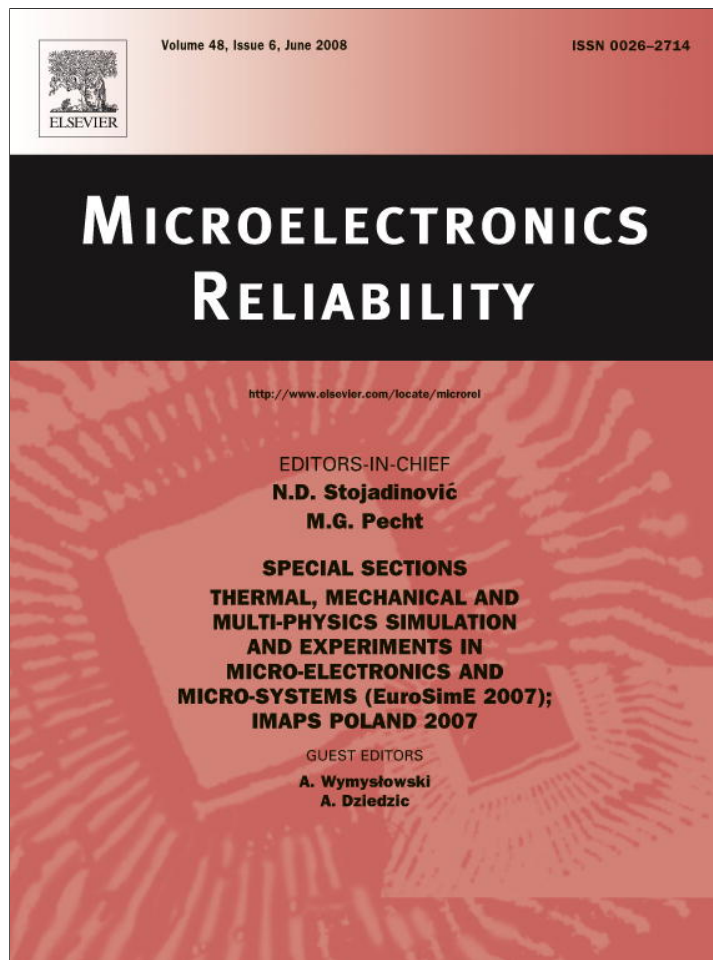


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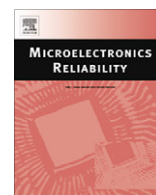
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Evaluation of conductive-to-resistive layers interaction in thick-film resistors

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ABSTRACT

Low-frequency noise spectroscopy is used to examine the interactions between resistive and conductive films that take place during thick-film resistor (TFR) fabrication. Two noise parameters are introduced to quantitatively describe the strength of these interactions. They refer to intensity and repeatability of the noise generated in the resistor interfaces. Extensive experimental studies performed on ruthenium dioxide and bismuth ruthenate TFRs terminated with gold, platinum–gold, palladium–silver and platinum–silver contacts from various manufacturers allow to establish criteria of pastes compatibility and to evaluate compatible systems of pastes for standard “on-alumina” and low-temperature co-fired ceramic (LTCC) resistors. It is found that gold contacts form low-size-effect, stable, low-noise interfaces both with ruthenium dioxide and bismuth ruthenate TFRs. Silver-containing terminations can be used with bismuth ruthenate but not with ruthenium dioxide resistors. Manufacturer optimized system of pastes for LTCC technology works best when used to produce high-resistive, co-fired devices.

