

ELECTRICAL RESISTIVITY MEASUREMENTS: A REVIEW

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World-wide interest on the use of ceramic materials for aerospace and other advanced engineering applications, has led to the need for inspection techniques capable of detecting unusually electrical and thermal anomalies in these compounds. Modern ceramic materials offer many attractive physical, electrical and mechanical properties for a wide and rapidly growing range of industrial applications; moreover specific use may be made of their electrical resistance, chemical resistance, and thermal barrier properties. In this review, we report the development and various techniques for the resistivity measurement of solid kind of samples.

1. Introduction

Electrical resistivity is one of the most sensitive indicators of changes in the nature of the chemical binding. In general, the electrical resistivity is inversely proportional to the carrier density and the carrier mobility. A change in the nature of the chemical binding primarily alters the carrier density, and the structural changes alter the carrier mobility. Very early investigation of metals showed that resistivity increases approximately by a factor of 2 at the melting point, while it decreases in silicon and germanium as they transform from semiconducting solids to conducting liquids. Electrical resistivity plays an important role in technical applications.

There is much interest at present for the study of ceramic materials. Since the realization of electrical and thermal conducting properties of these materials, there have been extensive investigations in to the transport properties of these compounds. The significant progress of ceramic materials has been attaching attention of a lot of scientists in various disciplines and encouraging their entry into field. The synergy of diverse scientific senses brings further spread of the study of these materials.

Recent technological improvements in measurement instruments and technology have opened up the way for new methodological applications and have led to new discoveries in the field of material science. There has been a great progress in the synthesis of a variety of inorganic colloidal Nano crystals (NCs) with highly controlled size and shape in the last decade¹. This achievement has promoted comprehensive researches on the fabrication and engineering of NC devices with novel structure and function with the aim of utilizing them in a wide area of applications, including electronics and optoelectronics, by using various NC assembling and patterning methods. However, colloidal NCs having

been extensively investigated so far are mostly metals, semiconductors and magnetic materials, and only limited investigations on oxide NCs from a point of view of NC ceramic engineering have been done.

Determination of the temperature dependent electrical resistance is a very important tool to explain the nature of ground state, phase diagrams, electrical, electronic and magnetic properties and instabilities observed of the studied materials for this kind of developments.

2. Electrical Resistivity Measurements

Various models and methods have been suggested to measure the electrical resistance. Factors affecting the suitability of various methods and precision attainable include contact resistance and shape of the sample i.e. whether is it in the form of single crystal, thin film, powder pellet or small crystallite. Among the methods to be discussed here, are two probes (ohmmeter or voltmeter – ammeter measurements) can be used for higher resistive samples and four probes methods (potential probe measurements) for the low resistive and single crystals. Whereas, Montgomery, van der Pauw and Smith techniques for the pellets and bulky samples.

The use of resistivity measurement in research work is old and many excellent accounts of various measurement techniques are already available²⁻⁹.

2.1. Two probes measurements

This is the simplest method of measuring resistivity and is illustrated in fig.1. In this method, voltage drop V across the sample and current through the sample I are measured. Then the resistivity is given as

$$\rho = \frac{VA}{IL} \quad (1)$$

This method is useful when the sample has large resistance.

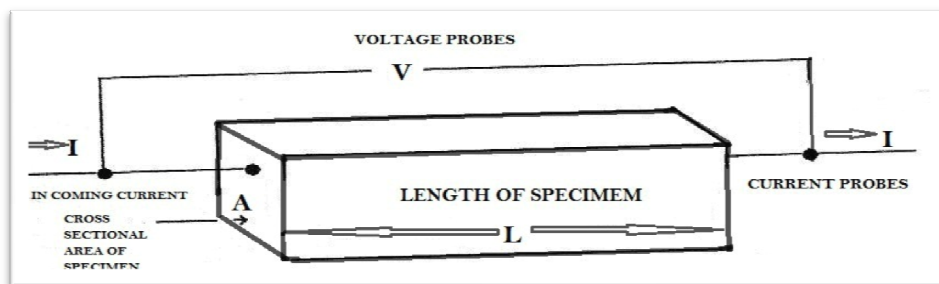


Figure 1: Electrical resistivity measurement by two probe method.

