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Microstructural and electrical properties of thick film resistors on oxide/oxide ceramic–matrix composites

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Abstract

The microstructural and electrical properties of thick-film resistors (R-311-A, R-312-A and R-313-A, ESL) fired on oxide/oxide composites were investigated with the purpose of determining the compatibility of these resistor materials with the new substrates. Normally, these resistor materials were developed for firing on alumina ceramic. Possible interactions between the thick-film resistors and the substrates were studied by scanning electron microscopy and energy-dispersive X-ray analyses. The sheet resistivities and resistance–temperature characteristic of the resistors fired on different substrates were measured. The results indicate that interactions between the alumina substrate and the resistor layer were not observed. However, at the interface between the composites (matrix) and the resistor layer an obvious transition layer is detected. In addition, the sheet resistivities on composites substrates are generally lower than that on alumina. Moreover, the resistors fired on alumina show a positive resistance–temperature characteristic (TCR) from 25 °C to 500 °C, while the TCRs for the resistors fired on the composites substrates are negative. Microstructural analysis and thermal expansion coefficient testing show that the underlying substrate strongly influences the final characteristics of the resistors in two ways: through chemical interaction and thermal expansion.

Keywords: B. Composites; C. Electrical properties; E. Substrates; Resistors

1. Introduction

Thick film resistors (TFRs) are made by screen printing thick film resistor pastes on insulating, mainly alumina, substrates. After printing and drying, the resistor pastes are fired in a belt furnace. Fired thick-film resistors basically consist of a conducting phase in an insulating glass matrix. The ratio of conductive to glass phases roughly determines the specific resistivity of the resistor [1]. During the firing cycle, the conductive phases of the resistor materials interact with the glass phase, forming conductive networks through the sintered layers [2–4]. On the other hand, the melted glass also interacts with the substrate [5–9]. The final electrical characteristics of the resistors are the result of these reactions [10].

Oxide fiber and reinforced oxide (oxide/oxide) composites, which exhibit low density, high electrical resistivity, excellent dielectric properties, high temperature oxidation resistance, and fine thermal shock damage resistance, have replaced the monolithic alumina ceramic (inherent brittle) in many applications [11–13]. Hence, adopting oxide/oxide composites to the thick film resistors may be a daring and interesting attempt, which can widen the family of thick film resistors substrates.

However, as the thick film resistors were developed for firing on alumina substrates, their compatibility with oxide/oxide composites needs to be evaluated.

2. Experimental procedures

2.1. Preparation of oxide/oxide composites

The reinforcement used in the oxide/oxide composites was Nextel^{TM} 440 continuous ceramic oxide fibers (from 3M

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Company, USA). The oxide fiber preforms were 2D-sewed by 15 layer oxide fiber fabrics using the same Nextel™ 440 oxide fiber yarns and the fiber volume fraction was about 41.5%. The diphasic Al_2O_3–SiO_2 sols (from Suzhou Nanodispersions Co., Ltd., China) were used as the precursor of oxide ceramic–matrix. The weight ratio of Al_2O_3 to SiO_2 particles is 1:1 in the sols. Subsequently, the 2D oxide/oxide composites were fabricated through a conventional sol–gel process. The detailed process could be found in Refs. [14–16]. The typical characteristics of the prepared oxide/oxide composites and 96% alumina ceramics are listed in Table 1. At last, the oxide/oxide composites were ground and polished for thick film resistors substrates application. The roughness R_a of the substrate surface is about 3–6 μm, while the thickness is 3 mm.

2.2. Preparation of thick film resistors

Thick film resistors were produced on two substrate materials: the prepared oxide/oxide composites substrates and 96% alumina as standard thick-film substrates. For this study, industry standard thick film pastes are selected. The low migration Ag/Pd conductor paste (Dupont 6177T) was used for electrode terminals, while pastes of three types of sheet resistivity, namely R-311-A (10 Ω/sq), R-312- A (100 Ω/sq) and R-313-A (1 kΩ/sq) ESL, were selected for resistor materials. The conductive phase in these resistor materials is based on RuO_2. And the glass phase contains asmain elements, lead, silicon, aluminum and barium oxides. Boron oxide, which is also present in the glass phase, cannot be detected in the EDS spectra. Subsequently, resistor layers were printed, dried and fired (PDF) after the electrode patterns were PDF in accordance to the manufacturer's instructions, to avoid an inter-diffusion of silver with the TFRs. Moreover, the thick film resistors with different substrates were fabricated strictly following the same processing parameters, so that a similar thickness was achieved.