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1. Introduction

In thick-film resistors (TFRs) the interactions of the resistive film with conductive terminations often result in dimensional effect and are far from being well understood. In the exemplary case of RuO₂-based film with Ag-containing terminations the increase of resistivity near the contacts extends up to millimeter in-depth of resistive layer [1,2]. Change of local resistivity is barely the first order effect reflecting overall chemical reactions between materials that form the interface. Higher order effects such as contact homogeneity and density of time varying defects can be inspected using low-frequency noise as a diagnostic tool [3], in particular noise spectroscopy [4]. Our recent measurements reveal that low-frequency noise of TFRs is dominated by *thermally activated noise sources* (TANSs) whose density and/or intensity increases in the film-termination interface [5]. Experiments show also that TANSs, especially those located in the interface, are subjected to the *switching phenomenon* which abruptly changes their contribution to the overall noise. Extensive experimental studies that consider the influence of various parameters of the fabrication process, sample geometry, substrate and operation exposures suggest that the most likely origin of the switching phenomenon is relaxation of mechanical stress which in TFRs appears due to the mismatch of the thermal expansion coefficients of the materials contained in resistive and conductive layers and the resistor's substrate. Therefore, the switching is observed in thermal cycling experi-

ments. It is connected with microstructural fluctuations that redistribute local currents and make noise non-stationary on the time scale of the experiment. Switching often appears in burst of pulses (PS) after which the system (noise level) returns to its initial state.

In the paper we explore this research further with the aim of quantitative description of the quality of resistive-to-conductive film interfaces made of various pastes in various technological conditions. Our data add new criteria to the description of compatible systems of pastes used in TFRs fabrication. These criteria are especially important for long-term stability and reliability of these passives.

2. Experimental method

Experimental method to be used in this study was described in the previous paper [5]. Noise spectroscopic data were gathered in temperature range 77–300 K and are presented in the form of noise-power-of-resistance-fluctuations-in-frequency-decade (δR^2), versus temperature T , measured for different regions of multiterminal TFR. Example plot of $\langle \delta R^2 \rangle(T)$ and shape of samples used in the experiment are shown in Fig. 1a and b, respectively. Maxima in the plots of Fig. 1a are produced by TANSs. It is evident that number and magnitude of these maxima strongly increases when noise signal is taken from resistor's terminations (1 and 7). One can also notice that positions of the maxima are different for different regions of the resistors. So, comparing the magnitude of noise generated in different part of the resistor in some specific temperature gives only limited information. Much more appropriate is the integral

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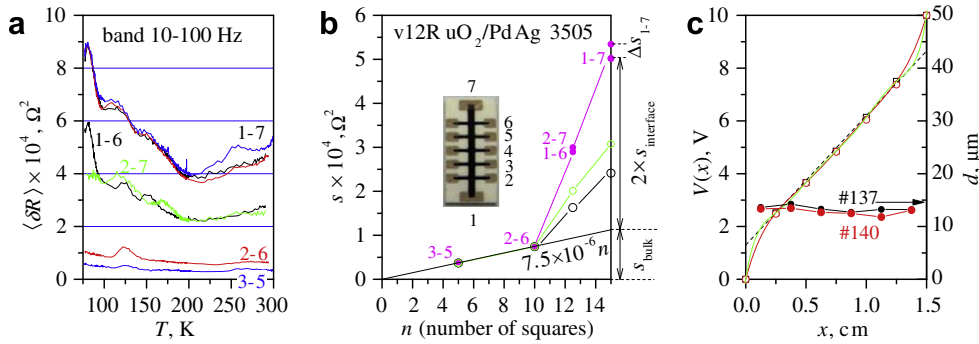


Fig. 1. (a) Mean square resistance fluctuations in the band 10–100 Hz versus temperature measured between various pairs of contacts of v12RuO₂/PdAg3505 resistor. (b) Data from (a), averaged over temperature range 77–300 K, versus number *n* of squares between respective side contacts. (c) Voltage *V* (film thickness *d*) measured on (between) successive side contacts.

$$s \equiv \frac{1}{223 \text{ K}} \int_{77 \text{ K}}^{300 \text{ K}} \langle \delta R^2 \rangle dT = \frac{1}{223 \text{ K}} \int_{77 \text{ K}}^{300 \text{ K}} \int_{f_l}^{f_u} S_R(f, T) df dT, \quad (1)$$

