Science Journal of Physics
ISSN: 2276-6367
http://www.sjpub.org/

Published By
Science Journal Publication
International Open Access Publisher

Research Article **Volume 2012 (2012), Article ID sjp-283, Issue 3, 6 Pages, doi: 10.7237/sjp/283**

Temperature Coefficient Resistance and Microstructural Analyses of Iron-clay Based Composite Resistors

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Accepted 22 March, 2012

ABSTRACT- Experimental investigations have been carried out to study the variations of Microstructure and Temperature Coefficient Resistance occurring in Fe-clay based composite resistors. The resistors were produced from natural materials taken from Ibadan , and fabricated by a compaction method. The pressure of the mould = $1.00 \pm 0.02 \times 10^7$ N / m^2. The resistors have a constant thickness of 3.5 mm and varying lengths ranging from 3.00 mm to 10.00 mm. The resistors of composition 60 wt%Fe to 90 wt%Fe were subjected to varying annealing schedules, (i.e. varying peak firing temperatures ranging from 100 °C to 1000 °C and firing times from 5 mins to 240 mins. Most of the resistors fabricated with the Ibadan clays have negative TCR values. Their values are as low as $\pm 2 \times 10$ ppm / °C which are comparable with thermistors and other standard resistors. Scanning electron microscope model 1990 at IITA Ibadan was use to do the analyses. From the micrographs, it could be suggested that relevant interactions of both conductive and resistive films took place, which in turn control the value of the resistance of the resistors.

Keyword: Temperature Coefficient Resistance, Microstructure, composite resistors, Conductive and Resistive films

Introduction

Resistors are devices specifically introduced into an electronic circuit to offer resistance to the flow of electric current. Resistors may be classified according to the general field of engineering in which they are used .We have the power resistors which are used in both the power and electronic fields of engineering, instrument resistors are designed to provide a voltage drop of 5 mV when a stated current passes through them while standard resistors are used for calibration purposes in resistance measurements and integrated circuits – Faleke (1996).

The iron-clay composite resistors, like other types of solid metal-insulator–based composite resistors consist of tiny metallic grains separated by an insulating medium, pressed together under high pressure to initiate mechanical interatomic bonds for further processing towards a stable and reliable structure. Sometimes the resistors might be subjected to suitable heat treatment to burn-off the organic vehicle and other volatile organic constituents to produce a solid of higher strength.

Thick films resistors (TFRS) are very complex composite materials whose electrical properties are also related to the substrate. The change of the resistance and its temperature coefficient of resistance (TCR) due to film substrate interactions and to a mismatch of the thermal expansion coefficients of the film and the substrates have been evidenced by Cattaneo et al (1980).

The temperature coefficient of resistance of a thick film material is determined by the separate temperature coefficient of its conducting constituents in their final form after firing, modified by the effects of mechanical stress. Anything that alters the ratios of the components or the stresses built into the resistor during manufacture is liable to affect the overall TCR – Prudenziati (1983). The TCR is often quoted as two values based on spot measurements e.g. 'cold - 40 °C to +25 °C and hot +25 °C to 100 °C. Firing at different peak temperatures for different times affects the chemical composition of the resistor and thus the resultant TCR. The rate of cooling from peak temperature will control the amount of inherent stress in the resistor and will thus affect its TCR. The TCR of any given resistor depends mainly on the properties of the materials present. It is therefore determined by the separate temperature coefficient of resistance of its constituents in their final form after firing. The microstructure of thick film resistor, differs significantly from that of a pure metal or pure insulator, it is a mixture of conductive and insulative phases. Consequently the electronic conduction mechanism and hence its resistivity will vary with particle size, the relative amount and the spatial distribution of these particles. It will also be affected by structural defects introduced during processing. The conduction in composite resistors is similar to that in cermet film resistors –Akomolafe and Oladipo (1996), Prudenziati (1991,1994).Extensive investigations by Prudenziati (1983) Vest (1975) and Ching (1979) have shown that the development of microstructure in thick film resistors is very complex and affected by a variety of parameters such as the composition, softening point, viscosity, thermal expansion coefficient and wetting properties of the glass, the ratio of the size of glass particles to that of metal-oxide grains - Ching (1979). The sintering properties of the conductor material are also one of the parameters known to affect the final microstructure of thick film materials, besides the temperature and time (in a defined firing cycle) - Prudenziati (1983). Also we can expect differences in the structure, and of course in composition, between resistors obtained from inks of different formulation. Nevertheless some general features emerge from the investigation of the structure of different resistors, which can be associated with the general features of their electrical properties. The knowledge of the microstructure of the systems surely plays an important role in understanding their electrical conduction mechanism and in finding a model not only able to produce a 'posteriori' of the particular experimental data but also in agreement with the system's microstructure. We report an accurate investigation on the dependence of resistance on working temperature for some