2.3. Analytical methods

For microstructural investigation, the resistors fabricated on alumina and those fabricated on oxide/oxide composites substrates were mounted in epoxy in cross-sectional orientation and then cut and polished using standard metallographic techniques. A JEOL JSM-5800 scanning electron microscope (SEM) equipped with an energy dispersive X-ray analyser (EDS) was used for overall microstructural and compositional analysis. Prior to analysis in the SEM, the samples were coated with gold to provide electrical conductivity and to avoid charging effects.

Table 1
The typical characteristics of the prepared oxide/oxide composites and 96% alumina ceramics.

Properties	96% alumina	Oxide/oxide composites
Density (g cm^{-3})	3.7–3.8	2.2
Flexural strength (MPa)	300	66.17
Young's modulus E (GPa)	250–330	24.3
Thermal expansion coefficient (K^{-1})	7.0×10^{-6}	5.3×10^{-6}
Thermal conductivity (W mK^{-1})	20–24	6
Dielectric constant	9–10	3.5–4
tg δ	0.01	0.005

The sheet resistivities of the resistors were measured with a Keithley 2001 Digital Multimeter 7 1/2 Digit DMM. The resistance–temperature characteristic of the resistors was measured by fixing it with a special alumina clamp and connecting it to a computer controlled multi-meter (KELTHLEY 2700 switch system and relay I/O card) through two pieces of platinum wires. A programmable furnace was used to heat the resistors at 10 °C/min to temperatures ranging from 25 °C to 500 °C. Five samples were tested to obtain the average value.

In order to understand the thermal expansion behavior of the resistor layer on the substrate, the R-311-A resistive paste was selected and dissolved in acetone first. When it dries, the obtained powders were sintered to a dense block under 850 °C for 10 min through a Spark Plasma Sintering process (FCT HPD5, Germany). The heating rate was 100 °C/min. Subsequently, the thermal expansion coefficients (TECs) of the alumina ceramic, oxide/oxide composites and the R-311-A SPS sinter were determined using a dilatometer (Netzsch DIL 402E, Germany). The tested samples were cut into rectangular pieces (25 mm × 5 mm × 5 mm). The measurements of the sample expansion vs. temperature were made from 25 °C to 500 °C with temperature intervals of 10 °C.

3. Results and discussion

Table 2 shows the sheet resistivities of the resistors fired on the alumina and oxide/oxide composites. As shown, the sheet resistivities of the resistors on alumina substrates are slightly higher than the ones on oxide/oxide composites.

In order to facilitate comparison of the resistance–temperature characteristic of resistors, the resistance values obtained in the experiments were normalized to the resistance value of each thick film resistor in the test, as follows:

$$R_{normalized} = \frac{R(T) - R_{25}}{R_{25}} \quad (1)$$

where $R(T)$ is the measured resistance at the temperature T and R_{25} is the resistance value at the room temperature.

Fig. 1 is a plot of resistance values after normalization. As shown, the resistors fired on alumina show a positive resistance–temperature characteristic from 25 °C to 500 °C. However, the resistance–temperature characteristic for the resistors fired on the composites substrates is negative. In addition, the normalized resistance of resistors with different sheet resistivities shows a different sensitivity to temperature.

Table 2
Sheet resistivities of the resistors fired on different substrates.

Resistor	96% alumina	Oxide/oxide composites
R-311-A	11.74 ± 0.24	7.79 ± 0.85
R-312-A	116.43 ± 18.60	77.41 ± 24.18
R-313-A	1064.95 ± 49.14	851.35 ± 57.06