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ANNEALING SEQUENCE EFFECTS ON COPPER-CLAY BASED CERMETS RESISTIVITY

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ABSTRACT
This paper studied the annealing sequence effect on copper-clay based cermets using electrically conducting and chemically active copper powder with clay in mass ratio. The fabricated cermets were 5 mm in diameter with varying lengths ranging from 5 mm to 25 mm with interval of 5mm length. The samples were measured in mass ratio and the cermets were fabricated by compaction method using pressure size of $(1.5 \pm 0.01) \times 10^{6}$ *Pa. Annealing to temperatures ranging from 200ºC to 500ºC with 100ºC interval was employed. The mass content of clay is ≥ 75% and Copper mass is ≤ 25% with deionized water as binder. Variations with annealing duration, temperature, and length were observed. Increasing annealing duration at 300 ºC and 500 ºC reveal the peak resistances trend for 10% and 20% copper compositions respectively.*

KEYWORDS: *Cermets, Redox, Temperature Coefficient of Resistance (TCR), Sintering, Annealing.*

INTRODUCTION

Annealing of materials existed for centuries ranging from ancient furnace design to present days forms of furnace. Cermets/Composite resistors are known to be Ohmic at low temperature and are usually less than 20% of metal by volume.

One of the main challenges in the development of electrically conductive composites is the ability to determine the ideal filler concentrations. Insufficient concentrations result in an insulating material, while excess leads to the loss of mechanical properties - Grunlan et al. (2001).

Resistivity has been determine theoretically by some researchers:

Ohm's model:

$$\rho = \frac{R}{l} A \quad ------------------ (1)$$

R is the resistance, l is the length, A is the cross sectional area and ρ is the resistivity.

$$R_{TCR} = \frac{R_2 - R_1}{R_1(T_2 - T_1)} \quad -------------(2)$$

Hopping conduction model:

$$= R_0 T^{1/2} \exp(\frac{T_0}{T})^{\frac{1}{4}} \text{ ---------------------- } (3)$$

Where T_o is a characteristic temperature of the conducting material, T is the cermet temperature, R_0 is the characteristic resistance of the conducting material, and R is the cermet resistance.

The firing sequence (firing time, firing atmosphere and peak firing temperature) and the length of the resistor varied remarkably with resistance/resistivity of the copper-clay cermet resistors -Babaolola and Akomolafe (2003), Babalola and Alabi (2010). Annealing processes gives microscopic variations of structure of the resistors. These also resulted in temperature shift from positive to negative of temperature coefficient of resistance (TCR) – Boyo and Akomolafe (2012). If the annealing time and/or temperature exceed a certain value, the development of interfacial structure leads to the formation of weak layers in the interfacial region and damage the bonds – Abbasi et al. (2001), Hannech et al. (2003), Peng et al. (1999). Finding indicates that, latent radiation damage is activated by annealing, thus causing a decrease in the load transfer capacity at the fiber/matrix interface and, hence, losses in the shear modulus and apparently accompanying a decrease in the ultimate strength of composites - Egusa et al. (1985). Annealing time controls the morphology by influencing the degree of crystallinity in the matrix and in the fiber–matrix interface and this formation restricts fiber–matrix debonding – Yilmaz (2010).

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 annealing. Anything that alters the ratios of the components or the stresses built into the resistor during manufacture such as firing is liable to affect the overall TCR. The sintering properties of the conducting materials are also part 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, 1991, 1994).

Explanation to the factors contributing to the complex behaviour exhibited by thick film resistor systems temperature coefficient of resistance (TCR), size effects, ohmic and non-ohmic resistances, piezoelectric and pyroelectric effects, thermally induced variations, etc were sought by researchers like Wimmer et al (1974), Mizsei and Lantto (1991), Akomolafe and Oladipo (1996), Afronte et al (1997), Kuzy (1997) and Stein et al (1997).

In the study of Iron-Clay composite resistor, annealing schedule is said to affect greatly the properties of cermets or composite resistors due to structural defects like impurity atoms, vacancies, interstitial and grain boundaries that are produced. The conduction in composite resistors is similar to that in cermet film resistor - Akomolafe (1996).

This paper work studies the annealing sequence effect on the resistance/resistivity behavior of copper cermets.

MATERIALS AND METHODS

The materials are copper powder with unique and fine granules of less than 75 µm and 99% pure imported from Aldrich in United State and carefully selected white clay for homogeneous physical properties from eastern part of Ilorin in Kwara State, using deionized water as binder/solvent. The characterization of clay revealed the clay composition and was found to contain dopant in measurable quantity. The copper-clay cermets were fabricated with clay compositions of 95%, 90%, 85%, 80% and 75% mass ratio respectively. A unique diameter of 5mm and lengths 5 mm, 10 mm, 15 mm, 20 mm, and 25 mm were fabricated with the aid of a designed mechanical-hydraulic device. The pressure size applied was about 15 bars (1.5×10^6 Pa) with deionized water as binder. The cermet resistors were allowed to stay for three days in a dust free environment at room temperature.