$S_R(f)$ is the power spectral density of resistance fluctuations, which could be recognized as a measure of average density of noise sources. Characteristic (corner) frequency of TANS is related to the temperature via $f_{TANS} \sim \exp(E/kT)$ [6], E is the activation energy. The limits of integrations in Eq. (1) fix then the range of energies of the fluctuators that build up noise spectrum. This range should be kept as wide as possible in order to smooth position dependent variations of fluctuators density. In practice it is limited due to the operating range of the apparatus (T_{min} , T_{max}) and noise measurement conditions. In our experiment noise spectra were gathered continuously as the temperature rose slowly. This introduces some minimum frequency (of ~few Hz), only above which the spectra are not influenced by f^{-2} component resulting from the drift [7]. On the opposite side of the frequency axis the spectra of resistance noise, which are generally of $1/f$ type, are influenced by the thermal noise. This introduces some maximum frequency for reasonable estimation of $S_R(f)$. As parameter s is to be used for comparative purposes the frequency limits in Eq. (1) should be identical for all measured samples. Some of them (low-resistive) are low-noise devices for which extending f_u above ~100 Hz is difficult. Therefore, we have fixed the f -limits of the integral in Eq. (1) to the band of 10–100 Hz.

Parameter s , when plotted as a function of length of the film extending between the contacts the signal is acquired from, can give bulk and “resistive-to-conductive-films-interface” components of the resistor’s noise. For the data of Fig. 1a s is plotted versus the number n of the squares between respective pairs of resistor side contacts. Line drawn through the origin and the first two points shows linear increase of the noise as expected for uncorrelated noise sources. The slope of this line gives the magnitude of bulk noise per square, s_{\square} . Taking into account the actual dimensions and sheet resistance R_{\square} of the resistor it can be used to calculate dimension-independent noise intensity: $C_{bulk} \equiv s_{\square} / R_{\square}^2 \times \text{square volume}$ (=1 mm² × thickness) capable for the comparison of noise intensities of various materials [8,9]. Noise of the films interfaces (Fig. 1b) can be estimated by subtracting bulk noise, calculated for the whole resistor length, $s_{bulk} = 15s_{\square}$ (see Fig. 1b), from total noise of the resistor measured between terminations 1–7, s_{1-7} . It stems from simple geometric considerations that interface noise scale with film width w as $s_{interface} \sim w^{-3}$, whereas bulk noise per square as $s_{\square} \sim w^{-2}$. Thus, the ratio $C_n \equiv ws_{interface} / s_{\square}$ is the width independent parameter characterizing interface noise with respect to the bulk noise. Its meaning is that it is the length of the resistive film which has the noise equal the interface noise. Values of this parameter together with values of bulk noise intensity and other quantities necessary to make

Table 1
Summary of the data for v12 RuO₂ series

Series	v12/ AuP303	v12/ PtAuP304	v12/ PdAg3505	v12/ PtAg1130C	v12/ Au3612
<i>d</i> (μm)	15.6	15.5	13.1	12.4	13.2
ρ_{tot} (Ω cm)	3.26	3.05	4.70	5.13	3.34
ρ_{bulk} (Ω cm)	3.03	2.81	3.45	3.34	3.12
C_n (mm)	3.5	4.8	26	80	0.25
NR (%)	4.3 (PS)	8	6.3	3	2.6 (PS)
$C_{bulk} \times 10^{24}$ (m ³)	17.8	16.2	14.1	12.8	17.7

