COMMUNICATIONS

METHOD OF MEASUREMENT OF THERMAL CONDUCTIVITY OF RESISTIVE LAYERS IN THICK–FILM STRUCTURE

Włodzimierz Kalita * — **Mariusz Węglarski** * — **Stanislav Slosarčík** **

Resistive layers are usually the main heat sources in thick-film structures. For accurate analysis of stationary and non-stationary temperature fields in structures of this kind it is necessary to know the thermal conductivity of resistive layers printed on the ceramic substrate. An unconventional method of measurement of the thermal conductivity of the resistive layer and transient region between the layer and substrate has been described. The results of measurements of the conductivity with the use of scanning electron microscopy for identification of the transient region and X-ray diffraction for measurement of temperature into the substrate have been presented.

K e y w o r d s: hybrid microcircuits, thermal conductivity, thick-film resistor, simulation of temperature states

1 INTRODUCTION

A resistor made in thick-film technology in principle consists of two layers. The first is the resistive (active) layer, which determines the thermal properties of the element. The second layer - substrate - plays a protective and isolation role as well as it takes part in heat dissipation. Moreover, as a result of interaction between these two layers in the firing process a transient region (with different properties) is observed. The resistive layer has structure dissimilarity in the transient region in direct neighbourhood with the substrate (Fig.1).

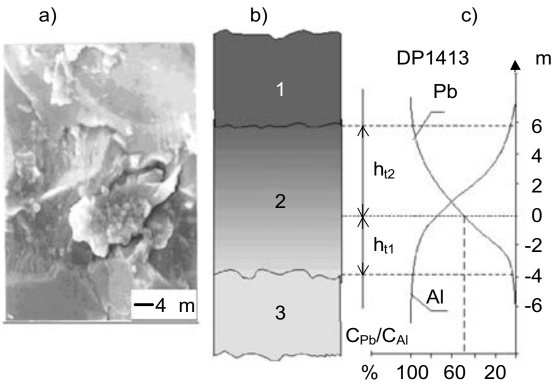

Fig. 1. Transient region: a) after firing (1-resistive layer, 2-transient region with PbO Al_2O_3 B_2O_3 SiO_2 glaze, 3-substrate, b) distribution of Al and Pb

This is the effect of partial dissolving (in the firing process) of the substrate material in the resistive layer glaze (10-15% Al_2O_3 for corundum substrate) and penetration of this glaze in the substrate structure. This mutual penetration of material layers is heterogeneous and takes place to a depth of $10\,\mu$m [1]. So, this value is relatively high in relation to layer thickness. Because of significant structural differences of the transient layer (relatively to the resistive layer or substrate) it is necessary to determine the thermal resistance of this region. The thermal resistance of the resistive layer printed on the corundum substrate has two components: thermal resistance of the main layer and thermal resistance of the transient region.

2 THERMAL CONDUCTIVITY IN TEMPERATURE FIELD ANALYSIS

For determining the heat resistance (Fig. 2a) the Waniczew method of elementary balances was used. It consists in heat balancing in single separate elements of the thick-film device. Because the dimensions of layers areas are much bigger than their thickness ($hr << a$, $hr << b$, $hs << a$, $hs << b$) it can be assumed that the sides of elementary regions are adiabatic areas and heat flow Q is along z-axis only($\partial T/\partial x = \partial T/\partial y = 0$) (Fig. 2b), as well as the principal layer region is homogeneous and isotropic as regards thermal properties. With those assumptions the boundary problem can be treated as 1-D [2].

Flux Q, which penetrates from the substrate trough the resistive layer (with thermal resistance R, area S and thermal conductivity λ; no generated heat) creates a temperature distribution in the cross-section (along z-axis) illustrated in Fig. 2c. The temperature differences $T_2 - T_1$ and $T_3 - T_2$ are expressed by

$$\begin{aligned} T_2 - T_1 &= QR_r, \qquad \text{where} \qquad R_r = \frac{h_r}{S\lambda_r} \\ T_3 - T_2 &= QR_t, \qquad \text{where} \qquad R_t = \frac{h_t}{S\lambda_t} \end{aligned} \tag{1}$$

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Equation (1) can be the basis to experimental estimation

$$q = -\lambda \nabla T, \qquad \text{where} \qquad \nabla T = \frac{\partial T}{\partial z} \tag{2}$$

of the R_r and R_t value but determination of temperature T_2 (directly over transient layer) is a big problem.