2.2. Four probes measurements

The potential probe is the most widely used method for resistivity measurements on the low resistive samples. In this method the potential drop is measured across two probes and distance between these probes D replaces the sample length L in equation 1. When the probes are not point contacts, in that case, the most accurate value for the probe distance is the distance between the centers rather than the closest distance between the probes. Fig. 2 shows the schematically arrangement for this method. In this case ρ is given by

$$\rho = \frac{V_D A}{D I} \quad (2)$$

Four probe method can be used to determine the resistance of the single crystal as well as the bulk specimen also. Here current passes through the outer contacts which are close to the edges of the sample. The potential difference is measured across the inner contacts. This method can eliminate the effects of contact resistance between the sample and electrical contacts and therefore is most suitable for low and accurate resistance measurements.

Contact and lead resistances are cancelled out by the four point method, however the contact resistance can still cause error if these are produce enough heat. Thus it is imperative that the contacts should have low resistance. Instrumental dc offsets also contributes to the error, but this can be easily corrected by subtraction. Self-induced voltage offsets in the circuit further add to the error. This problem can be corrected by reversing the flow of current through the sample. When the low level of the voltage (in the range of μV) is produced across the sample, signal noise also adds to the error. By using the proper shielded cables and low thermal contactors, as well as making single point grounding, noise problem can be reduced.

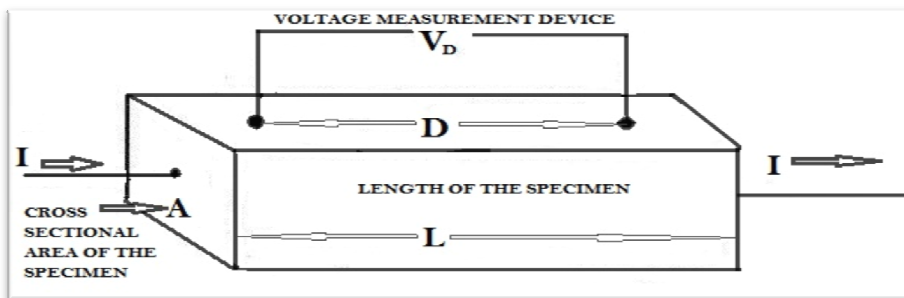


Figure 2: Electrical resistivity measurement by four probe method

2.3. Four point probes measurements

The 'four point probe' method has proven to be a convenient tool for the resistivity measurement of small size (of the order of mm) specimen. This method is applicable when the distance between the probes is small compared to the smaller dimension of the sample, and provided none of the probe is too close to an edge of the sample. The arrangement of probes is shown in fig. 3. Detailed description of the method is given by L. Valdes¹⁰. This gives the functional relationship between the resistivity ' ρ ' and the voltage and current ratio for various geometries.

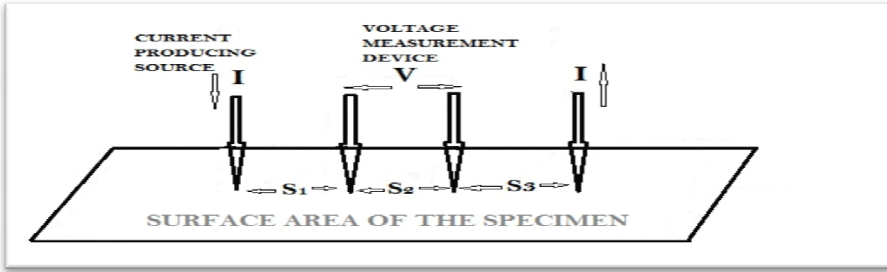


Figure 3: Electrical resistivity measurement by four point probe method

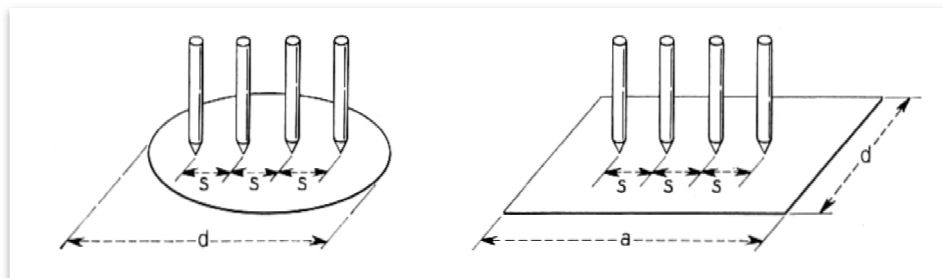
Later, A. Uhler¹¹ has evaluated functions, which gives the relationship for additional geometries. All these treatments are concerned with three dimensional structures and infinite in at least one direction. In the case of a four point probe on a sheet, the two outside current points represent the dipole. Therefore, the resistivity in this case can be given by¹¹

$$\rho = \frac{V_D}{I} 2\pi s \quad (3)$$

Here, the distance between all the four points is equal. I , is the current flowing through the sample, V_D is produced voltage across two inner points and S is the distance between the adjacent points. If the distance between contact points is not equal and it is given as S_1 , S_2 and S_3 respectively, then the resistivity is given as

$$\rho = \frac{V}{I} \left[\frac{2\pi}{\left(\frac{1}{S_1} + \frac{1}{S_3} - \frac{1}{S_1+S_2} - \frac{1}{S_2+S_3} \right)} \right] \quad (4)$$

