boundary between the GΩ range and the MΩ range at 80% humidity. When the sample resistance is below the lower limit of the set range, the voltage generated by the injected current becomes very small so that the resultant small current reduces the measurement accuracy. These results are expected to be improved by frequent switching of the measurement range when the sample resistance falls outside suitable limits.

5.3.3. Estimation of resistance between current electrodes and contact resistance

Figure 8 shows the high sensitivity of $R_{\text{sample}}$ and possibly of $R_{\text{contact}}$ to changes of absolute humidity. Figure 9 shows estimated values for $R_{\text{sample}}$ and $R_{\text{contact}}$. Not only $R_{\text{sample}}$ but also $R_{\text{contact}}$ greatly decreased with increasing absolute humidity, further showing the necessity of controlling humidity in the laboratory for measurements of dry rock resistance.

$R_{\text{contact}}$ was much larger than $R_{\text{sample}}$ and accounted for most of each $R_{\text{measured}}$ value in Fig. 8(C). This suggests that the area of the current path present on the electrode bonding surface is small with respect to the apparent electrode size: current appears to flow between the rock surface and the electrode only through part of the contact area observed by CT scanning.

Figure 8(C) confirms the dependence of $R_{\text{measured}}$ on absolute humidity, which is attributed to moisture absorption by the sample. Alvarez (1973) and Okuyama (1972) reported that moisture greatly changes the resistance and resistivity of dry rock. Alvarez (1973) concluded that the adsorption of water molecules to minerals changed the resistance of rock samples, as also suggested by the present results.

Linear fitting to the estimated results (Fig. 9) is used to investigate whether the relationship between absolute humidity and $R_{\text{sample}}$ can be expressed by a simple function. The estimated $R_{\text{contact}}$ appears mostly consistent with the fitting results, which implies the presence of an exponential relationship between absolute humidity and $R_{\text{contact}}$. We interpret the changes in $R_{\text{contact}}$ to reflect atmospheric moisture penetrating the contact surface and filling minute gaps between the electrode and rock surface, thus increasing the contact points. It is reasonable to assume that moisture adsorption would occur even at the contact surface. The observed exponential relationship suggests that the number of contact points on the contact surface changes exponentially with changes in humidity. The fitting function assumes an exponential relationship for $R_{\text{contact}} = e^{aH_A}$, where $C$ and $a$ are constants, and $H_A$ is absolute humidity.

6. Electrical Measurements Using An Electrode Array

We performed electrical measurements on intact rock using many electrodes in a simple configuration as a first step toward electrical tomography measurements. A constant current was injected into the sample, and the resulting potential distribution on the sample’s cylindrical surface was measured using an electrode array. Sample resistivity was determined by comparing forward modelling with the measured potential distribution and current.
6.1. Measurement procedure

6.1.1. Measurement set-up and procedure

Figure 10(A) and (B) shows a photograph and a schematic diagram of the cylindrical surface of the granite sample on which 40 electrodes were arranged for the tomography measurement.

During measurement, a constant direct current was injected. The electrical potential of each potential electrode was measured via terminal switching. The positive terminal of $V_1$ was switched among the electrodes by the switch unit, whereas that of $V_2$ was fixed during measurement. (Fig. 10(B)). Sampling was every 1 s. Electrical potential was measured for 600 s for each potential electrode. From this measurement, the potential distribution relative to the potential of the electrode connected to $V_2$ was obtained. The potential was ignored for the first 5400 s from the start of the current injection to avoid the effect of the inrush current. At the end of the sequence, we re-measured the potential at an electrode previously assessed at the beginning of the sequence to confirm that the potential did not temporally change. During the measurement, the temperature was kept at about 27 °C, and the relative humidity was kept at about 80%; therefore, the absolute humidity was kept at about 21 g/m$^3$.

6.1.2. Forward modelling

We determined $\rho_{sample}$ by forward modelling as described in Section 5.2. The modelling assumed the medium to have homogeneous resistivity. To consider fluctuations in intensity of the injected current, electrical potentials at each potential electrode were separately calculated by using the current intensity at the corresponding timing of observation. The value of $\rho_{sample}$ was determined so that it minimised the total difference between the measured and calculated potential at 38 potential electrodes, excluding the current electrodes.

