CONDUCTIVITY MEASUREMENT TECHNIQUES AND METHODS: AN EMPIRICAL STUDY

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ABSTRACT

Strategies for estimating dc and ac electrical conductivity of single precious stone are displayed in this part. DC electrical conductivity can be estimated utilizing electrometer and ac conductivity and dielectric properties of the precious stone can be estimated utilizing an impedance analyzer. A kiethley programmable electrometer (model 617) is utilized with internal hotspot for dc measurements and Hioki impedance analyzer (model 3532), having frequency run 42 Hz to 5 MHz, is utilized for the ac conductivity measurement. The conductivity cell utilized for these measurements is talked about in detail. Strategy for growing a single gem is additionally talked about. For the development of the gem, a constant temperature shower with carefully programmable temperature controller is utilized. Since conductivity and dielectrics are anisotropic properties that ought to be estimated every conceivable way, expansive single crystals ought to be developed. Developing huge single precious stone with optical quality is a monotonous procedure. Another goal is the distinguishing proof of the crystallographic planes. The crystallographic planes can be recognized utilizing a notable strategy named "stereographic projection". In this proposal a point by point portrayal is given, how the crystallographic planes are distinguished utilizing Stereographic projection. This can be cross-checked by a PC program "Shape".

1. CONDUCTIVITY MEASUREMENT METHODS

Conductivity measurement has across the board use in mechanical applications that include the measurement of conductivity on materials, for example, metals, crystals, amorphous materials and so forth. The unit of conductivity is Siemens/cm (S/cm), which is indistinguishable to the more established unit of mhos/cm. In this area, techniques for getting conductivity data on crystals are depicted. The utilization of conductivity measurements in inquire about work is vital, and numerous incredible accounts of different measurement methods are now accessible. Different strategies have been utilized to gauge conductivity properties. In this chapter the electrical properties of some glaserite crystals are considered.

Dc Electrical Conductivity Measurement

The conductivity of a material is estimated regarding its resistivity. Resistance is frequently estimated with a computerized multimeter. Resistance in the gigaohm and higher extents must be estimated accurately. These measurements are made by utilizing an electrometer, which can gauge both low present and high impedance voltage. Two techniques are utilized to quantify high resistance, the constant voltage strategy and the constant current technique. In the constant voltage technique a known voltage is connected and electrometer ammeter is utilized to quantify the subsequent current. In the constant current strategy, a constant current is constrained through the precious stone and the voltage drop across the gem is estimated.

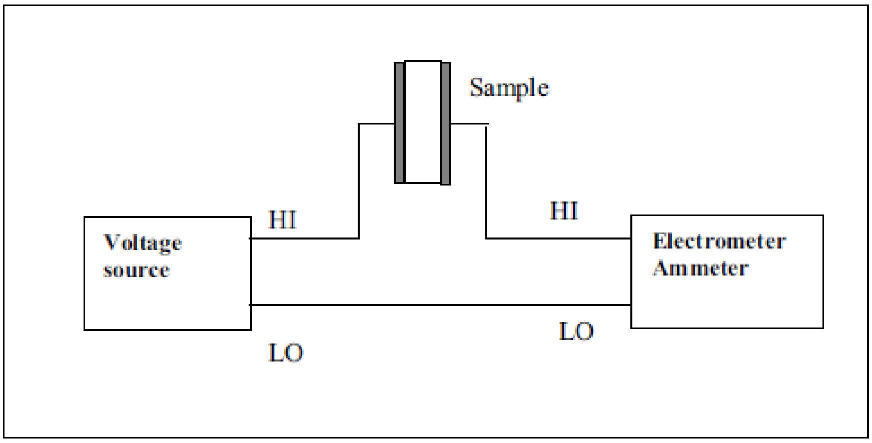

Figure 1: Resistivity measurement strategy

The essential Figure of the constant voltage strategy is appeared in Figure1. In this strategy a constant voltage is connected in arrangement with the precious stone sample and an electrometer. Since the voltage drop across an electrometer is irrelevant, basically all voltage shows up across the gem sample. The subsequent current is estimated by the electrometer and the resistance is computed utilizing the Ohm's law. The resistivity is Figure 1 from the geometry of the electrode and the thickness of the sample. For accurate measurements, the high impedance terminal of the electrometer is constantly associated with the high impedance purpose of the circuit to be estimated. If not, wrong measurements may come about. This test technique is depicted in detail in ASTM.

