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Room Temperature Electrical Conductivity Estimation of Al-Si-fly ash Cenosphere Composites

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Abstract: This paper presents the results of an attempt made to review the various methods to measure the electrical conductivity of metals, composites and foams – a new class of material. Among the various methods reviewed, it is found that the van der Pauw method is reliable and gives accurate results for homogenous sheet specimens especially disc shaped specimens. Some results of conductivity measurements with wire samples are also highlighted. This paper demonstrates the broad applicability of the van der Pauw technique in estimating room temperature electrical conductivity of Al-Si-fly-ash cenosphere composites which helps in its characterization and identification of certain crucial mechanical properties. Some details of room temperature electrical conductivity measurements for disc shaped specimens having a small thickness to area ratio are reported. The variation of electrical conductivity at room temperature with percentage by weight of al-si cenosphere appears to follow the power law.

Keywords: electrical Conductivity; van der Pauw technique, eddy currents; Aluminium fly-ash; cenosphere composite.

1. INTRODUCTION

Cenosphere composites (Al-Si-fly ash), metal mixtures and above all, cellular metal foams are a relatively new class of material which are of interest because of the superior adaptability of their mechanical, thermal, acoustic, electrical, and chemical properties [1]. By suitably varying their physical structure like porosity (relative density), pore geometry (shape, orientation and size), and cell topologies (open cell and closed cell structures), the conductivity and hence some mechanical properties can be altered. Some major applications of these materials are in energy absorption, thermal management, lightweight structures, and automotive industry. Electrical conductivity depends not only on the relative density but also on the defects in the foam such as corrugation, broken cell walls and micro cracks [2,3]. Therefore, electrical conductivity information can be used for evaluating the mechanical properties, such as compressive elastic modulus, plateau stress and densification strain. It can also be used to measure other properties such as porosity and thermal conductivity. The measurement of electrical conductivity of non-ferrous metals and alloys is of particular interest to the coin production and handling industries and to the aerospace industry as it provides a measure of the quality of parts. Conductivity is also increasingly used in the specification of coins and in the detection of coins in sorting, handling and vending processes. Thus for these industries it forms part of the tools for combating fraud. The estimation of room temperature electrical conductivity of metal alloys, composites and metallic foams has been a non-trivial task and more complicated experimental arrangements would be required at higher and lower temperature ranges. It is of interest to review the various methods available for electrical conductivity measurement since they are common as far as all the above said new class of materials are concerned. In a broad sense, conductivity measurements fall under two major categories Viz; AC conductivity and DC conductivity measurements. A study of present literature also indicates that little information is available on electrical conductivity measurement of metals and foams especially ‘aluminium fly ash composites’. Therefore in this work an attempt is made to identify a simple method for estimating the electrical conductivity using easily available but yet sensitive instruments instead of resorting to costly instrumentation and frequency analysers[7]. Electrical conductivity of a sample can also be used as a means of determining: i. Type of metal or alloy ii. Type of heat treatment (for aluminium this evaluation should be used in conjunction with a hardness examination), iii. Aging of the alloy and iv. Effect of corrosion.