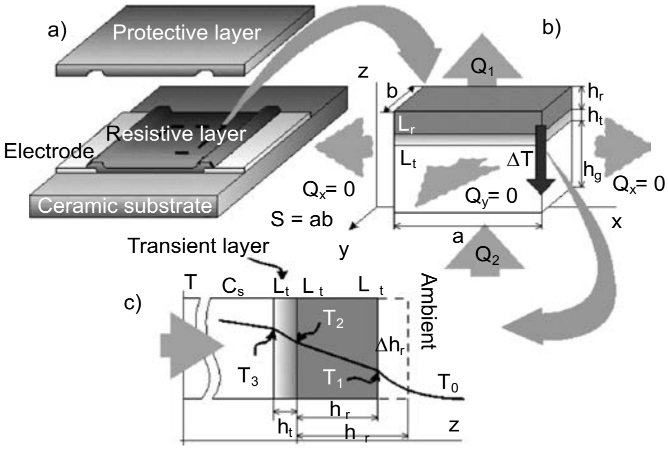

Fig. 2. Resistor in thick-film: a) spatial configuration, b) heat flow, c) temperature profile (T_1 - surface temperature, T_2 - temperature resistive layer directly over transient region, T_3 - substrate temperature directly under resistive layer

Also knowing the material thermal conductivity is insufficient because of geometrical-structural heterogeneity of the transient region. For those reasons two circuits with different thickness of resistive layer h'_r and h''_r have been considered to determine the above mentioned thermal resistances. It was assumed that in both cases the heat flux as well as temperature of layer surface have the same values: $Q' = Q'' = Q$; $T'_1 = T''_1 = T$ (indexes $'$ and $''$ refer to particular values of layers with thickness h'_r and h''_r, respectively). From the systems of equations (1) for h'_r and h''_r the following expression can be obtained

$$T'_3 - T''_3 = Q(R'_r - R''_r) \tag{3}$$

which after taking account the relation $R''_r = R'_r \cdot h''_r / h'_r$ leads to equation

$$R'_r = \frac{h'_r\,(T'_3 - T''_3)}{Q\,(h'_r - h''_r)} \tag{4}$$

The measurement of temperature T'_3 and T''_3 (on the substrate under resistive layer) and determination of the heat flux power on the basisi of the product $Q = g \cdot S$ (g - is power density) allows to calculate the heat conductivity

$$\lambda_r = \frac{h'_r - h''_r}{T'_3 - T''_3} g \quad \text{where} \quad \lambda'_r = \lambda''_r = \lambda \tag{5}$$

$$R_t = \frac{h'_r\,(T''_3 - T_1) - h''_r\,(T'_3 - T_1)}{Q\,(h'_r - h''_r)} \tag{6}$$

$$\lambda_t = \frac{g\,h_t\,(h'_r - h''_r)}{h'_r\,(T''_3 - T_1) - h''_r\,(T'_3 - T_1)}$$

After similar transformations of equation (1) expression can be established which describes the thermal resistance of the transient region.

3 EXPERIMENTS

Expressions (5) and (6) are the basis for determining the thermal resistance value of the resistive layer referred to the substrate. It requires establishing of the values h_r and T_1. In the experimental investigations the layer thickness h_r was established using the gravimetric method [3], which guarantees high accuracy with taking into account irregularity of the surface. Temperature T_1 was measured using a point IR method. Another problem is the measurement of substrate temperature under resistive layer T_3. A special test method has been elaborated on the basis of X-ray diffraction. It is based on the determination of the changes of crystal lattice in the substrate material (using diffraction method) under temperature changes. It requires establishing X-ray penetration depth in the tested region and layer thickness (which is the source of information in the form of diffraction lines). The calibration of the diffractometer should be also made before tests. It consists in establishing of a unique dependence between the diffraction lines and temperature value of the examined material layer. The intensity of the radiation beam I_z after transition through the material layer with thickness z can be described by formula [4]

$$I_z = I_0 e^{-\mu z} \tag{7}$$

where I_0 means the intensity of the incident beam and μ is the coefficient of linear absorption. Analogous methods of μ coefficient determination are not useful for a heterogeneous structure of the resistive layer. For those reasons an indirect experimental method was used. It consists in measuring the intensity of diffraction lines I_{zs} from the substrate without a resistive layer and measuring the same line I_{zr} with the resistive layer (Fig.3).

