Evaluation of Ohmic Peak Characteristic of Metal/Clay Based Cermet

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Abstract: The Ohmic peak of copper/clay based cermet is evaluated to know the Ohmic boundary and response of the fabricated cermet resistors, using electrically conducting and chemically active copper powder with mass 5%, 15% and 25% using Archimedes' principle. Considering annealing temperatures ranging from 200 °C to 500 °C. Some compositions exhibit inverse size feature earlier or later. The results showed that, the threshold resistivity for copper compositions gets closer to peak from 400 °C while the 25% copper exhibited higher resistance peak than the lower compositions.

Keywords: Intercalate, Percolation, Annealing, Ohmic, Threshold.

I. INTRODUCTION

 \mathbf{T} oday, ceramics are routinely implemented in the combustor part of the jet engines because the blade provides heat resistant chamber [1]. Cermet consist of a thorough mixture of a ceramic hard phase and a suitable metal binding phase [2] that enables Ohmic property (Ohm's law $R = \frac{\rho L}{A}$, R is resistance, ρ is resistivity, L is length and A is the cross sectional area). The resistance/resistivity of the metalclay cermet resistors varied remarkably with conductive grain content, firing sequence (firing time, firing atmosphere and peak firing temperature) and the length of the resistor [3], [4], [5]. Exchange and redox reactions make the microstructure and electrical properties of the insulating layers very variable according to the firing condition [3], likewise re-firing which resulted in temperature shift from positive to negative of temperature coefficient of resistance (TCR) [6]. Cermet structured resistors, like thick film resistors (TFRs), exhibit a high stability and very low TCR [7]. The TCR of a thick film composite is determined by the separate temperature coefficient of its conducting constituents in their final form after annealing [8], [9]. Hence, the conduction in composite resistor is similar to that in cermet film resistors [10]. Annealing sequence is said to affect greatly the electrical properties of cermet/composites due to structural defects like impurity atoms, vacancies, interstitial and grain boundaries that are produced [7], [11]. 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 [12]. The change in the electrical properties of cermet is due to physical, chemical and mechanical changes within its structure which is responsible for the strong cermet's resistance dependence on applied voltage at low annealing temperatures [13]. Thus,

conduction takes place at threshold when percolation must have occurred due to increased temperature and/or duration that enhanced the diffusion rate of conducting grains [5], [14]. Either of the two types of percolation transitions is a characteristic feature of composite materials which are in lowviscous state during the manufacture [15]. Unlike the dynamic percolation, the optimal value of percolation concentration cannot be changed by varying the manufacturing conditions of the composite because it is defined by statistical percolation theory with threshold that is defined by the conductive filler type, its aspect ratio, surface state of polymer, uniformity in the distribution of the metal filler and the content in polymer matrix [16]. Cermet materials with high and negative values of TCR have properties which are comparable to those of thermistors [10], [17].

This paper evaluated the Ohmic characteristic of copper-clay based cermet by considering length and compositions within a range of temperatures.

II. METHODOLOGY

The fabrication of the cermets were done using copper powder with unique, fine granules of 99% purity, particle size (75 µm) imported from Aldrich in United State, and clay from Ilorin East of Kwara State with compositions of 95%, 85% and 75% mass respectively, using volume displacement method (Archimedes' principle). A unique diameter of 5 mm and lengths 5 - 25 mm in steps of 5 were produced by compaction. The pressure size applied was about 15 bars $(1.5 \pm 0.01 \text{ MPa})$ with deionized water as binder. The cermet resistors were allowed to stay for three days in order to be strong and well dried in a dust free environment at room temperature. The resistors were annealed at 200 °C for one hour duration and resistance values were taken for all the compositions. Subsequently, the furnace temperature was raised to 300 °C – 500 °C with hourly annealing for 5%, 15%, and 25% copper compositions. The resistance values were also taken after furnace-cooling.

III. RESULTS AND DISCUSSION

3.1 Results

The results gotten after annealing at different temperatures using a unique diameter and varying lengths for the three compositions are as shown below.



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



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



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



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

3.2 Discussion

Fig. 1 shows sintering setting in for 15% copper composition showing response to ohm's law with minimum resistance, R_{min} , of 0.55 Ω and maximum resistance, R_{max} , of 1.12 Ω which is the optimal. This is attributed to the effect of heterogeneous nature of the constituents and oxidized impurities that intercalate the insulative layer. For 25% copper, exponential shape of the curve revealed inverse size effect emanating due to higher resistance from 5 mm length to that of 10 mm which is non-ohmic. The peak/maximum resistance at this temperature is 1.40 Ω and minimum of 0.25 Ω . This trend disobeys Ohm's law, while 5% copper composition did not show any significant effect of length on resistance. The elemental composition of clay and redox effect must have contributed to these responses.

Fig. 2 reveals the trends of resistances to length which is direct by Ohm's law but shows inverse size property. Compositions with 15% and 25% copper showed negative sinusoidal trend in resistance. The earlier decline in the resistance of 15% and 25% copper could be as a result of the constituent grains conduction of the cermets due to insulation absence. This significantly shows the size effect which can either be directly or inversely and not both at this temperature. The minimum resistance/resistivity at this temperature is seen to be higher than the maximum for 200 °C for both compositions (25% has R_{min} of 2.42 Ω and R_{max} of 4.28 Ω , while 15% has R_{min} of 3.5 Ω and R_{max} of 4.5 Ω). The Ohmic rule is expected to be obeyed by any composite resistor or cermet at temperatures below threshold.

Fig. 3 shows dynamic percolation for 5% and 15% composition with increase in the length. This reveals the peak of insulating breakdown with annealing rising from 300 °C to 400 °C. 25% copper composition has a progressive trend with threshold resistance's increase from 4.91 Ω and peak value of 6.79 Ω . Despite this, it is observed that the gap between the start and the threshold is being reduced as temperature increases. This threshold rise 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 set dynamic phase and hardened the strength of cermet with conductivity enhancement while 25% copper is yet to peak.

Fig. 4 indicates the positive trend of resistance with increase in temperature. This shows positive TCR for one hour annealing. It significantly shows that 5% copper composition will experience a higher annealing temperature before the insulating bond can be broken. On the other, 15% composition of copper tends to have insulation breakage between 300 °C and 400 °C. This might be due to defects in the resistor, inhomogeneity of the clay particles size or furnace gas effect that results in temporary conduction. The TCR of a thick film material is determined by the separate temperature coefficient of its conducting constituents in their final form after annealing [8], [9], [14].

IV. CONCLUSION

The peak characteristic of each composition indicates the optimal performance of that particular composition and the deformation effect during fabrication is reduced as sintering is increased. All lengths were noted to perform optimally and TCR of these cermets is stable with copper composition up to 15% as shown by Figs. 1 to 4 and conductive grains and impurities concentrations of the constituents contribute to their performances which also make them comparable to convectional resistors. Fig. 3 shows that 25% copper will attain peak resistivity at higher temperature than others and the TCR will be positive. The cermets are capable of functioning as a resistor to optimal temperature of 500 °C effectively and the peak can be raised higher by dissolution of clay in polar solvent for hours for extraction of some conducting grains in the clay formation.

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