Where, V is the floating potential difference between the inner probes, and I is the current through outer pair of probes. A detailed study has been done by F. M. Smit¹² for different geometries with various correction factors in the resistivity as shown in fig. 4 and correction factors are tabulated in the table 1.

Figure 4: Arrangement for the measurement of sheet resistivity with four point probe method¹².Table 1: Correction factor C for the measurement of Sheet resistivity with the four point probe method¹²

$$\rho_s = \frac{V}{I} C$$

d/s	circle diam d/s	$a/d = 1$	$a/d = 2$	$a/d = 3$	$a/d \geq 4$
1.0				0.9988	0.9994
1.25				1.2467	1.2248
1.5			1.4788	1.4893	1.4893
1.75			1.7196	1.7238	1.7238
2.0			1.9454	1.9475	1.9475
2.5			2.3532	2.3541	2.3541
3.0	2.2662	2.4575	2.7000	2.7005	2.7005
4.0	2.9289	3.1137	3.2246	3.2248	3.2248
5.0	3.3625	3.5098	3.5749	3.5750	3.5750
7.5	3.9273	4.0095	4.0361	4.0362	4.0362
10.0	4.1716	4.2209	4.2357	4.2357	4.2357
15.0	4.3646	4.3882	4.3947	4.3947	4.3947
20.0	4.4364	4.4516	4.4553	4.4553	4.4553
40.0	4.5076	4.5120	4.5129	4.5129	4.5129
∞	4.5324	4.5324	4.5324	4.5325	4.5324

2.4. Resistivity measurement for a disc of arbitrary shape (Pauw Method)

This is the method discussed by vander Pauw¹³ to measure resistivity of flat disc (pellet) of arbitrary shape without knowing the current pattern. This method is applicable only when satisfying the following conditions:

- The contacts are at the circumference of the sample.
- The contacts are sufficiently small.
- The sample is homogeneous in thickness.
- The surface of the sample is singly connected, i.e. the sample does not have isolated holes. A sample of comparatively low resistance and of arbitrary shape satisfies all conditions (A) to (D) as shown in fig. 5.

The resistance $R_{AB,CD}$ is defined as the potential difference $V_D - V_C$ between D and C contacts per unit current through the contacts A and B. Here the current enters in the sample through contact A and leaves it through B. Similarly resistance $R_{BC,DA}$ is defined. If the sample has an uniform thickness “d”, the resistivity ρ as a function of $R_{AB,CD}$, $R_{BC,DA}$ and ‘d’ of the sample can be determined as:

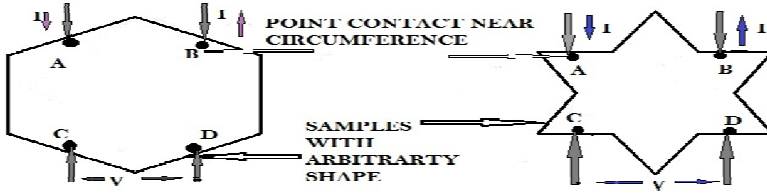


Figure 5: Samples of any arbitrary shape with four small contacts for electrical resistivity measurement

$$\rho = \frac{\pi d}{\ln 2} \left[\frac{R_{AB,CD} + R_{BC,DA}}{2} \right] f \left(\frac{R_{AB,CD}}{R_{BC,DA}} \right) \tag{5}$$

Where f is the function of the ratio $\left(\frac{R_{AB,CD}}{R_{BC,DA}}\right)$ only and satisfies the relation

$$\left(\frac{R_{AB,CD}}{R_{BC,DA}}\right) = \frac{f}{\ln 2} \operatorname{arccosh} \left\{ \frac{\exp\left(\frac{\ln 2}{f}\right)}{2} \right\} \tag{6}$$

f can be given approximately as and plotted against the ratio $\left(\frac{R_{AB,CD}}{R_{BC,DA}}\right)$ in figure 7

$$f \cong 1 - \left[\frac{R_{AB,CD} - R_{BC,DA}}{R_{AB,CD} + R_{BC,DA}} \right]^2 \frac{\ln 2}{2} - \left[\frac{R_{AB,CD} - R_{BC,DA}}{R_{AB,CD} + R_{BC,DA}} \right]^4 \left\{ \frac{(\ln 2)^2}{4} - \frac{(\ln 2)^3}{12} \right\} \tag{7}$$

However, this method is very useful to determine electrical resistivity of arbitrary shaped samples with some limitations but it is also modified by some scientists later on.