6.2. Results and Discussion

6.2.1 Inspection and processing of time-series data

Figure 11(A-1) to (E-1) shows the potentials obtained in each z-line indicated in Fig. 10(B). The midpoint between the current electrodes was set as the reference point of the measured potential distribution for comparison with the numerical results. At each point, data obtained at seven timings (i.e., 60, 100, 200, 300, 400, 500, and 600 s) in the 600 s time-series were plotted: their invariance confirms the stability of the time-series data after the inrush current; nevertheless, we used the average of the last 100 s of the 600 s of data, as the end of the time-series is expected to have the least noise due to terminal switching. The current measurement similarly used the average of the last 100 s of the 600 s of data.

6.2.2. Obtained potential distribution and modelling results

Forward modelling giving results corresponding to the optimum value of $\rho_{sample}$ are shown in Fig. 11 (A-2) to (E-2). The potentials were calculated for injected current of an intensity set as the averaged
measured current for the corresponding 100 s of potential observation. The measured voltage values were normalised to values corresponding to the average current intensity.

All z lines showed consistent measured and calculated potentials, indicating the validity of the measurement. Electrical tomography has previously been considered to be possible only under water-saturated conditions (Stacey, 2006); however, the present results demonstrate that our procedure can perform tomographic measurements even for dry rocks.

7. Future Work

Our new method has promising future applications in tomography measurement, because its performance was verified using multiple very small electrodes. This work represents some progress toward electrical tomography of rocks, although further steps are required. Test measurements for samples composed of two materials with known resistivity will be useful for evaluating the detection accuracy on multi-component structures, although sample processing may encounter difficulties. Development of an inversion method for estimating the resistivity structure of cylindrical samples is also required, because the numerical calculation code (Suzuki et al. 2017) used in the forward modelling in the present work has not yet been extended to inversion modelling. These developments would make tomography measurements practically useful. An interesting target of electrical tomography is samples containing heterogeneous structures such as fractures. Compression tests of granite samples of the same size as used here have been widely performed, and to prepare samples with heterogeneous structures is not difficult. Comparison of electrical and CT measurement results may provide useful information about the electrical properties of heterogeneous structures. Previous studies have used CT imaging to investigate fracture distributions in granite samples (e.g., Kawakata et al. 1999).

8. Conclusions

We propose a reliable procedure for stable multi-point electrical measurements on a rock sample with high contact resistance. The method employs conductive epoxy adhesive electrodes to achieve secure attachment and high conduction on a dry rock surface. Stable current was injected into high-resistance samples using a constant direct current source with high internal resistance. Voltage was accurately measured in high-resistance samples via differential measurement with two high input resistance voltmeters. The problem of leakage current through the negative terminal of the voltmeter was solved by shorting it to the negative terminal of the current source. Contact resistance and potential fluctuation in the negative current electrode were eliminated by the differential measurement. Temperature and humidity in the laboratory were controlled using a humidifier, a dehumidifier, and a vinyl tent. Potential sources of signal noise from human activity were eliminated by using remote terminal switching and instrument operation.

We applied the new method to dry granite samples, and evaluated its precision and stability under multiple absolute humidity conditions. At each humidity, the measurements were highly reproducible,
thus indicating the validity of our new method. Nonetheless, atmospheric moisture did greatly influence the sample resistance and contact resistance, showing that humidity, alongside temperature, is an important environmental factor that must be controlled in the laboratory. Very high resistance exceeding 100 GΩ can be measured repeatedly by the new method even while using a small 100 mm² electrode.

We also performed electrical measurements on a dry granite sample using many electrodes as a first step toward electrical tomography. Current was injected stably for a long period. An array of 40 electrodes acquired the potential distribution on the rock’s surface caused by the injected current. The potential distribution calculated by forward modelling was consistent with the measured distribution, indicating the robustness of the measurement procedure and its potential capability for electrical tomography on high-resistance rock samples with high contact resistance (e.g., dry rocks).

**Abbreviations**

$H_A$: absolute humidity

$R_{\text{measured}}$: resistance measured between the current electrodes

$R_{\text{sample}}$: sample resistance between the current electrodes

$R_{\text{contact}}$: contact resistance at the electrodes

$R_{\text{IN}}$: input resistance

$R_V$: insulation resistance between the negative terminal and chassis ground

$V_{P1} - V_{P2}$: potential difference between the potential electrodes

$V_{C1} - V_{C2}$: potential difference between the current electrodes

$\rho_{\text{sample}}$: sample resistivity

$I$: injected current

**Declarations**

**Availability of data and materials**

The data that support the findings of this study are available upon request from the corresponding author.