2. BRIEF REVIEW OF PRESENT ELECTRICAL CONDUCTIVITY MEASURING METHODOLOGIES

Non-destructive methods used for DC electrical conductivity measurement are the two point technique, four point techniques [1] and the van der Pauw method [2,3]. The majority of conductivity reference standards produced by manufacturers of eddy current conductivity meters are traceable through conductivity standards measured using a direct current method [13]. However, AC measurement is based on eddy current probing technique. These methods are applicable to any conducting material in general. In the present work we have considered the application of these techniques to homogeneous metal foams in general and Al-Si-fly-ash cenosphere composites in particular. Recent studies on eddy current measurements of conductivity and porosity of metal foams [4] have indicated that frequency range is critical in achieving reliable results. Recently reported cell morphology studies of Aluminum alloy foams using powder metallurgy techniques [5] indicate that the cells are uniform and closed and the relative density had a more pronounced influence on the conductivity compared to the cell diameter demonstrating the applicability of the percolation theory approach [6]. An attempt has been made in the present work to study and understand the present state of the art and report the difficulties involved in the measurement of electrical conductivity of metal composites and metallic foams and arrive at a simple acceptable method to estimate the conductivity. The traditional four point /probe method is widely used. It can be used to measure the bulk conductivity provided the samples are sufficiently thin. Many common experimental errors have also been listed [7]. In bulky materials such as rocks, plastics and paper the reliability may not be good since it is not possible to make 4 point probe measurement besides, very high voltages are needed to get measurable currents and also it suffers from disadvantage of very long time constants which prevents steady-state measurement. In highly conducting metals driving large current to produce appreciable voltage drop would often results in spark over and heating of contacts and associated problems. There are few reports which mention currents of order of 30kA using measuring bridges. Some reports recommend [7] the use of specialized commercial instruments to over come the above problems. However, conductivity measurement under DC. for homogenous, composite and foam materials has been a preferred topic for researchers. It should also be noted that transfer of electro-magnetic energy to the test sample to study field induced effects is not possible under DC due to the time invariant nature of the applied electric field. Among the various methods, the two probes, 4 probe and van der Pauw technique is more promising even though sample preparation (making thin disc shape Among the various methods the sheet resistivity and hence the conductivity of a homogeneous sample is measured by van der Pauw method [2, 3]. The advantages of van der Pauw method are highlighted. The present work involves the measurement of electrical conductivity of LM6(Cu 0.1%, Mg 0.1%, Si 10-13%, Fe 0.6%, Mn 0.5%, Ni 0.1% and Zn 0.1%) sample reinforced with fly-ash of varying composition by weight percentage (3 and 5%).

2.1. AC Conductivity Measurements

Although sensitive instrumentation is required for both AC and DC Measurements, AC measurement technique, besides being non-invasive, has inherent advantage of imparting electromagnetic energy into the sample and one can study the reaction effects. The most common effect is the interaction between the main field and induced eddy current field which is often used in commercially available absolute probe conductivity meters. Recently, experimental work [2, 3] involving the measurement of phase signature invariance of the test materials for a given coil geometry and frequency range using an impedance analyzer has been reported. Two type of coil sensors widely used to accommodate different shaped samples are planar pancakes and solenoidal windings. The experiments are conducted in the frequency range of 1Hz to 1MHz [8]. It is also reported that the selection of an effective operation frequency range is critical which depends largely on the coil configuration and properties of samples [2]. Attempts have been made to theoretically study the effect of the finite length of a sample on the complex impedance of a solenoid coil using 3D FEM package for frequency range of 100Hz to 158 kHz for 30mm & 300 mm long aluminum samples [7, 8]. These studies have shown that the phase frequency response of the normalized eddy current signal of a solenoid coil is independent of radius, electrical conductivity and magnetic permeability of test samples. For non-magnetic conductive samples the measurements reported in literature are based on a calibration curve of the coil relating the impedance change and the electrical conductivity of the sample with a known conductivity. For porous, Fe samples the measurement results were dependent largely on the data at low frequencies. Thus one can infer that, even though AC measurement has the advantage of cancellation of thermally induced effect because of the polarity reversal during measurement, the frequency range selection is critical which can result in large variations in the estimated values of conductivity.

2.2. Eddy Current Probe techniques (AC Measurements)

Eddy currents [1] induced inside the test sample by the applied magnetic field attenuate with depth below the test sample surface. This attenuation is mainly governed by the test object's electrical conductivity σ, magnetic permeability μ, and the applied frequency f for a given test geometry. The standard depth of penetration δ can be used to characterize this diffusion phenomenon, which, for plane geometry, is mathematically given by

$$\delta = \frac{1}{\sqrt{\pi f \mu \sigma}} \qquad \text{………….} (1)$$

For nonmagnetic electrically conductive foam the value of μ virtually is the permeability of free space. Since the probing area depends on the skin depth, multifrequency testing is preferred to investigate metallic foams or conducting materials like aluminum alloys. In case of DC measurements this would not be required.
The penetration of eddy currents into any material for sinusoidal excitation is given by the Diffusion Equation (2)

$$\nabla^2 \boldsymbol{A} + jw\mu\sigma\boldsymbol{A} = -\mu\boldsymbol{j} \qquad \text{............ (2)}$$

The electric field intensity E is given by equation (3), where the symbols have their usual meanings. Normally, induced voltage in the sensing coil is computed taking the line integral of the vector E over the coil loop, thereby obtaining the coil impedance.