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of the Iron – clay composite resoistors and using the SEM to study the role-played by preparation parameters and in order to evidence the correlation between transport properties and microscopic structure.

EXPERIMENTAL METHODS

PROPERTIES OF IRON

Iron is the fourth abundant element in the earth's crust (5.1%). Pure Iron is a silvery-white, rather soft metal, which is both malleable and ductile at room temperature. Its physical properties however, are profoundly altered by the presence of trace amounts of other elements and therefore its magnetic properties are very dependent upon the presence of impurities. Iron absorbs hydrogen especially at high temperature to form solid solutions. Some form of finely divided iron is pyrophoric in air at room temperatures. Massive iron begins to oxidize in dry air above 150 °C, Fe2O3, and Fe_3O_4 being the major products in excess of oxygen.

PROPERTIES OF CLAY

The clay minerals are an ill-defined group of secondary minerals formed at or near the earth's surface by weathering or hydrothermal alteration of feldspars and other aluminous silicates They are characteristically fine –grained, often below the resolution of a light microscope, making them difficult to identify by optical means. Most clay minerals are phyllosilicates with structures based on combinations of brucite-type layers of octahedral coordinated cations and Si_4O_{10} layers of tetrahedral coordinated cations (Si_4+ or Al_3+).

MATERIAL COLLECTION

The sodium silicate binder, which acts as vehicle, was bought from the chemical laboratory at Ilorin. The iron fillings used for the cermet mixtures were of 99.9% purity (British drug house limited). The clay sample used was collected at Ekotedo, Ibadan. The clay is light brown in color, inorganic with some sands and trace of gravel with trace of mica.

Preparation of the clay samples

The clays were dried between temperatures of 105 °C to 110 °C for 24 hours. Each sample was grounded on cooling in fine powder state.

Wet Sieving

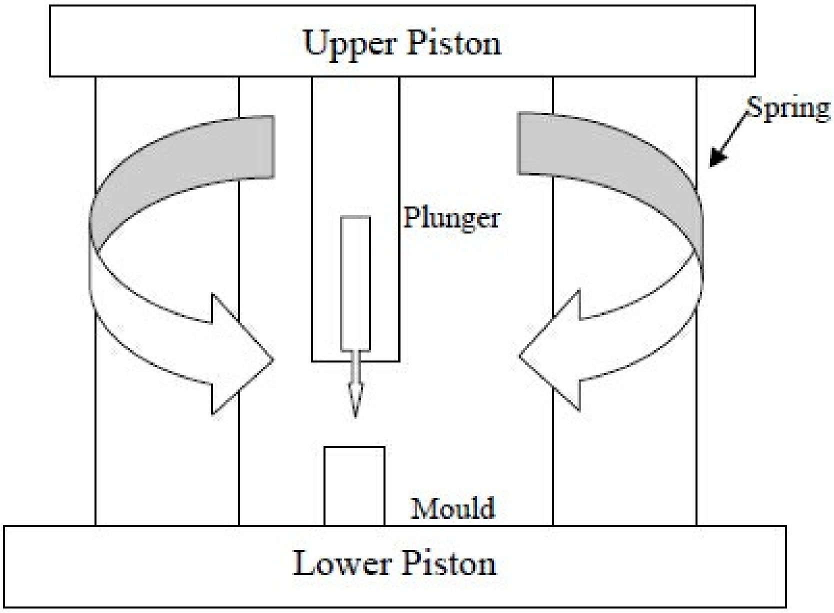
The objective of the wet sieving was to remove sand and gravel contained in each of the samples. The separation was done by wet sieving each sample with mesh number 150 µm aperture size.