**Competing interests**

The authors declare that they have no competing interests.
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Authors’ contributions

TS developed the measurement method, applied the method to samples, interpreted the obtained results, led the discussion, and wrote the first draft of the manuscript. RY, KY, and NO contributed to analysis and interpretation of data, and assisted in the preparation of the manuscript. All authors revised and improved the manuscript. All authors read and approved the final manuscript.

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Consent for publication

Not applicable.

Ethics approval and consent to participate

Not applicable.

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Tables

Table 1 Physical properties of conductive epoxy adhesive (Chemtronics CW2400)

| Operating temperature range | −91 to 100 °C |
| Volume resistivity           | < 10 μΩ·m     |
| Main component               | epoxy resin, silver, and hardener |
| Curing times                 | 5–10 min from 65–121 °C, 4 h at or above 25 °C |

Table 2 Accuracy specifications for resistance measurement by the electrometer (Keithley 6514)
| Range [Ω] | Resolution [Ω] | Direct injection current [A] |
|-----------|----------------|-----------------------------|
| $2 \times 10^6$ | $1 \times 10^1$ | $0.9 \times 10^{-6}$ |
| $2 \times 10^7$ | $1 \times 10^2$ | $0.9 \times 10^{-6}$ |
| $2 \times 10^8$ | $1 \times 10^3$ | $0.9 \times 10^{-6}$ |
| $2 \times 10^9$ | $1 \times 10^4$ | $0.9 \times 10^{-9}$ |
| $2 \times 10^{10}$ | $1 \times 10^5$ | $0.9 \times 10^{-9}$ |
| $2 \times 10^{11}$ | $1 \times 10^6$ | $0.9 \times 10^{-9}$ |

**Table 3 Stability of temperature and relative humidity in each sequence**

| Humidity setting [%] | Mean and standard deviation | Number of recordings |
|----------------------|----------------------------|----------------------|
|                      | Temperature [°C] | Humidity [%] |                  |
| 40                   | 29.4 ± 0.7       | 40.6 ± 2.1     | 136                 |
| 50                   | 29.3 ± 0.3       | 51.1 ± 2.5     | 67                   |
| 60                   | 29.3 ± 0.3       | 61.4 ± 1.6     | 87                   |
| 70                   | 29.4 ± 0.4       | 69.3 ± 2.5     | 79                   |
| 80                   | 29.5 ± 0.2       | 79.9 ± 2.5     | 190                  |
| 90                   | 30.3 ± 0.3       | 89.1 ± 2.8     | 116                  |

**Table 4 Statistical comparison of repeated measurements**
| Humidity setting [%] | Mean and standard deviation | Number of measurements |
|---------------------|----------------------------|------------------------|
|                     | Four-terminal measurement  | Two-terminal measurement |
|                     | Current [nA] | Potential difference [mV] | Log potential difference /current [Ω] | Log resistance ($R_{measured}$) [Ω] |
| 40                  | 1.05 ± 0.03 | 167 ± 14.4 | 8.20 ± 0.08 | 10.8 ± 0.01 | 6 |
| 50                  | 1.03 ± 0.04 | 90.5 ± 23.3 | 7.94 ± 0.23 | 10.4 ± 0.07 | 33 |
| 60                  | 1.04 ± 0.04 | 45.4 ± 12.8 | 7.64 ± 0.25 | 9.80 ± 0.16 | 43 |
| 70                  | 1.05 ± 0.04 | 26.8 ± 11.0 | 7.41 ± 0.38 | 9.16 ± 0.20 | 39 |
| 80                  | 1.08 ± 0.08 | 1.87 ± 6.02 | — | 7.89 ± 0.25 | 94 |
| 90                  | 937 ± 1     | 1150 ± 370 | 6.09 ± 0.29 | 7.41 ± 0.19 | 24 |

Note: The log quotient of potential difference and current in at 80% humidity is not calculable because the standard deviation of potential difference in the sequence is very large.