$$\mathbf{E}=-\partial\mathbf{A}/\partial t \qquad \text{............ (3)}$$

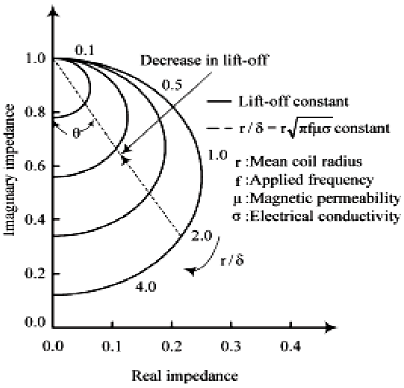


Figure 1: Normalized impedance curves for planar circular coil varying with reference numbers *r/δ* (coil radius/skin depth) and lift-off (coil-to-sample spacing) [1]. Figure 1 above illustrates the variations of real and imaginary impedance components for electrically conductive (nonmagnetic) materials under different values of reference number and lift-off (coil-to-sample spacing) [2, 3]. The reference number is defined as *r/δ*, i.e., the ratio of mean coil radius r and skin depth. It is found that the phase signature lift-off invariance is significant in that it indicates the existence of a relationship between phase angle θand the reference number *r/δ*. For the block and sandwich shaped samples using planar pancake coils, a linear relationship has been observed between the cotangent of the phase angle and the reference number *r/δ* through a least square fit to the measured data of bulk materials [21]

$$\cot\theta = b + a\frac{r}{\delta} \qquad \text{............ (4)}$$

The electrical conductivity of the material σ is thus solved in the value of

$$\sigma = \frac{(cot\theta - b)^2}{\pi\mu a^2 r^2 f} \qquad \text{............ (5)}$$

The values of parameters a and b in (4) and (5) are application dependent mainly on the coil sensor geometry and can be calibrated using the measurement data of bulk materials. For the cylindrically shaped samples using solenoid coils, the corresponding relationships are [21]

$$\frac{1}{r\sqrt{w\mu\sigma}} = X(\theta) \qquad \text{............ (6)}$$

The electrical conductivity σ can be evaluated using the precalculated values of $X(\theta)$ by

$$\sigma = \frac{1}{w\mu\,(rX(\theta))^2} \qquad \text{............(7)}$$

Where $X(\theta)$ can be calibrated based on the impedance curves of bulk materials as their electrical conductivity σ is already known. Appropriate coil selection is the most important part of solving an eddy current application and various categories of coils commonly employed [13] are;

- Surface probes used mostly with the probe axis normal to the surface.
- Encircling coils that are normally used for in-line inspection of round products.
- ID probes used for heat exchangers have their axis along the center of the tube.
- The use of absolute, differential and reflection type of probes is discussed in earlier work [13]. These tests are carried out as per ASTM E1004-09 standards [16].

Four point method (DC Measurements)

Two common methods using DC are the in line two and four probe methods. In the two probe method, a constant DC voltage is applied to the two ends of a bar shaped sample and the current through it is measured (in contrast to a current source driving a constant current through the sample in the 4-probe method). In the four probe method, a constant direct current source drives a current and the resulting potential drop is measured. Further, in the four-probe method [1], an inline four-point probe is placed on the surface of a sample sufficiently thick so it can be approximated to be semi-infinite (Fig. 2).

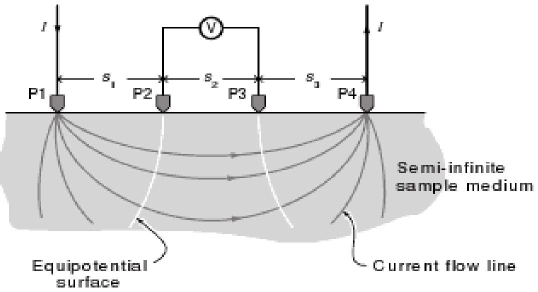


Figure 2. Four-point probe method for measuring the electrical conductivity [1].

A direct current is passed through the specimen between the outer probes (P1 and P4), and the resulting potential difference is measured between the inner probes (P2 and P3). By using separate probes for the current injection and for the determination of the electric potential, the contact resistance between the metal probes and the material will not show up in the measured results. If the sample is sufficiently thick, the electrical resistivity *r* is given by [1]:

$$r = \frac{2\pi\left(\frac{V}{I}\right)}{\left[\frac{1}{S1}+\frac{1}{S3}-\frac{1}{S1+S2}-\frac{1}{S2+S3}\right]} \qquad \text{………….} (8)$$

Where s1, s2, and s3 are the probe spacings shown in Fig.1.