WEIGHING

The iron and clay were weighed by using laboratory chemical weighing balance Ganllenhamp Mettler P165. A filter paper and its powdery content (clay and iron) were placed on the scale pan one after the other and each weighed accordingly.

MOULDING OF THE RESISTORS

The moulding assembly consists of a rectangular steel bar with a central hole of 3.5 mm, a base steel plate mould cap of less than 1 cm in length to block the hole at the bottom side before loading the resistor materials and a long nail acting as the piston to compress the loaded materials as shown in Fig.1 The diagram shows the upper piston, lower piston, plunger, the mould and the spring. The weighted iron filings and clay powder were mixed thoroughly into homogeneity inside a beaker. The mixture was stirred thoroughly with 2 or 3 drops of the silicate binder until the paste becomes thick with a very uniform texture. The paste was then loaded into the mould and compressed with mould cap. The compression takes place by carefully placing standard weights on the mould cap for



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some minutes, then removed. This enhances a proper application of moulding force to the material. The resistors were then ejected out of the mould safely. All mouldings were done at room temperature. The moulding was done for different percentages of iron and clay and different resistors of varying lengths were moulded. The diameter of the resistors is constant at 3.5 mm. All the moulded resistors, which have been sufficiently dried at room temperature were subjected to firing at temperatures in the range 100 °C – 1000 °C. The firing was done using the Vecstar furnace model 1982. The resistors were then furnace cooled to room temperature after firing. Constantan wire was then soldered to the metal contact ends of the samples; this was to give electrical terminals to the resistors. The pressure for the moulding is calculated as,
Pressure applied = Force applied /cross sectional area A

Since the force applied = 150N and the
Cross sectional area = πr^2 = 3.142 x 0.0035m
Then the pressure applied = $(1.55 \pm 0.12)\ 10^7\ \mathrm{Nm}^{-2}$

The Robin made volt-ohm-millimeter multimeter model 1400, which is capable of reading d.c and a.c voltages, currents, and resistance measurements was used to measure the resistances of the resistors.

RESULT AND DISCUSSION

Temperature Coefficient of Resistance (T.C.R.)

Since the electrical resistance of a conductor is dependent upon collisional processes within the wire, the resistance could be expected to increase with temperature since there will be more collisions. An intuitive approach to temperature dependence leads one to expect a fractional change in resistance, which is proportional to the temperature change

$$\alpha R/R_o = \alpha \Delta T \qquad (1)$$

where α = temperature coefficient of resistance.
The temperature coefficient of resistance shall be calculated from the following formula,

$$\mathrm{T.C.R\ (ppm/^oC)} = \{(R-R_o)/R_o\}\{1/(T-T_o)\} * 10^6 \qquad (2)$$

where: R is the value of resistance at temperature T
R_o is the measured resistance at T_o
T is the Absolute Temperature (°C)
T_o is the Base Temperature (°C)
Ibadan Clays

Fig 1 shows the relationship between TCR and working temperature. For resistors of lengths 4.00 mm, 7.00 mm, 10.00 mm fired at T_f = 600 °C and having compositions of 80 wt%Fe and 90 wt%Fe, their TCR is linear and increases with increase in working temperature, at almost all percentage weight of iron except for resistors of length 4.00 mm fired at T_f = 300 °C and having a composition of 80 wt%Fe whose TCR values are negative. The negative value of TCR could be due to another material been present in the resistor, i.e. the incomplete combustion of the organic vehicle.