Ronald Chwang¹⁴ et al. was investigated the effects on van der Pauw’s resistivity and Hall coefficient measurement due to finite size contacts with selected shapes on a square sample. For the sheet resistivity measurement, correction factors for the apparent measured values at zero magnetic fields were determined from both electrolytic tank experiments and computerized over-relaxation calculations. For the Hall coefficient, correction factors for the effect of voltage shorting due to current electrodes and for the effect of current shorting due to Hall electrodes were calculated (by use of a fast-convergent over-relaxation technique) through a range of Hall angle from $\tan \theta = 0.1-0.5$. The current shorting contribution to the correction factor at zero magnetic field was also closely estimated by use of an electrolytic tank. In the symmetrical structures studied the Hall errors introduced by the voltage and current electrodes were approximately equal. The study shows that contacts of appreciable size relative to that of the sample can be a good approximation to van der Pauw’s infinitesimal contact. Thus, one can utilize the simplicity and other advantages of finite size ohmic contacts for these measurements in

normal semiconductor materials evaluation and still obtain precise data by using the appropriate correction factors determined in this paper.

Jonathan D. Weiss¹⁵ et al. along with two others has observed that van der Pauw formula for determining the electrical resistivity of an irregularly-shaped material was derived by him with the help of conformal mapping and is inherently two-dimensional. Thus, it is valid in the limiting case of a sample of infinitesimal thickness. In this paper, they demonstrate how the same formula can be derived from classical electrostatics for a rectangular sample of non-zero thickness which is allowed to approach zero. The calculation is carried out with the current probes at two adjacent corners and the voltage probes at the other two. In addition, the effects of non-ideality, particularly non-zero thickness and non-point contact voltage and current probes, are examined using the analytic formulation derived here and a semiconductor device code.

Later on Jonathan D. Weiss¹⁶ and others have reported that the original van der Pauw relationship could be derived from three-dimensional electrostatics, as opposed to van der Pauw's use of conformal mapping. The earlier derivation was done for a conducting material of rectangular cross section with contacts placed at the corners. Presented here is a generalization of the previous work involving a square sample and a square array of electrodes that are not confined to the corners, since this measurement configuration could be a more convenient one. As in the previous work, the effects of non-zero sample thickness and contact size have been investigated. Buehler and Thurber derived a similar relationship using an infinite series of current images on a large and thin conducting sheet to satisfy the conditions at the boundary of the sample. The results presented here agree with theirs numerically, but analytic agreement could not be shown using any of the perused mathematical literature. By simply equating the two solutions, it appears that, as a byproduct of this work, a new mathematical relationship has been uncovered. Finally, it is concluded that the application of this methodology to the Hall Effect measurements is quite useful.

2.5. Resistivity measurement of a pellet using Montgomery Method

The samples are obtained in powder form, used for pellet study. The pellet should be made by pressing the powder up to a sufficient and known pressure without using any binder. Usually these are shaped in the form of circular discs. A pellet of uniform thickness and circular in shape has four point contacts on the top surface of it arranged in the form of rectangle ABCD. The distance between the contacts at A and B is 'b' and that between the contacts at B and c is 'a'. The thickness of the pellet is 'd' must be less than or of the order of $0.3\sqrt{ab}$. As shown in the figure 6.