The electrical conductivity σ (units,$\Omega^{-1}\mathrm{m}^{-1}$) is the reciprocal of the measured resistivity. The resistance *R* (in **Ω**) of a piece of specimen of length *l* and cross-sectional area *A* normal to the direction of current flow is given by:

$$R = \rho\frac{l}{A} = \frac{l}{\sigma A} \qquad \text{………….} (9)$$

2.3. Van der Pauw Method

The van der Pauw Method is a technique commonly used to measure the Resistivity and the Hall Coefficient of a sample. Its power lies in its ability to accurately measure the properties of a sample of any arbitrary shape without the knowing the current pattern, as long as the sample is approximately two-dimensional (i.e. It is much thinner than it is wide) and the electrodes are placed on its perimeter. It allows avoiding problems due to the incorrect knowledge of sample geometry. From the measurement made, the conductivity of the material and the mixture type can be estimated. The method was first propounded by Leo J. van der Pauw [2]. The advantages of this method include low cost, simplicity and elimination of problems due to current distribution. The van der Pauw technique can be used on any thin sample of material and the four contacts can be placed anywhere on the perimeter/boundary, provided certain conditions are met viz., the contacts are on the boundary of the sample (or as close to the boundary as possible), the contacts are infinitely small and is thin relative to the other dimensions. Further, it is reported in the recent literature that the method based on the Van der Pauw effect for measuring conductivity has proved to be a reliable, low uncertainty technique for measuring block-shaped references at DC, although its application is limited at ACdue to the considerable challenges in the measurement [19]. The discrepancies between AC and DC measurements have been highlighted and it has been reported that AC values of conductivity are higher that DC values by 0.5% [19].

3. Experimental details

3.1. Sample Preparation:

There are four conditions that must be satisfied during preparation of sample before experimentation to use van der Pauw technique, they are: 1. the sample must have flat shape of uniform thickness 2. The sample must not have any isolated holes 3. The sample must be homogeneous and isotropic, 4. All four contacts must be located at the edges of the sample [17]. Al-Si (LM-6) alloy was used as matrix material and fly - ash cenosphere as reinforcement material. Al-fly-ash composite was prepared by dispersing fly-ash cenosphere particle in aluminum matrix using stir-casting technique. Three casings were prepared each having 0%, 3% and 5% of fly-ash by weight. These castings were machined to 18mm diameter cylindrical rods. From each rod samples were cut at three different locations i.e. at two ends and at the center. During machining and finishing of the sample, care was taken to maintain a uniform thickness of 0.8mm to satisfy the conditions imposed by van der Pauw method. Initially attempts were made to estimate the electrical conductivity of aluminum. The sample was a rod of 2mm diameter and length 1m. A Kelvin double bridge was used to measure the resistivity and hence the conductivity was estimated. The values of the conductivity thus estimated agreed with the standard values with an error of ±5%. This was repeated for homogenous LM6 samples and the results were repeatable. This indicated that as long as the samples could be cast in the form of rods of length much larger than the cross sectional area, the conductivity values could be estimated with reasonable accuracy. Preparing such samples (wires) with metal composites such as the ones used in the present work is difficult and not practicable especially with foamed structures. In the current investigation, experimental set up shown in figure 2 was used. As-cast LM6 and LM6 with addition of 3% and 5% fly-ash (by weight) of cenosphere composites is used to estimate electrical conductivity by the van der Pauw method. In each case three samples were tested at four different locations. The average values are tabulated.

The LM6 rod of 18 mm diameter was sliced into a thin disc of 0.8 mm and the conductivity measurements were carried out with the standard equipment (Keithley Integra Series Model 2700 Multimeter/Data Acquisition System) – see Fig.3.