The variation of temperature coefficient of resistance with working temperature is shown in Fig 2 for resistors of lengths 4.00 mm and 10.00 mm fired at T_f = 300 °C and of compositions 60 wt%Fe and 70 wt%Fe. The TCR decreases with increase in temperature and have negative values. The little deviation from linearity of TCR may be a consequence of incomplete annealing and the sudden drop in the values of TCR i.e. electrical resistance could be due to incomplete annealing and incomplete ordering of constituents during firing. The TCR for lengths 7.00 mm and 10.00 mm fired at T_f = 300 °C and 600 °C of compositions 60 wt%Fe and 70 wt%Fe are linear.

We notice that there are no turning points in the temperature dependence of resistance curves as in the ruthenium and palladium - based resistors, and our resistors have negative values of TCR. The absence of turning points is an indication that conduction took place via ordinary particle contacts in the iron – clay based composition resistors. The TCR of our moulded resistor is of the order of ± 2 ppm / °C, which could serve as miniaturized circuit component as a result of very small sizes and low TCRs. The high TCR value of ± 10 ppm / °C implies that the resistors are suitable for high precision applications where there is a wide variation of temperature. A negative TCR and decrease of resistance with increase in temperature shows the behavior of a thermistor. A thermistor is a temperature sensitive device and operates in air condition fuel injection and central systems. If the typical properties of modern thick film resistors are examined carefully two clues to the process of electrical conduction can be obtained: low value resistors tend to have a positive TCR whereas high value resistors tend to be negative i.e. the more conductive material looks metallic while the poorly conductive material is more like a semiconductor or insulator. For a typical mid-range resistor, a decrease in temperature increases the negative value of TCR, and an increase in the temperature gives a more positive value. These observations indicate a transition between metallic and non-metallic behavior. TCR becomes less negative or even turn positive due to the phase transitions of the conductive grains.

According to Vest et al (1975) on the dependence of charge on microstructure in thick film resistors they observed that a good resistor will have a TCR less than 100 ppm / ° C which is one of the most desired features of thick film resistors, that is their low TCR. Our result is in agreement with these facts.

MICROSTRUCTURE ANALYSES

IBADAN CLAYS

Fig. 3 shows the surface morphology of 70 wt%Fe resistors of length 4.00 mm, fired at T_f = 300 °C. It was observe that the resistors have bright agglomerates randomly distributed on its surface. The resistor structures consist of conductive grains separated by dielectric layers. Some of the conductive grains make contact through dielectric layers. This structure supports the electron percolation mechanism for resistors. Also there is no change in the surface morphology of the resistors because of inhomogeneity which might have been introduced into the structures as the length of the resistors is increased. This may result in a larger separation between conductive grains, thus reducing the probability of electron

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tunneling by percolation. We also observe brighter large regions, which are essentially free of iron grains.

The surface morphology is shown in Fig 4 for 70 wt%Fe resistor of length 7.00 mm, fired at T_f = 300 °C. Figure shows that there is a canyon like zones in the layer of the resistor. These microstructures suggest that sintering and glass flow have not yet taken place. The layer has large exposed surfaces and it is greatly disconnected. One should anticipate a poor conduction of the material in this situation. The figures have darker surfaces. The dark areas observed on samples fired at high temperatures are rich in silicon and its oxides.

A 70 wt%Fe resistor of length 10.00mm fired at T_f = 300 °C surface morphology is shown in fig 5. The resistor layers are compact with dark surface. The dark surface observed on resistor morphology shows that they are rich in silicon and its oxide.

Fig. 6 shows the surface morphology of 70 wt%Fe resistor of length 4.00 mm fired at T_f = 600 °C. The resistor structures have darker surfaces suggesting that oxidation of the iron and silicon has started at this temperature.

Fig. 7 shows the surface morphology of 70 wt%Fe resistor of length 7.00 mm fired at T_f = 600 °C. The resistor structures have darker surfaces suggesting some oxidation. The micrograph shows that the resistor is a fiber or needle - like microstructure with bright large regions, which are essentially free of iron grains. The latter small white dots are dispersed in the darker region. The compositional contrast in this picture suggests the presence of elements with lower atomic number in the surrounding metal grains. The regions form a continuous channel network between the resistor terminations. It turns out that the iron –clay grains are partially sintered and or agglomerated during the thermal treatment.