the calculations for various resistors are gathered in Table 1. Film thickness was directly measured (Vistronik C1) in-between side contacts. Bulk and total resistivities were estimated from (measured) distribution of the voltage along the sample length, as shown in Fig. 1c. Additional information stored in Table 1 is the magnitude of noise non-stationarity (non-repeatability) characterized by dimensionless parameter NR. Values of noise measured between terminations 1–7 in several independent experiments are scattered (Fig. 1b) due to the switching phenomenon mentioned in Section 1. For the resistor of Fig. 1, of the three measurements two give nearly the same noise versus temperature traces and one with a bit larger noise in the range $T > 200$ K. After integration the traces give values of s_{1-7} that differ by some amount (points labeled 1–7 in Fig. 1b). Maximum deviation relative to average value of the total noise defines parameter $NR \equiv \max \Delta s_{1-7} / \langle s_{1-7} \rangle$, which gives quantitative estimation of non-stationary effects of the interface noise. Acronym PS placed near the value of parameter NR supplies additional information referring to the specific type of non-stationarity as discussed in Section 1. When placed near the value of C_{bulk} it means that PS was observed in the inner part of the resistor, that is in the resistive film.

For high-resistive samples $1/f$ noise is also higher so that it is possible to move the band of integration in Eq. (1) to higher frequencies. Then different region of fluctuators density is probed so we expect different values of noise parameters. For the exemplary case of v12RuO₂/PdAg3505 resistor calculations were done for the bands 10–100 Hz (Fig. 1) and 100–1000 Hz. We have found $C_{bulk} = 18.8 \times 10^{-24}$ m³, $C_n = 24$ mm and $NR = 5.8\%$ for the band 100–1000 Hz and $C_{bulk} = 14.1 \times 10^{-24}$ m³, $C_n = 26$ mm and $NR = 6.3\%$ for the band 10–100 Hz. As expected, numbers are different but both sets of data lead to similar conclusions concerning spatial distribution of the fluctuators. Their density increases in the interface and the number of excess fluctuators equals the number of fluctuators in a pure (not modified) film of ~2.5 cm length.

3. Samples

Samples were made from laboratory-prepared as well as commercial resistive pastes. The lab-made pastes consist of RuO₂ (10% and 12% RuO₂ by vol.) and lead borosilicate glass (10% B₂O₃, 15% SiO₂, 65% PbO). Commercial pastes (Du Pont and ITME – Institute of Electronic Materials Technology, Warsaw) have sheet resistivities in the range 100 Ω/□–10 kΩ/□. Conductive material in these pastes is either RuO₂ (Du Pont) or bismuth ruthenate (ITME). Contacts were made with pastes from Metech, Du Pont, Electro-Science Laboratories (ESL) and ITME which contain Au, Pt, Pd and Ag as basic ingredients. Samples for measurements were manufactured (i) in conventional high-temperature process on alumina substrates; they were screen printed through 200 mesh screen and after firing at peak temperature, T_p = 800 °C or 850 °C or 900 °C, for 10 min gained the average thickness of 10–20 μm, (ii) as LTCC resistors on Du Pont green tape DP 951 as surface devices; contacts and resistors were fired together (co-fired) or separately (post-fired).

4. Results

For all samples the resistance origin of the measured noise was verified. Voltage mean square fluctuations $\langle \delta V^2 \rangle$ were measured for different dc bias *V* and occur to scale linearly with *V*². During thermal cycling experiments the samples were always kept in this linear region. Biasing voltages were never greater than 15 V and currents were usually lower than 0.3 mA. This value corresponds to current density of 0.02 mA/cm² which was low enough to prevent self-heating of the samples at the lowest temperatures. The only exception were low-resistive LTCC resistors for which the biasing current of ~0.5 mA was necessary to rise resistance noise above thermal background. Then self-heating was considerable and cooling these resistors down to 77 K occurred (was) impossible.