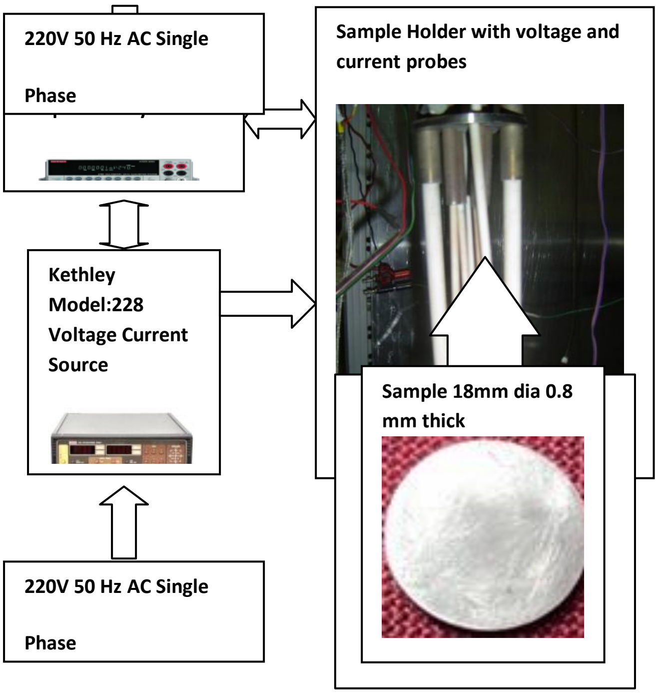
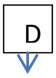


Fig.3 (a) Experimental set-up and typical van der Pauw measurement points ABCD on the test sample.

The sample holder shown in Figure. 3(a) is only used for holding the specimen and to have proper contact between the specimen and the four probes. The limitation of this setup is that it can only be used for small size samples of length or diameter around 10mm to 20mm and thickness below 1mm. The setup consists of thermally insulated metal rods, to minimize the effect of temperature during resistivity measurement. For room temperature electrical resistivity measurement even bare conductors can be used. A pair of the probes was connected to the constant current source (0-10 A) and the other pair was connected to the multimeter to measure the voltage drop. To measure the voltage drop keithley model 2700 multimeter/data acquisition system was used –Fig 3.

A current of $I_{AB,p}$ was applied with the help of current source across the points A and B see Fig 3 (b). The voltage drop $V_{CD,p}$ was measured, across the points C and D, with the help of a multi-meter see Fig. 3 (b) . The polarity of the input current was altered by swapping the positive and negative poles of the current leads. Again the voltage drop $V_{CD,n}$ was measured, across the points C and D, with the help of a multimeter. The voltage drop across the points C and D was then calculated as



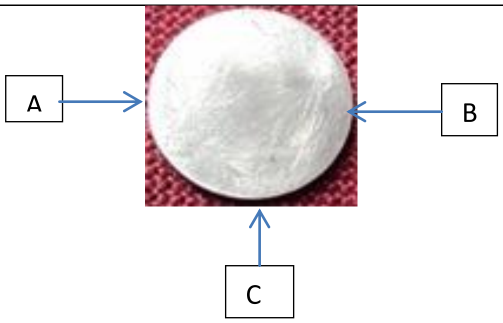


Figure: 3 (b)

$$V_{CD} = \frac{V_{CD,p}+V_{CD,n}}{2} \quad \text{…………(10a)}$$

The resistance R_{AB} is calculated as

$$R_{AB} = \frac{V_{CD}}{I_{AB}} \quad \text{………… (10b)}$$

Then a current of $I_{Ac,p}$ was applied with the help of current source, across the points A and C. The voltage drop $V_{BD,p}$ was measured, across the points B and D, with the help of a multimeter. The polarity of the input current was altered by swapping the positive and negative poles of the current leads. Again the voltage drop $V_{BD,n}$ was measured, across the points B and D, with the help of a multi-meter. The voltage drop across the points C and D was then calculated as

$$V_{BD} = \frac{V_{BD,p}+V_{BD,n}}{2} \quad \text{………… (10c)}$$

The resistance R_{AB} is calculated as

$$R_{AC} = \frac{V_{BD}}{I_{AC}} \quad \text{………… (10d)}$$

The resistivity of the specimen was calculated as

$$\rho = \frac{\pi}{\ln 2} \frac{R_{AB}+R_{AC}}{2} f(r)T \quad \text{………… (10e)}$$

where T = sample thickness,

f(r)= function depends on the resistance ratio

$(R_{AB}/R_{AC}) \approx 1$ To compensate for thermally induced effects polarity of both the current and voltage terminals were reversed. Since
the points ABCD shown in Fig 1 (b) forms a square, the shape correction factor [2] becomes unity. The voltages were measured across points CD and BD for an excitation current of 0.5A and 1 A and the average was taken as shown in Table 1 below:

Table: 1

Typical room temperature electrical conductivity for pure LM6 Specimen (Suffixes p and n indicate positive and negative polarity) is shown in the table below:

I (A)	**V_{CDp} (µv)**	**V_{CDn} (µv)**	**V_{CD} (µv)**	**V_{BDp} (µv)**	**V_{BDn} (µv)**	**V_{BD} (µv)**	**σ MS/m**
0.5	6.8	9	7.9	2.3	3.6	2.95	25.4188
1.0	6.7	5.2	5.9	6.1	5.9	6	25.1293

4. RESULTS AND DISCUSSION

The experiments were repeated for Al fly-ash composites reinforced with 3% and 5% fly ash by weight. The corresponding values of conductivity were 16.9841 MS/m and 14.2857 MS/m. It can be seen that the conductivity decreases as the fly-ash percentage increases.