The surface morphology of 70 wt%Fe resistor of length 10.00 mm fired at T_f = 600 °C. is shown in Fig 8. We observe brighter large regions, which are essentially free of iron grains, We have higher oxidation and grain growth at this temperature. The resistor structures consist of conductive grains separated by dielectric layers, some of the conductive grains make contact through dielectric layers. This structure supports the electron percolation mechanism for resistor. Also there is no change because of inhomogeneity which could have been introduced into the structures as the length of the resistors is increased. This may result in a larger separation between conductive grains, thus reducing the probability of electron tunneling by percolation.

Fig. 9 shows the surface morphology of 70 wt%Fe resistor of length 4.00 mm fired at T_f = 1000 °C. The resistor structures consist of conductive grains separated by dielectric layers. Some of the conductive grains make contact through dielectric layers. These grains have been fully oxidized and have crystal growth, which can be said to be secondary growth. The grains are irregular in nature and this is also termed coarsening. This structure supports the electron percolation mechanism for resistors. Also there is no change in the surface morphology of the resistor because of inhomogeneity which might have been introduced into the structures as the length of the resistors is increased. This may result in a larger separation between conductive grains, thus reducing the probability of electron tunneling by percolation. We also observe that the resistors have brighter large regions, which are essentially free of iron grains. The latter small white dots are dispersed in the darker region. The compositional contrast in this picture suggests the presence of elements with lower atomic number in the surrounding metal grains. The regions form a continuous channel network between the resistor terminations. It turns out that the iron – clay grains are partially sintered and or agglomerated during the thermal treatment.

Fig 10 is the surface micrograph of 70 wt%Fe resistor of length 7.00 mm fired at T_f = 1000 °C. The micrograph shows that the resistors have dark surface suggesting it is Si rich and other oxides in the resistor structures, which consist of conductive grains, separated by dielectric layers. Some of the conductive grains make contact through dielectric layers. This structure supports the electron percolation mechanism for resistors. Also there is no change in the surface morphology of the resistor because of inhomogeneity that might have been introduced into the structures as the length of the resistors is increased. This may result in a larger separation between conductive grains, thus reducing the probability of electron tunneling by percolation. Fig. 11 is the surface morphology of 70 wt%Fe resistor of length 10.00 mm fired at T_f = 1000 °C. The morphology of the resistor shows a crystalline flakes on the resistor. There is secondary growth in the material too. We still observe high degree of oxidation and sintering of the particles.

We notice from Figs 3 to 11 that there are changes in the surface morphology of the resistors of lengths 4.00 mm, 7.00 mm and 10.00 mm at firing temperatures of 300 °C and 600 °C, but as the firing temperature increases to 1000 °C there is almost no significance difference in the resistors surface morphology. This is due to stability in the sintering, grain growth and oxidation of the iron grains.

CONCLUSION

The experimental investigations on the microstructure and electrical properties of iron clay thick composite resistors have provided valuable information and allowed some progress in the knowledge of these materials. We have being able to show that the Temperature Coefficient of Resistance of resistors depend on their length and on firing time, firing temperature. This length dependence property is in agreement with previous report of Garvin and Stein (1970).

The results of our investigation emphasis the following main points: -

- Exchange reactions and redox reaction (including oxidation of carbonaceous residues) make the microstructure and electrical properties of the layers very variable according to the firing condition.
- Our results clearly give further evidence of the correlation between the electrical properties of the resistors and their microstructure.

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-Most of the resistors have negative TCR values, which could be due to the role played by iron oxide for Ibadan cities. We record TCR values of the order of ± 2 (ppm / °C).

-We notice that composition of the resistors and the peak firing temperature do not affect the sign of TCR for Ibadan clay though, these factors affect the magnitude of TCR for Ibadan clay.

-The research has shown that refiring processes give macroscopic variations of structure of the resistors easily detected by scanning electron microscope.

- The pictures show evidence that a substrate interaction occurs, with iron dissolution in the layer. It confirms that electrical transport in these Fe-clay composite resistors does involve conductive paths in the intergranular layers, made somehow conductive.

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