**Figures**
Figure 1

Circuitry of (A) the conventional four-terminal method and (B) the differential measurement method adopted here. The circled arrow is the direct current source, V and V1 and V2 are voltmeters, RIN is input resistance, RV is the insulation resistance between the negative terminal and chassis ground, Rc2 is the contact resistance between the electrode and sample surface, and Rs is the sample resistance. HI labels positive terminals, LO labels negative terminals, C1 and C2 are current electrodes, P1 and P2 are potential electrodes. In (A) when the sum of Rs3 and Rc2 is much smaller than RIN or RV, no current flows into the voltmeter. However, in a voltmeter, RV is usually much smaller than RIN. When RV is less than the sum of Rs3 and Rc2, current flows into RV through the negative terminal of the voltmeter, preventing correct voltage measurement. In (B), RV does not contribute to the current path, and when RIN is larger than the
sum of Rs3 and Rc2, most of the current does not flow to the voltmeter. The differential method uses two voltmeters, whose positive terminals are connected to P1 and P2, and whose negative terminals are shorted to the negative terminal of the current source. The voltage between P1 and P2 is obtained as the voltage difference between the measured values of V1 and V2.

Figure 2

Photographs of measurement set-up. (A) The unpolished, cylindrical rock sample (52 mm diameter, 100 length) overlaid with the dimensions r, θ, and z defining its coordinate axis. (B) The electrode attachment area as controlled by insulating masking tape. (C) Conductive epoxy adhesive attached to the rock surface as a high-conductivity electrode. A wire attached to the adhesive provided a connection for measuring instruments.
Figure 3

(A) Layout of measurement instrumentation and (B) photograph of the measurement system in the laboratory. Humidity was controlled using a dehumidifier and a humidifier. The vinyl tent was used only when making very high humidity conditions, otherwise each condition was maintained throughout the laboratory. A thermo-hygrometer placed near the sample recorded temperature and humidity. The granite sample, thermo-hygrometer, and switching unit were placed on the same desk. All repeated measurements were performed without direct human intervention, because manual terminal switching and instrument operation would cause serious signal noise. The switching unit performed the connection of terminals and electrodes during repeated measurements. The instruments and switching unit were controlled using LabVIEW software on the PC.
Figure 4

Measurement scheme. $z$ and $\theta$ are coordinates defined in Fig. 2(A). The circled arrow is a direct current source, the dotted square is a resistance meter, $V_1$ and $V_2$ are voltmeters, $A$ is an ammeter, $R_{IN}$ is input resistance, $R_V$ is insulation resistance between the negative terminal and chassis ground, HI labels positive terminals, LO labels negative terminals, $C_1$ and $C_2$ are current electrodes, and $P_1$ and $P_2$ are potential electrodes. In this study, a resistance meter was used as a direct current source. The resistance meter injects known direct constant current, measures the voltage caused by injected current, and obtains resistance. An ammeter (Model 3458A, Keysight; Santa Rosa, California, USA) was placed in order to monitor the amount of injected current. Electrometers (Model 6514, Keithley, Cleveland, Ohio, US.) with
RIN = 200 TΩ acted as the resistance meter and the voltmeter. Each measuring instrument was connected to the conductive epoxy adhesive used as electrodes shown in Fig. 2 (C).

![Figure 5](image)

**Figure 5**

Microstructures of the contact surface observed by micro-focus X-ray computed tomography (CT). (A) Sample photograph showing the 100 mm² electrode. CT images of the (B) x–z and (C) x–y planes; the pixel size is approximately 4 μm, and the scale bar in each image is 500 μm. Black in the CT images represents areas of X-ray transmission; the white areas are opaque to X-rays. Here, black represents mainly air, dark grey is mainly granite, and light grey is the conductive epoxy adhesive and wires. The images show roughness at the rock sample surface, and the good attachment of the electrodes to this rough surface.
Figure 6

Estimation of resistance between current electrodes. The coordinates $z$ and $\theta$ are as defined in Fig. 2(A). C1 and C2 are current electrodes, and P1 and P2 are potential electrodes. VC1, VC2, VP1, and VP2 are electrical potential at C1, C2, P1, and P2. $R_{\text{measured}}$, $R_{\text{sample}}$, and $R_{\text{contact}}$ are respectively the measured resistance between the current electrodes, the sample resistance between the current electrodes, and the contact resistance at the current electrodes. I is measured current. The left diagram depicts the measurement area (i.e., the sample's side surface), and the right diagram represents the numerical model of the measurement area with its calculation grids. Performing numerical calculations under various sample resistivities searched for a potential distribution that mostly explains the observed VP1–VP2. The calculations assumed the sample to have a homogeneous resistivity structure. VC1–VC2 is extracted from the determined potential distribution, and used to obtain $R_{\text{sample}}$ by dividing it by I.
Representative time-series data for resistance, current, and potential difference. The results are those measured for 600 s at 40% relative humidity and 30°C using the measurement layout in Fig. 3. (A) Resistance between C1 and C2 measured by a resistance meter. (B) Current measured by an ammeter. (C) Potential difference between P1 and P2. The injected current took several tens of seconds to stabilise after measurement started. It was initially larger than the specified current used by the resistance meter to measure resistance in the GΩ range, and was interpreted as inrush current. This inrush current caused the resistance also to take several tens of seconds to stabilise, after which resistance increased. This was interpreted as charging. The minimum in the resistance data for 600 s is therefore considered the most representative value, because the effects of inrush current and charging are likely smallest. Current and
potential difference were taken as their values at the time when resistance was lowest, as indicated by the dashed line.
Figure 8