Alternating Current Scaffold Technique

Exchanging current measurements are generally used to beat certain troubles in dc measurements. Among these are polarization impacts in ionic conductors and electrolytes, boundaries at internal surfaces and contact resistance. Accepting that the sample is spoken to by a parallel mix of capacitance and resistance, the values of arrangement resistance and capacitance at that point depict the obscure straightforwardly. Current supply for the scaffold is frequently an ac oscillator or signal generator with frequencies from 20Hz to 20MHz. In the easiest course of action as appeared in Figure 2 (a 2-electrode cell), a voltage is connected to two level plates submerged in the arrangement, and the subsequent current is estimated from Ohm's Law, the conductance = current/voltage. Actually there are numerous practical challenges. Utilization of dc voltage would soon drain the particles close to the plates, causing polarization, and a higher actual resistance. This can be for the most part overwhelmed by utilizing ac voltage, yet all things considered the instrument architect must right for different capacitance and different impacts. Present day refined 2-electrode conductivity instruments utilize complex ac waveforms to minimize these impacts.

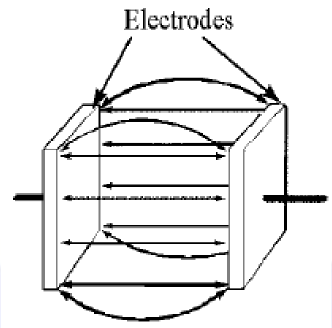

Figure 2: A simple two-electrode cell.

The ac impedance technique can be connected additionally to ionic conducting materials giving more data about the idea of conductivity. In this work there is likewise another preferred standpoint in utilizing the ac impedance strategy: in light of the fact that the samples were sliced into particular bearings to quantify the conductivity toward that path. Because of generally little and about rectangular (1 mm x 1 mm x 2 mm) samples, the most advantageous approach to gauge the conductivity is to place the sample between two electrodes.

The weakness in the ac impedance system is because of the blunders caused by the contact resistance between the electrodes and the sample. In any case, when moderately high frequencies are utilized, (for example, 1 KHz) the impact of the contact resistance is irrelevant.

In ac conductivity measurements a sinusoidal voltage is connected and the present going through the sample is estimated. Accordingly impedance Z* is a mind boggling introduction of the ratio of voltage and current maxima (Figure 3a)

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$$Z^* = V_{max}/I_{max}$$

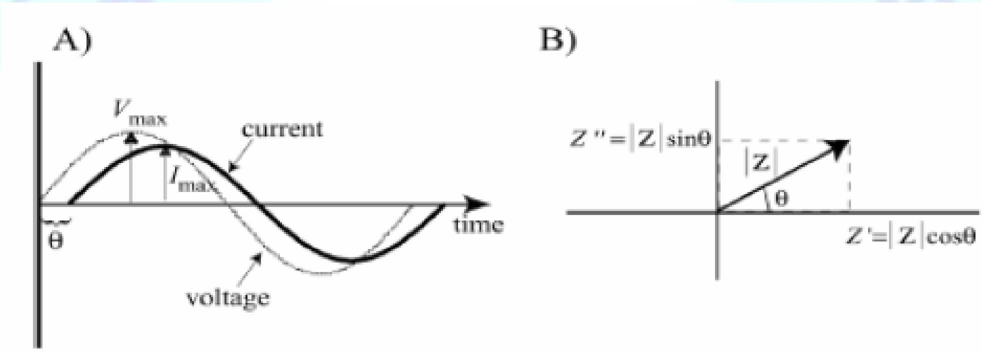

Figure 3: a) In the ac impedance method a sinusoidal voltage is applied and the current passing through the sample is measured. b) Impedance spectrum presented in complex plane. Z' and Z'' represent the real and imaginary components of impedance Z*.

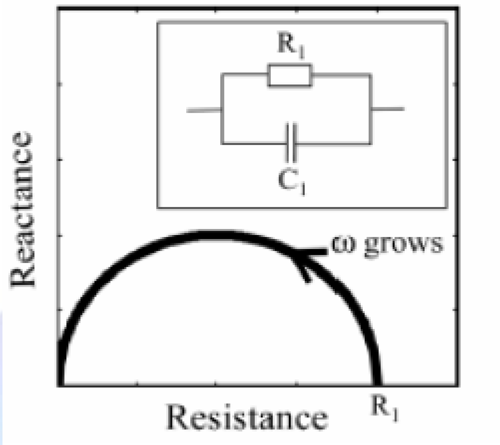

Figure 4: The Zc, Zcc or resistance-reactance bend for a resistor and capacitor associated in parallel. The bolt demonstrates the bearing of the expanding frequency.

The outcome may likewise be spoken to in an intricate plane with Z* = Z'-jZ" (Figure 3 b). In the ac impedance measurement the impedance is estimated as an element of frequency. The impedance spectrum is commonly introduced in the Z'-Z" - complex plane. It is conceivable to build an equivalent electrical circuit comprising of resistors and capacitors, which have an indistinguishable frequency reaction from the deliberate material (for instance Figure 4). These equivalent circuits give additional data on the conduction forms in the material.