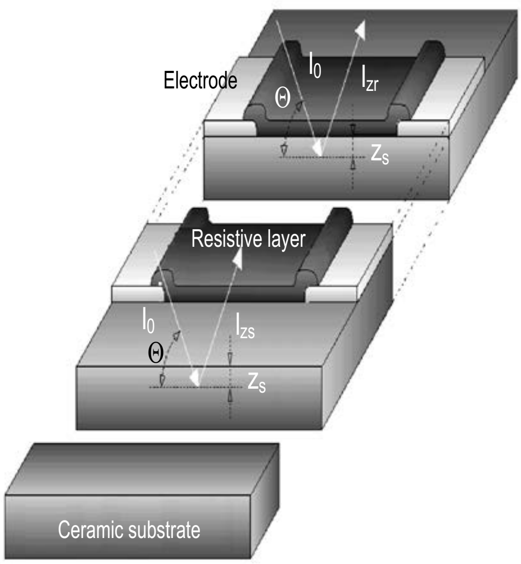

Fig. 3. The idea of measurement of linear absorption coefficient

The intensity of the diffraction line can be determined on the basis of expression (7) and for the first and second case is given by equations

$$I_{zs} = I_o \exp\left(-\frac{2\mu'}{\sin\Theta} z_s\right) \tag{8}$$

$$I_{zr} = I_o \exp\left(-\frac{2\mu'}{\sin\Theta} z_s - \frac{2\mu}{\sin\Theta} z_r\right)$$

where $|Theta$ means the Bragg angle, μ is the linear absorption coefficient of the substrate material, z_s is the depth of penetration of radiation in the substrate and z_r is the known thickness of the resistive layer. Simple transformations of equations (8) lead to formula

$$\mu = \frac{\sin\Theta}{2z_r} \ln \frac{I_{zs}}{I_{zr}} \tag{9}$$

which allows to determine coefficient μ from diffraction measurements of I_{zs} and I_{zr}. The measurement of the linear absorption coefficient allows establishing the effective depth of penetration of radiation in resistive layer and substrate from equation

$$G_z = 1 - \exp\left(-\frac{2\mu}{\sin\Theta} z\right) \tag{10}$$

which describes part G_z of the total intensity of radiation beam from the material layer with depth z. It allows to estimate the thickness of the resistive layer and diffraction angles where are possible reflections from the substrate carrying information about the temperature directly under layer.

The above-mentioned analysis is the basis to using the X-ray diffractometer for temperature measurements. The temperature change causes a volume change of the crystal lattice cells in the analysed region. As a result, displacement of diffraction lines is observed. For this reason the measurement of temperature T_3 was based on the determination of displacement of one diffraction line. It required calibration of the diffractometer for the mentioned line in accordance with expression

$$\Theta = f(T) \tag{11}$$

Experimental investigations for determination of the heat resistance of the resistive layer consist of four steps: preparation of samples, calibration of the diffractometer, measurement of temperature in samples and establishing of heat resistance.

For the laboratory tests three resistive compositions based on ruthenate (with different gravimetric compositions and sheet resistances) were selected (Tab.1).

Table 1. Compositions of samples

No	Ruthenate %	Glaze %	R_0 kΩ	μ cm^{-1}
1	49	51	0.3	1000
2	37	63	1.7	960
3	18	82	230.0	940

For those compositions the linear absorption coefficient and thickness of investigated layers have been determined. After this, the depth of radiation penetration (for selected line with deflection angle $2\Theta = 150$ deg) in layers on the basis of particular composition was established (Fig.4).