Current, potential difference, and resistance with respect to absolute humidity. (A) Current measured by ammeter and (B) measured potential difference between P1 and P2 as a function of the absolute humidity. (C) Measured resistance between C1 and C2 as a function of the absolute humidity in log scale. Measurements were repeated in six sequences of relative humidity (40%, 50%, 60%, 70%, 80%, and 90%) under constant temperature (30 °C), with the symbols indicating the measurement sequence. Multiple measurements were performed for each sequence to confirm reproducibility. In each sequence, the minimum value observed in 600 s of resistance data was selected and plotted. The plotted current and potential difference are those at the time of minimum resistance. The injected current amount depended on the resistance measurement range (Table 2). The potential difference depended on the current amount.
difference. Dashed lines indicate the boundary of the measurement range. Each sequence of repeated measurement results shows high repeatability.

Figure 9

Estimated contact resistance and sample resistance with respect to absolute humidity. Resistances between current electrodes C1 and C2 are plotted as a function of the absolute humidity in log scale. The dashed line represents exponential fitting results, and the symbols indicate the measurement sequence. Sample resistance was estimated numerically from the measured current and potential difference values in Figs. 8(A) and (B). Contact resistance is half of the value obtained by subtracting the estimated sample resistance from the measured resistance value in Fig. 8(C). Both contact resistance and sample resistance decreased exponentially as the absolute humidity increased. Most of the resistance measured by the resistance meter appeared to be contact resistances. The relationship between contact resistance and absolute humidity was mostly represented by an exponential function.
Figure 10

Photograph and schematic diagram of the measurement set-up for electrical tomography. (A) Cylindrical rock sample (52 mm diameter and 100 mm length) overlaid with the dimensions \( r, \theta, \) and \( z \) defining its coordinate axes. Electrodes were attached to the cylindrical surface, and wires attached to electrodes provided a connection for measuring instruments. (B) Measurement scheme. \( z \) and \( \theta \) are coordinates defined in Fig. 10(A). The dotted square is a constant direct current source, \( V_1 \) and \( V_2 \) are voltmeters, \( A \) is an ammeter, \( R_{\text{IN}} \) is input resistance, \( R_{\text{V}} \) is insulation resistance between the negative terminal and chassis ground, \( H_1 \) labels positive terminals, and \( L_0 \) labels negative terminals. The direct current source (Model 6243, ADC; Saitama, Japan) injected a known constant current. An ammeter (Model 3458A, Keysight; Santa Rosa, California, US.) monitored the amount of injected current. Electrometers (Model 6514, Keithley; Cleveland, Ohio, USA) with \( R_{\text{IN}} = 200 \, \Omega \) acted as the voltmeters. Each measuring instrument was connected to the conductive epoxy adhesive used as electrodes as shown in Fig. 2 (C).
Figure 11

Profiles of electrical potential in the circumferential direction $\theta$ at each $z$ from tomographic measurements. The coordinates $z$ and $\theta$ are defined in Fig. 10. (A) Profile at $z_1 = 16.7$ mm, (B) at $z_2 = 33.3$ mm, (C) at $z_3 = 50$ mm, (D) at $z_4 = 66.7$ mm, and (E) at $z_5 = 83.3$ mm. Left panels (A-1 to E-1) show the measured value with elapsed time (60, 100, 200 s, etc.) from the start of the measurement at each potential electrode. The symbols indicate the data acquisition time. The reference point for the measured potential distribution used for comparison with the numerical results is the midpoint between the current electrodes. Right panels show numerical results of forward modelling (A-2 to E-2). The orange and blue dots respectively indicate the experimental and numerical results. In experimental results, error bars are smaller than the symbols. Each measured value is the potential at each electrode averaged over the last
100 s of measurement. The numerical results were calculated by using the average of all 100 s current intensity data at the corresponding timing of potential observation. The measured values were normalised by the average current for comparison.

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