Annealing of the resistors at 200 ºC for one hour duration was done and resistances were taken for all the compositions with the aid of digital ohm-meter repeatedly. Subsequently, the furnace temperature was raised to 300 ºC – 500 ºC with hourly annealing for 5%, 15%, and 25% copper compositions to allow sintering. The resistance values were also taken after furnace-cooling.

Annealing of 5 mm, 10 mm, and 25 mm length for 10% and 20% copper were done at 300 ºC and 500 ºC respectively with durations of 30 minutes intervals.

RESULTS AND DISCUSSION

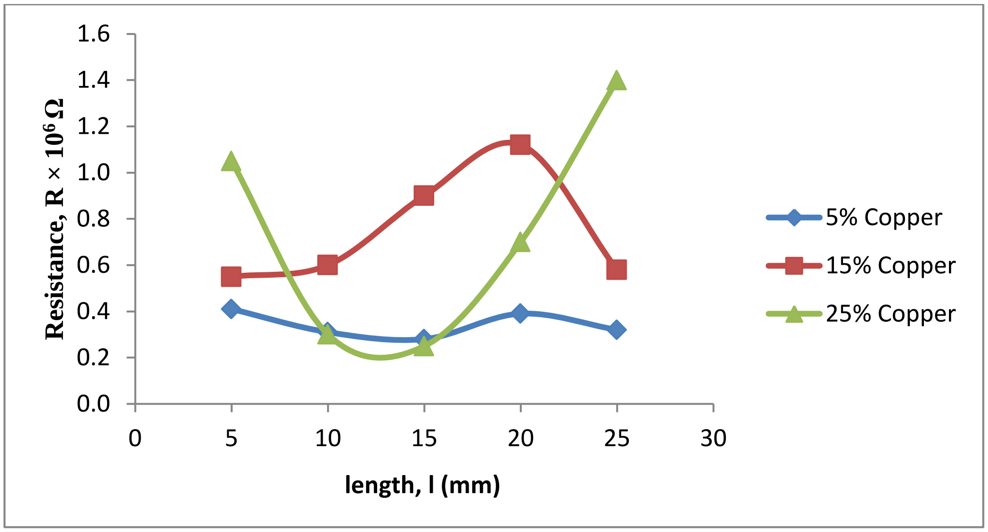

Fig. 1: Resistance against length at 200 °C with one hour annealing.

Fig. 1 shows increament in the resistance value of 15% copper cermet with length before declining at 20 mm length. Annealing as contributed to the result due to rise in temperature which enables sintering to set in for 15% cermets. For 25% copper, exponential shape of the curve reveals inverse size effect emanated earlier due to higher reistance from 5 mm length to that of 10 mm. the furnace oxidation effects may also be responsible.

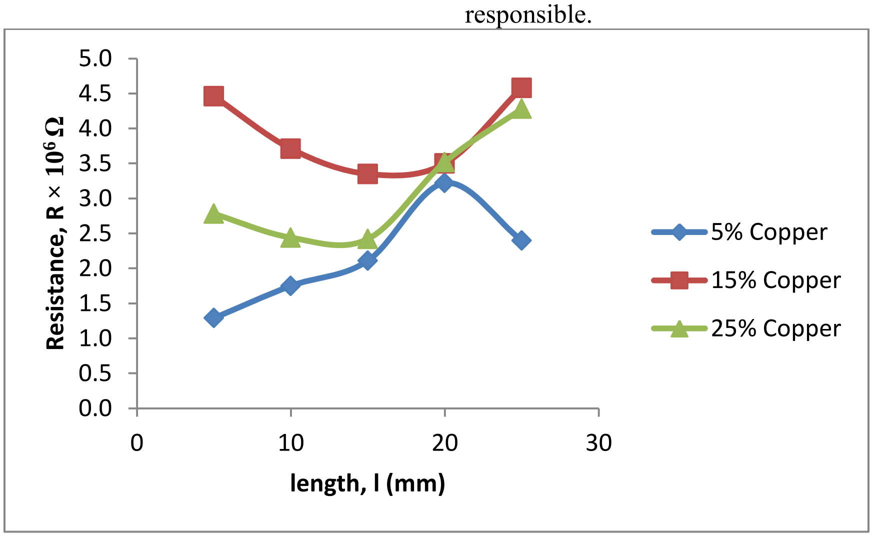

Fig. 2: Resistance against length at 300 °C with one hour annealing.