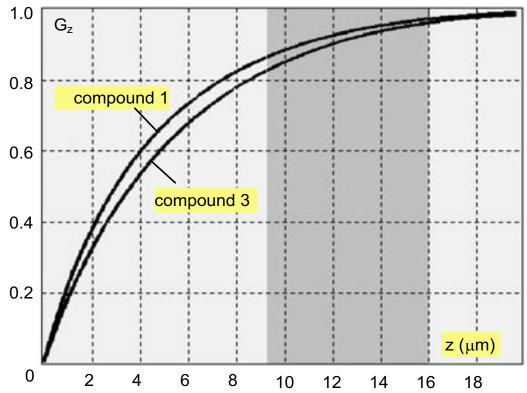

Fig. 4. G_z *vs* z for composition with μ from Table 1 and $2\Theta = 150$

Calibration of the diffractometer was carried out using a high temperature unit. It allowed determining the dependence (11) for the above-mentioned line. On a substrate with dimensions $30 \times 50 \times 0.6$ mm resistive layers with area 25×39 mm and thickness 9 to16 μm were printed. From one side of the substrate the layer was used for measuring, from the other side was the source of heat flux penetrating through the investigated area (Fig.5). The heat flux with the same power was obtained by ensuring the same temperature ($T_1 = 425$K) on the surface. It was dependent on power P (about 7W) supplied to heating layer 3 controlled using IR and compensation thermometers.

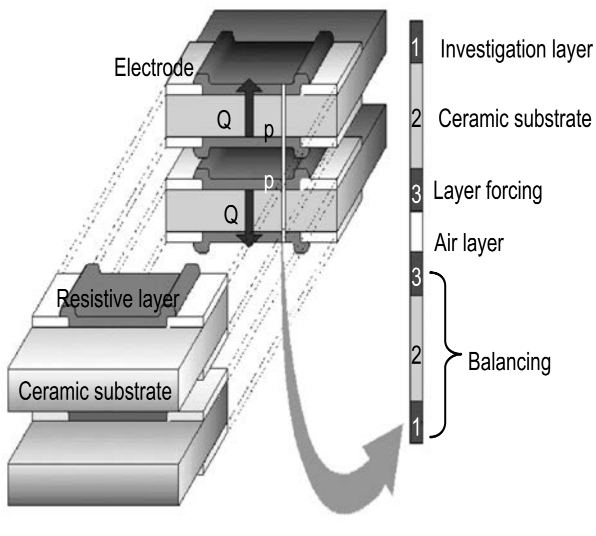

Fig. 5. Idea of making of the test samples

The symmetry of the tested samples allowed direct flux from layer 3 to investigated area.

The substrate temperature T_3 (directly under resistive layer) was established on the basis of dependence (11) determined during scaling of the diffractometer. The obtained results of calculations and measurements are presented in Tab.2.

Table 2. Values of the thermal conductivity and thermal resistance (results are arithmetic average of the measurements. Measurement area $S = 32 \times 10^{-6}\text{m}^2$

No	λ_r W/(mK)	R_t K/W	R'_r K/W	R''_r K/W	h'_r μm	h''_r μm
1	0.16	0.10	3.0	1.9	15.5	10.0
2	0.18	0.08	2.5	2.1	14.3	12.4
3	0.28	0.16	12.6	11.0	15.6	13.7

Because the value of thermal conductivity λ_r of resistive layer is established on the basis of dependence (5), the maximal relative error was assumed as the accuracy of measureing the average value (it is equal to 1%).

4 CONCLUSIONS

The analysis of value λ_r for particular resistive compositions (with considerable differences of the sheet resistance) leads to conclusion that the heterogeneous structure - to a large extent - equalizes their thermal properties in macroregions. The existence of a separate transient layer (with thermal conductivity about two times higher than the resistive layer) was confirmed. Two phases are interpenetrating: from one side - glaze, ruthenate and air, from the other - Al_2O_3 ceramic.

The obtained results confirmed usefulness of the presented X-ray method of temperature measurement for determination of the thermal resistance of resistive layers. It plays a very important role in the identification of heat exchange processes in hybrid microcircuits. The used method has a universal character and can be also applied in other branches of technology.

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