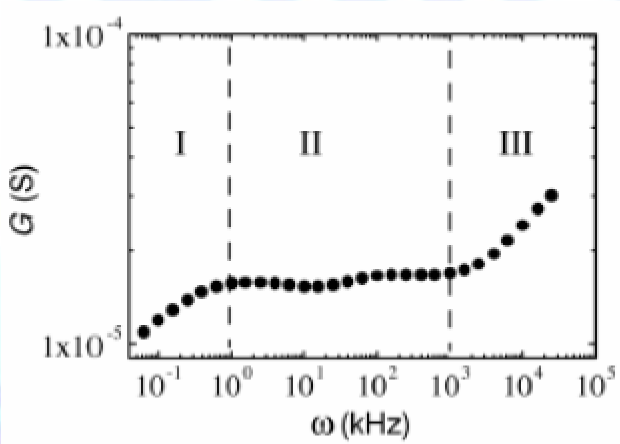

Figure 5: A case of conductance, G, as an element of frequency.

It is conceivable to recognize three administrations from the conductivity versus frequency plots (Figure 5). The primary administration is in the low frequency I and can be credited to the electrode polarization. In the center frequency administration II the conductivity is frequency independent comparing to dc conductivity. In the high frequency administration III the conductivity versus frequency plot complies with Jonscher's Universal Power law.

2. THE CONDUCTIVITY CELL

For the measurement of ac and dc conductivity a conductivity cell was utilized. The conductivity cell was composed such that, it should well fit into the temperature circulator shower which was utilized as the temperature controller (Julabo, Labortechnik GmbH, Germany, model FP 50). The metallic external case is 22 cm long. The best cover is removable and is made vacuum tight by utilizing an elastic "O" ring of 10 cm diameter. BNC pins are settled in this top for four-probe measurement. The base of the external vessel is brazed to a thick copper plate keeping in mind the end goal to get great warm contact with the shower liquid. A metallic tube is welded at the external case, and can be associated with the vacuum chamber so the cell can be evacuated. The sample holder is embedded through the highest point of the cell.

The sample holder comprises of four little strung poles with plates settled to these bars utilizing nuts. The base plate is made of copper with the goal that a decent warm contact can be accomplished when it is placed in the conductivity cell. The sample is placed in the middle of two graphite electrodes, all around protected from the external chamber. The sample alongside electrode is held in the middle of two glass plates with help of a spring-stacked arm settled at the highest point of the sample holder. A thin film platinum resistor Pt 100, model CRZ 2005 (Hayashi Denko Japan) is embedded into the copper plate by penetrating a little gap in it. The adjustment in the resistance with temperature was noted. The temperature coefficient of the RTD is certain. A guess of the platinum RTD resistance change over temperature can be ascertained by utilizing the constant 0.00385:/0C.This constant is effortlessly used to ascertain the outright resistance of the RTD at any temperature.

$$RTD(T) = RTD0 + T \times RTD_0 \times 0.00385$$

Where RTD (T) is the resistance value of the RTD component at a temperature T^oC, RTD_0 is the predetermined resistance at 0^oC and T is the surrounding temperature of the RTD. The value of RTD0 for a Pt 100 platinum resistance is 100.

3. DC CONDUCTIVITY MEASUREMENTS

The dc conductivity of the sample was estimated utilizing a Keithely (model 617) programmable electrometer with an internal source. In this setup the sample is considered as a resistance. Across the sample a voltage is connected and comparing current was noted. Utilizing Ohm's law the resistance R was ascertained. From the resistance the resistivity U was discovered utilizing the connection R = Ul/A where an is the territory of the sample and l is the thickness. The conductivity is the equal of resistivity.

4. AC CONDUCTIVITY MEASUREMENTS

Ac conductivity is given by

$$\sigma_{ac}=\omega\, \varepsilon_0\, \varepsilon$$

Where ω is the angular frequency, $\omega=2nf$

ε_0 = is the permittivity of free space,

ε^n= is the imaginary part of dielectric constant $\varepsilon^n = \varepsilon' \tan\delta$

Where tan δ is the loss tangent related to the phase angle θ,tan δ = [1/tan θ]

ε' is the real part of dielectric constant, $\varepsilon' = Cd/A\varepsilon_0$

Where C is the capacitance, d is the thickness and A is the area of the sample. Capacitance C and the stage angle T are quantifiable quantities. At that point air ac conductivity is

Then ac conductivity is

$$\sigma_{ac}=2nf\, \varepsilon_0\, \varepsilon' \tan{}_{\delta}$$

Conducting an Impedance Spectroscopy (Is) Experiment

The accompanying insurances must be taken while doing the impedance spectroscopy (IS) experiment. When directing IS at lifted/decreased temperatures the sample holder must be steady at the working temperatures, which can fluctuate from - 30qC to 180qC. Additionally the holder must be electrically idle and non-generative of any misleading streams or voltages. The holder must be intended to hold the sample at an even weight and with no development amid the experiment cycle. The sample holder must connection the sample to the electronics, producing the flag and examining the reaction. Every single electrical association are made utilizing protected wires and kept to the base conceivable length. The phone contraption ought to be completely electrically protecting. This will prepare for outside inductive impacts. Such impacts can significantly affect the outcomes by changing the current and voltage delivered by the sample. The leads utilized were of comparable length to diminish contrasts in the protection or any capacitance impacts, and ought to be short as could be expected under the circumstances. The entire framework was adjusted utilizing known resistors to lessen the effects of any inner capacitance because of wiring or associations.