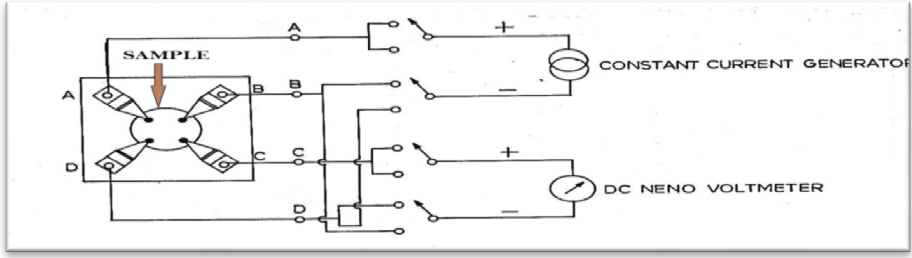


Figure 6: Circuit arrangement for electrical resistivity measurement of a pellet

The resistance R_1 is determined by sending current through AB and the potential difference is measured across CD. Now, the resistance R_2 is determined by sending current through AD and the produced potential difference is measured across BC. To change the direction of current and measure the resistances R_1 and R_2 a schematic arrangement as shown in the figure 5 has made. The theory behind the method of calculating the resistivity of the pellet from the knowledge of resistances R_1 , R_2 and thickness of pellet d is described by Montgomery¹⁷.

We have considered a rectangular pellet of width 'b', length 'a' and the thickness 'd' and if ABCD represent the corners of the top face of such a pellet, then according the theory developed by Montgomery, the resistivity can be given by

$$\rho = H E R_1 \tag{8}$$

Here, H is the geometric parameter which is a function of the ratio of width and length (b/a), and E is an effective thickness of the specimen. The effective thickness E, which is determined by the plot of a curve drawn in between the ratio (b/a) and d . However for $\frac{d}{(b/a)} < 0.3$, E does not depend on b/a significantly, and is almost equal to thickness of the sample.

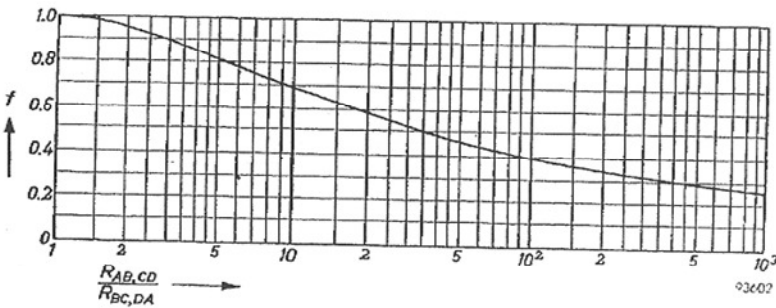


Figure 7: The function f used for determining the electrical resistivity of a sample, plotted as a function of $R_{AB,CD} / R_{BC,DA}$.

For the pellet, which is of the uniform thickness but not rectangular in shape, an effective value for b/a can be obtained from the ratio of R_2/R_1 . Montgomery has drawn a graph of R_2/R_1 as a function of b/a . The graph is insensitive to the actual thickness of the sample. To determine the resistivity of the sample, the effective value of b/a is obtained from the measurement of R_2/R_1 . Then the value of effective thickness E and geometric factor H are determined for this value of b/a . The value of H can be obtained directly in terms of R_2/R_1 from the table given by Montgomery¹⁷. Thus by putting the values of H corresponding to the different values of E in equation 8, one can determine the resistivity of the given pellet.

2.6. Resistivity measurement by using pulse probe method

A modified dc method known as the pulse probe method has reported by C.R.B. Lister¹⁸ can also be used to determine the resistivity of some samples by some modifications. Fig. 7 shows the block diagram for the pulse method. In this method, a short pulse of high voltage is applied to the sample and the current or the potential drop across a pair of probes of the sample is measured, usually by means of a fast oscilloscope or other amplifying and recording systems. Since the pulse is of very short duration and repeated only a few in times in a second or even less. The current density during the time of application of the pulse is very high without unduly heating or affecting the sample. This technique is most suitable for the samples having small dimensions, more fragile and having Joule heating problems.

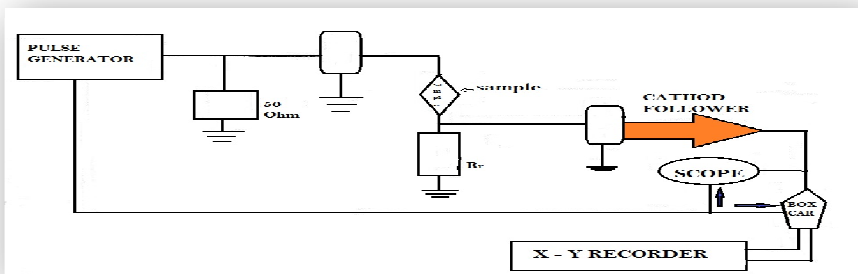


Figure 7: Block diagram for electrical resistivity measurement by pulse method

2.7. A spreading resistance technique for resistivity measurements

A technique is developed by R. G. Mazur and D. H. Dickey²¹ for determining local sample resistivity from the measured spreading resistance associated with a metal to semiconductor, small-area pressure contact is described. The major problems encountered in earlier attempts to derive quantitative resistivity data from small area pressure contacts on sample have been circumvented by making the measurements at bias