Fig. 2 reflects a significant response of the cermets to annealing as regards the trends of resistances to length. The earlier decline in the resistance of 15% and 25% copper could be due to fabrication defect, conduction of the constituents impurities and the metallic composites due interfacial weakness in the interfacial region and damaged bond. This significantly shows the size effect which can either be directly or inversely and not both at this temperature. 15 mm length revealed the benchmark length for good cermet fabrication and of ≥15% copper composition which showed inverse size effect. Annealing temperature was not sufficicient at start but with duration tends to be ohmic.

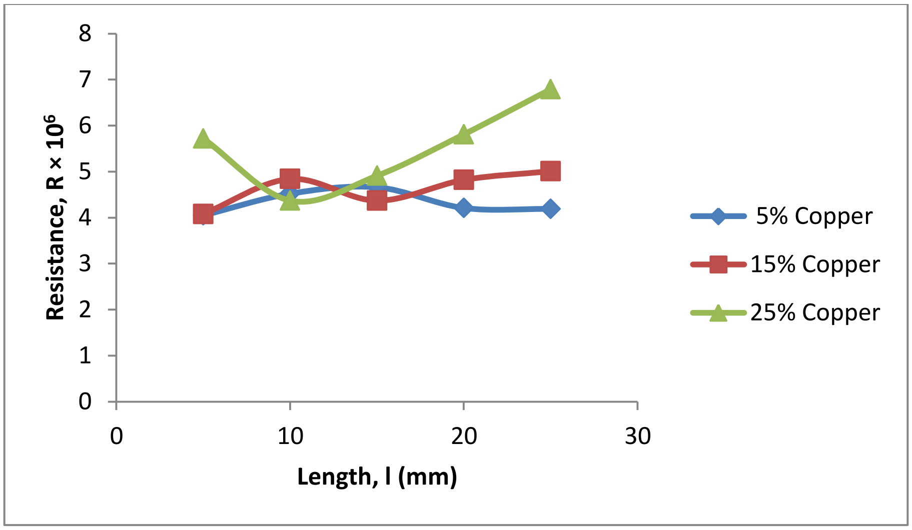

Fig. 3: Resistance against length at 400 ºC with one hour annealing.

Fig. 3 shows intermitent redox reaction for 5% composition as the length increases. This also proves the increasing breakdown of insulating layer when compared with annealing at 300ºC. 25% Copper composition has a progressive trend with threshold resistance's increase. This threshold increament is shown by the cermets and could be due to thick insulating layer formed in the cermet. 15% composition of Copper depicts closeness to the peak resistance. Annealing has been able to break the insulating phase and hardened the strength of cermets with conductivity enhancement. As the temperature increases the threshold resistance increases and moves closer to the peak.

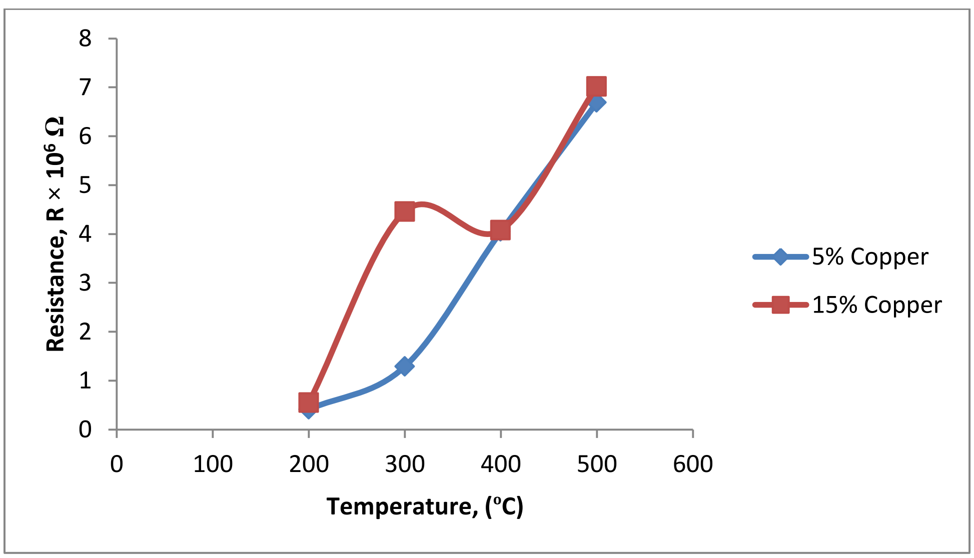

Fig. 4: Resistance against Temperature for 5mm with one hour annealing.

Fig. 4 reflects the positive trend of resistance with increase in temperature. This depicts positive temperature coefficient of resistance (TCR) for one hour annealing. 15% copper composition shows slight decline between 300 ºC and 400 ºC, this could be due to furnace gas effect that may have contributed to little breakdown in the insulating phase of the cermet.

