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Characterization of the electrical response of geological samples as a function of uniaxial stress

Robert P. Dahlgren [1)], Vern C. Vanderbilt [2)], Malcolm J. S. Johnston [3)]
Greg G. Carlson [4)], Rebecca N. Nakaba [1)], and Friedemann T. Freund [1,2)]

1) *The SETI Institute, 189 Bernardo Ave., Mountain View, CA 95043, USA*

2) *NASA Ames Research Center, Moffett Field, CA 94035, USA*

3) *U.S. Geological Survey, 345 Middlefield Rd., Menlo Park, CA 94025, USA*

4) *Electric Machinery Co., Inc., 800 Central Ave. NE, Minneapolis, MN 55413, USA*

Electrical characterization of rocks as a function of stress is a commonly performed laboratory procedure in geophysics and petrology [1-7]. In this study a detailed set of experiments were performed to understand the deterministic and random noise of the experimental setup, and also increase the signal-to-noise ratio (SNR), stability, repeatability, and reproducibility of measured current and voltage data. Understanding of error sources, taking sufficient data to provide statistical confidence, and knowing the error bars are the hallmarks of quality data; and the requirement for the latter is no longer limited to experimental papers [8]. In one of the canonical experimental geometries, rock samples are bar or plinth-shaped, and have two or more conductive contacts. Uniaxial compressive force F is applied to a subvolume of the sample by external means, typically a hydraulic press. In most configurations a stack of metal platens and thin polymer insulators are employed to electrically isolate the sample and electrical contacts from the external system, and current is measured with a picoammeter or voltage is measured with an electrometer. Several aspects of the experiment were evaluated and optimized: (1) sample geometry; (2) load cycling; (3) mechanical alignment; (4) platen material and design; (5) insulator material; (6) electrode design; (7) instrument setup; and (8) electrical discharging.

The bar-geometry was selected for this series of experiments because it is commonly used and the shape allows straightforward calculation of resistivity and other parameters if it is approximated as a 1-dimensional problem. A standardized geometry facilitates comparison of different samples of the same and different rock types. Additionally, the symmetry of the shape permits the design of further experiments such as the effect of force as a function of position along the sample, and various differential experiments.

During the mechanical optimization of the experiment, a major source of non-repeatability was quickly identified in the form of random signal transients or offsets. These occur as a result of microcracking at high stress and during macroscopic movement of the sample in the hydraulic press at low stress. The latter effect was particularly strong during the transition from $F \leq 0$ to $F > 0$ and vise-versa, because the sample is cantilevered and thus mechanically unstable at low force levels. Once this was understood, it was easily mitigated by using a non-zero "pedestal" force on the order of 10 kN to always clamp the stack together. After this technique was adopted, repeatable current and voltage measurements as a function of force could be made on the same sample. Repeatability was the highest for the second load cycle and beyond, if the maximum force of the first load cycle was not exceeded and during the run there were no irreversible events such as creep or microfracturing. These effects were reduced by constraining the applied force such that the peak stresses were well below the fracture strength of the rock and yield strength of the insulators.

Improvements to the test fixture, tools, and best practices were also carried out as part of this study. To perform a quick instrument calibration a precision 100 MW resistor connected in series with an Alkaline AA battery provided ~16 nA of current in a known direction. This simple device also served to double-check the sign of the recorded signal and proved invaluable for debugging bad connections and cables. An ersatz geological sample made from an aluminum bar and a 50 MW series resistor was used to evaluate electrostatic interference and characterize insulator materials. While it was known that insulating materials can cause spurious signals [9], it came as a surprise that the happenstance choice of aluminum for the control sample and platens had a dramatic influence on the recorded data. Therefore, a set of precision, polished platens were fabricated from 1018 low carbon steel to correct this issue and the recently-adopted aluminum platens and control sample were retired.

Continued investigation of the polymer insulators with the steel control sample identified deformation-induced (and therefore force-dependent) charge injection between the insulators and the sample. To the extent the time rate of change of this charge is coupled to the instrumentation, it is observed as a unipolar current transient ~ 1 nA whose sign is dependent on the sign of dF/dt. Similarly, an electrometer may observe an artifact from the experimental setup, in this case it is a voltage offset from the baseline voltage when force applied. These error signals are exacerbated by inadvertent tribological charging or transfer of charge from the human body during the assembly of the experiment, and are reduced by discharging the system before testing. It is difficult to mitigate this effect completely because of the high resistivity coupled with stored-charge effects in polymers. Replacing the high-density polyethylene (HDPE) insulators with ultra-high molecular weight (UHMW) polyethylene (PE) reduces stored-charge effects due to decreased free volume in the bulk material. However, the upgrade to UHMW PE reduces but does not eliminate the unipolar artifacts. The optimized steel platens and UHMW PE generated unipolar transients with a magnitude of 6.4±0.8 pA with the steel control sample (approximately 150× reduction).

A number of electrical contact designs and approaches have evolved over the years [1, 10] and for this study adhesive-backed copper tape is employed. Although this material is convenient to cut and solder, drawbacks include poor adhesion on wet samples, poor corrosion resistance, and low operating temperature. When metallic contacts are applied to a geological sample, an EMF of a few hundred mV DC is routinely observed, even for samples not under stress and after a long discharge time. This long-known effect arises from a combination of Seebeck effect, contact potentials, incomplete dielectric polarization relaxation, self-potential (SP), electrochemical effects, sample isotropy, and so on. During measurement this EMF creates a baseline that is quasi-static or monotonically changing as a function of time, independent of force. Electrode contact resistance and contact potential are strong functions of sample material, pressure, burnishing, and sample cleanliness. For some electrode topologies force-dependent current and voltage instability was observed possibly arising from changes to the thickness and effective area as the adhesive was driven into the pores of the rock. The solution is to move the copper tape from under the insulators and platens to the endfaces of the rock where force-dependent perturbations are considerably reduced. This removed a major source of force-dependent error signals, provided one contact is kept as close as possible to the stressed subvolume as indicated by finite-element modeling [11]. The gain of contact stability and symmetry is a tradeoff made at the expense of contact size and shielding of the insulators.

Geological samples that contain pore fluids have a markedly lower resistance than their dry counterparts. In order to permit measurement and comparison of hydrosaturated samples to dry samples, it is necessary to present the instrumentation with similar source impedance values. The solution for aqueous or brine saturated rock samples is to add series resistance, employing an external fixed resistor to raise the effective impedance to a higher value in the range of $10^6 \sim 10^8$ ohms.

In conclusion, through a series of systematic rock-stressing experiments, confounding signals and experiment artifacts were identified along with mitigating approaches, resulting in an optimized experimental setup and test protocol. Noise levels were reduced to < 2 pA p-p and < 2 mV p-p when using the steel control sample. For typical rocks, e.g. fine-grained gabbro, the random noise levels increased to ~ 10 pA p-p and ~ 6 mV p-p. It is speculated that this is due to contact instability, SP-electrode instability, microfracturing, and other non-deterministic sources. Environmental sources were also troublesome: on some days significant electromagnetic interference (EMI) of unknown origin made measurements impossible. Even with ideal shielding this kind of experiment is fundamentally limited by compression-induced voltages and currents arising from the finite stiffness of the polymer insulators. The understanding of experimental error sources and increased SNR described above can only help to improve the robustness of inferences made from these types of data. Future work will model the underlying physics of the experimental setup, which may yield a methodology to remove the spurious transients and offsets from the experimental data.

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