levels of a few millivolts and by using a particular osmium-tipped probe arrangement to provide contact reproducibility. The method provides a three-dimensional spatial resolution in resistivity measurements on sample on the order of 1μ , and, using a calibration curve determined for a particular silicon surface finish, yields an experimental reproducibility $\leq 15\%$ for sample resistivity in the range. Several examples of the application of the technique to problems of current interest in the technology are given.

2.8. Noncontact electrical resistivity measurement technique

A noncontact technique of measuring the changes in electrical resistivity (or resistivity) of conducting liquids is reported by Won-Kyu Rhima and Takehiko Ishikawa²². The technique is based on a conducting drop that is levitated by the high-temperature electrostatic levitator in a high vacuum. This technique, which utilizes the principle of the asynchronous induction motor, measures the relative changes in torque as a function of temperature by applying a rotating magnetic field to the sample. Changes in electrical resistivity are related to the changes in measured torque using the formula developed for the induction motor. Validity of this technique was demonstrated using a pure aluminum sample around its melting temperature. When the measurement results were calibrated by a literature value of resistivity at the melting point then it is found that those are in close agreement with the literature, confirming the validity of the present technique.

The electrical resistivity controls the flow of melts under the influence of electromagnetic force in the process of refining or growing semiconductor crystals, and it is a sensitive measure of concentration fluctuation in a critically mixed liquid alloy near the critical point in the homogeneous liquid phase. In a conventional rotating magnetic-field method for electrical conductivity, a cylindrical container holding a molten liquid is suspended by a tungsten wire, and the torque experienced by the hanging cylinder is measured when a rotating magnetic field is applied²³. However, chemically reactive liquids, especially molten refractory materials, contained in the cylinder may be easily contaminated, altering intrinsic electrical properties. Furthermore, one cannot expect those liquids to reach a deeply undercooled state as long as the container provides heterogeneous nucleants that trigger early solid-phase nucleation.

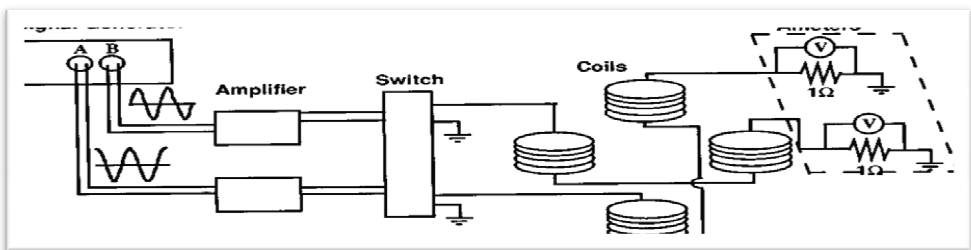


Figure 8: A schematic diagram of an electronics assembly that generates and channels appropriate electric currents to the coils to produce the desired magnetic field at the sample position²³.

3. Summary

In conclusion, we say that there are several methods to determine the electrical conductivity. An extremely important aspect of electrical resistivity measurement is to choose an appropriate method to the nature of the material is being studied. It is also being depended on the resistivity range, the temperature range, current flow and other condition of measurements. The bad effect from improper contacts may be most serious for these studies.

During the experiment for the resistivity measurement at room temperature or with the low temperature variation, there are a number of precautions, which must be cared to obtain good and accurate results. These cares can also save a lot of time consumed in the measurements on the breaking of the samples, unstabilization of the temperature, change in the dimensions of the sample or sample mount, Joule heating and breaking off the electrical connection from the sample etc. during the observation. These all problems may be eliminated out, if one can take care of some important factors. These are as given below and are purely dependent on the type of the study or measurement, nature of the sample and limitations of the experimental equipment's etc.

- I. Sample preparation.
- II. Adaptation of suitable method of measurement as per shape and nature of the sample.
- III. Suitable sample mount.
- IV. Temperature control at the sample.
- V. Electrical contacts with the sample.
- VI. Suitable value of input current or signal.
- VII. Evaluation of the homogeneity.

It is expected in these measurements that it needs a steady hand and a keen eye to work with some fine wires and small, brittle and fragile samples.

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