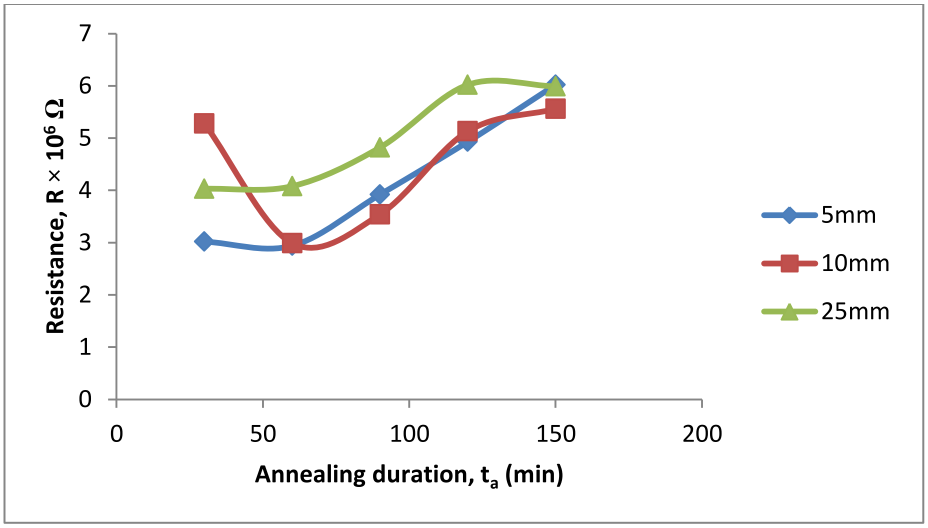

Fig. 5: Resistance against Annealing duration for 10% Copper at 300 ºC.

Annealing duration effect at 300 ºC temperature in Fig. 5, shows the peak of resistance and beginning the shift in temperature coefficient of resistance (TCR) from positive to negative. This result reveals the annealing effect on the conductive grains that contributes to the conduction.

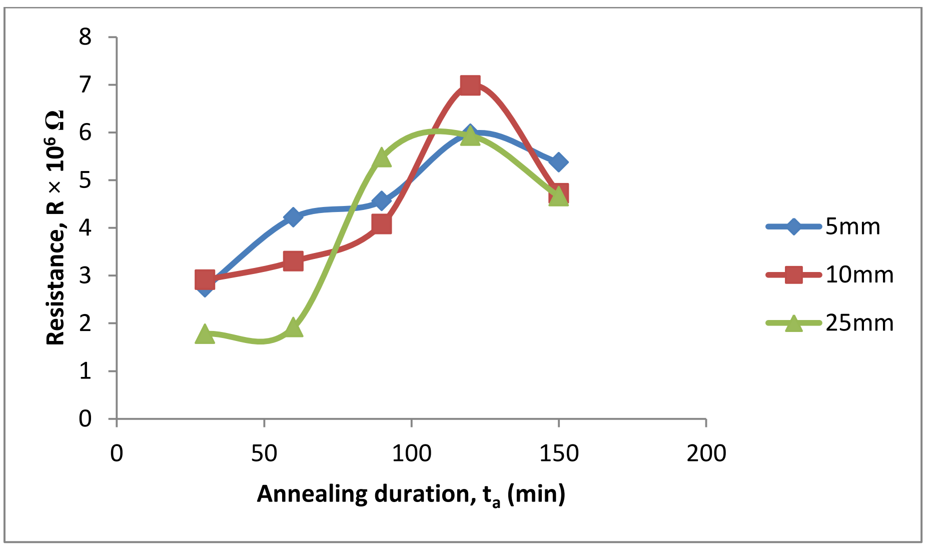

Fig. 6: Resistance against Annealing duration for 20% Copper at 500 ºC

Fig. 6 shows that the conduction of constituent particles due to sintering also increased the diffusion rate of conducting ions. Thus, annealing duration enhances the diffusion rate. At 300 ºC and 500 ºC, in Figures 5 and 6 respectively, they both show that the increase in temperature with annealing duration brought about threshold and resulted in the negative temperature coefficient of resistance (TCR).

CONCLUSION

Increasing annealing duration and temperature shifted the trend of temperature coefficient of resistance from positive to negative. This shows the effect of annealing that allows movement of conducting elements as temperature increases and makes the fabricated cermets resistors function effectively at a regulated duration of about 120 minutes and peak temperature 400 ºC irrespective of length. Annealing also reduced the fabrication defect as temperature increases due to sintering. The cermet will function best with a regulated-time delay system. The more the length and metal matrix composition, the higher the threshold resistivity attained as shown in Figs. 3 and 5.

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