The exact control of temperature is imperative when playing out an IS experiment. For this a Refrigerated Circulator Bath (Julabo GmbH, Germany, display FP 50) having temperature range from – 50°C to + 200°C with temperature exactness 0.01oC was utilized. Since the experimental temperature ranges from 30 o C to 150 °C, transformer oil was utilized as fluid in the shower.

Conductive graphite glue is covered on either side of the precious stone sample before mounting the sample in the graphite terminal. This will guarantee that the territory of the terminal is the compelling region of the sample precious stone just and subsequently decreasing any air capacitance. Silver glue can't be utilized in light of the fact that silver glue can diffuse into the precious stone grid, which will influence the conductivity estimations. Figures8 and9 demonstrates the photos of the dc conductivity estimation setup and a nearby perspective of the electrometer utilized

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Figure 6: DC conductivity estimation setup.

Figure 7: Close up view of keithley electrometer.

Figure 8: AC conductivity and dielectric measurement setup.

Figure 9: Close up perspective of LCR meter.

5. CRYSTAL CUTTING AND POLISHING

Subsequent to recognizing the crystal faces the mass crystal has been cut utilizing a moderate speed diamond wheel saw. This diamond saw comprises of a thin metal cutting edge with micron measured diamond powder inserted on the surface of the plate. The sharp edge is settled to a pole driven by a speed controlled engine. The crystal to be cut is settled (by sticking it) to an accuracy versatile arm. This portable arm contains the goniometer, micrometer and stabilizer arrangements. The arm can be brought down with the goal that the crystal lays on the pivoting sharp edge. The photo of the crystal shaper is appeared in Figure10. A nearby perspective of the goniometer of the crystal shaper is appeared.

Figure10: Photograph of the crystal polishing unit.

Parallel countenances can be cut by altering the micrometer screw without aggravating the stuck crystal. The cut samples have been cleaned well. For this a crystal-cleaning unit is utilized which comprise of an all around confronted circular circle made of stainless steel. This plate is appended to a pivot component driven by a speed controlled engine. A removable circular glass plate can be joined over the steel circle with the goal that diverse evaluations of grating paper can be stuck on to a circular glass plate for better outcome. At long last the crystal is cleaned well utilizing a cerium oxide powder. Crystals with great optical quality have been readied utilizing these techniques.

6. CONCLUSION

Expansive single crystals are important for the electrical conductivity and dielectric estimations, since these properties are measured every conceivable way. These crystals are not promptly accessible and thus they are developed in the laboratory by moderate evaporation strategy at steady temperature as a piece of the present work. Crystal development contraption has been manufactured and a carefully programmable temperature controller having an accuracy of ± 0.05 K was used to keep up steady temperature of the shower. The developed crystals have been cut by moderate speed diamond saw in the wake of recognizing the crystallographic headings to acquire tests for examination. The Obtained tests were cleaned well to optical quality.

REFERENCES

[1]. Goswamy and A. P. Goswami, *Thin Solid Films***16**, (2013).

[2]. A.E.Hamed, Journal of Materials Science Letters, **20**, 1533- 1536, (2001).

[3]. K. Jonscher, A new understanding of the dielectric relaxation of solids. *J. Mat. Science*,**16**, 2037, (2011).

[4]. K. Jonscher, L. A. Dissado, and R. M. Hill, Comments on many-body dielectric relaxation in solids. *Phys. Stat. Sol.* **102**, 351- 356, (2010).

[5]. P. Q. Mantas, *Journal of the European Ceramic Society*,**19,** 2079-2086, (2009).

[6]. M. Kleitz, and M. C. Steil, Microstructure blocking effects versus effective medium theories in YSZ. for ion conductors *Phys. Stat. Sol (b)*,**17**, 819- 829, (2007).

[7]. N. S. Hari, P. Padmini, and T. R. N. Kutty, Complex impedance analyses of n-BaTiO3 ceramics showing positive temperature coefficient of resistance. *J. Mat. Science.: Mat. Elect***8**, 15-22, (2007).

[8]. A. R. West, N. Sinclair, and N. Hirose Characterization of electrical materials, especially ferroelectrics, by impedance spectroscopy. *JElectroceram*.**1(1**), 65-71. (2007).