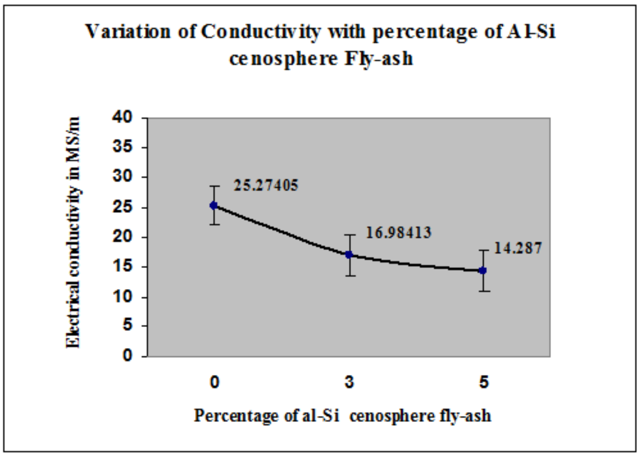


Figure. 4. Graph showing electrical conductivity versus percentage by weight of Al-Si-fly ash Cenosphere Composite

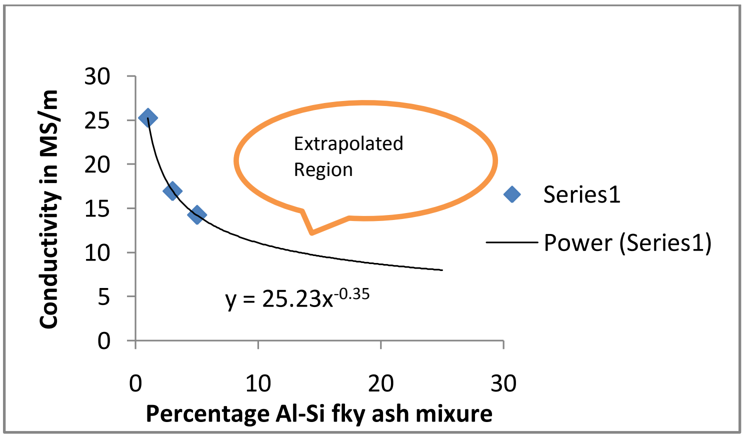


Figure. 5. Conductivity variation with percentage variation of Al-Si Fly ash mixture with Power Law curve (extrapolated region extends to 30 % by weight mixture)

The spread in the values of estimated conductivity values were ± 10% as shown in Figure.5 above. The values indicated in Fig.4 were averaged over three trials. The values of conductivity for 5 %(by weight) fly-ash mixture were more consistent as compared to 3 % (by weight) fly-ash mixture. The best fit for the curve was the “Power Law” curve. The curve fitting was done taking an upper limit of 30 % by weight mixture. It is speculated that it follows the Power Law (equation - $y = 25.232x^{-0.356)}$. This was best fit curve was the power law curve for the scatter indicated by the experimental results. However, more experiments are required to establish this. These differences in estimated values are attributed to thickness variations in the sample and in homogeneity in the mixture.

5. CONCLUSION

The methods applicable to estimate the electrical conductivity of metal alloys, mixtures, composites and foams were reviewed and it was found that vand der Pauw method gives reliable results besides being simple. It can be seen that electrical conductivity of Al-Si-fly-ash chemosphere composite reduces with increase in percentage by weight of fly-ash. The percentage drop in the electrical conductivity of Al-Si-flyash cenosphere composite sample when reinforced with 3% fly ash, by weight, is about 32.80% and when mixed with 5 % fly ash, by weight, the percentage drop is about 43.47%. It is speculated that this variation follows the “Power Law”. The present work has demonstrated the use of the above van der Pauw non-destructive technique for characterizing metal matrix composites and also indicates the applicability of other methods of conductivity estimation.

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