In Table 1 results are gathered for the resistors made of lab-made resistive paste composed of 12% RuO₂ by volume (v12) and several conductive pastes from Metech (3505, 1130C, 3612), and ITME (P304, P303). In all cases both bulk parameters: resistivity of ρ_{bulk} ≈ 3.10 ± 0.27 Ω cm and noise intensity of C_{bulk} ≈ 15.8 ± 2.0 × 10⁻²⁴ m³ are scattered within the limits which can be attributed to nonuniform film thickness. Detailed calculations performed for v12RuO₂/AuP303 resistor [10] show that interface noise of ~17 × 10⁻⁶ Ω² found for both ITME pastes (columns 2, 3) can also be attributed to this reasoning, namely the thinning of resistive layer in the vicinity of the terminations. RuO₂–Ag interaction observed for two Metech's pastes (columns 4, 5) leads to well known abnormal dimensional (size) effect [1,2], that is the increase of resistivity near the terminations, and (less known) strong increase of the interface noise [11,12]. Interesting question is whether the latter results only from increasing resistivity of the film or also from enhanced intensity of locally fluctuating quantities. For the case when the magnitude of random processes remains unchanged against Ag doping, the resistance noise can be estimated as

$$S_R \sim \int \langle (\delta r(x))^2 \rangle dx \sim \int [\rho(x)]^2 dx \sim \int \left(\frac{\partial V(x)}{\partial x} \right)^2 dx. \quad (2)$$

Simple calculations made for v12RuO₂/PdAg3505 resistor show that indeed, considerable part of the interface noise may result only from increasing resistivity. How large this part can be depends on the precise form of voltage distribution near the terminations, which is unknown. For the trial *V*(*x*) functions, shown in Fig. 1c, results of calculations are shown as open circles in Fig. 1b. It occurs that at least 40% of the interface noise can be attributed to the

increasing resistivity, provided all the assumptions behind Eq. (2) are fulfilled. Apart from those mentioned above, Eq. (2) assumes that some random process *y*(*t*) couples to resistivity via the mechanism for which one may write δ*r* ~ ρδ*y*. Modulation of the tunneling rates or activation energies are good examples of such process.

To complete the comparison, note that ITME conductive pastes when interacting with modifiers-free resistive composition (our v12 pastes) produce large non-stationary noise. Metech products are much better for this issue. Of these, the gold-based composition 3612 seems to be the best as for it neither dimensional nor non-stationary effects have been observed. Also interface noise is the lowest of the five and, in fact, negligible. These observations remains unchanged when parameters of technological process change. In Table 2 data for selected series fired at different temperatures are summarized. For all these series, bulk parameters, i.e. resistivity and noise intensity, increase when firing temperature increases. Also the size effect, which can be characterized by the ratio ρ_{tot}/ρ_{bulk}, observed for PtAg series increases with T_p increasing. There is no systematic effect of firing temperature on interface noise. It remains huge but stationary for Ag-based terminations and almost negligible for Metech Au-based paste. It seems that ITME P304 paste gives less noisy contacts when fired in lower temperature. Both bulk and interface characteristics change significantly upon the change of ingredients of resistive material. Commercial pastes R344 and R343 contain bismuth ruthenate (Bi₂Ru₂O₇), instead of ruthenium dioxide [13]. Data in Table 3 show that R344 paste form well behaved low-interface-noise, no-size-effect system with all Metech's pastes. Much worse is ITME P304 paste for which both small size effect and non-stationary noise were observed. Same conclusions are valid also for R343 paste of

Table 2
Selected v12 RuO₂ series versus firing temperature

Series	v12/PtAuP304			v12/PtAg1130C			v12/Au3612		
	T _p (°C)								
	800	850	900	800	850	900	800	850	900
ρ _{tot} (Ω cm)	2.67	3.05	7.67	3.36	5.13	11.9	2.82	3.34	5.92
ρ _{bulk} (Ω cm)	2.59	2.81	7.1	2.68	3.34	6.55	2.74	3.12	5.51
C _n (mm)	0.65	4.8	4.5	38	80	56	0.18	0.25	0
NR (%)	1	8	1.25	1.6	3	0.7	2.3	2.6	5
C _{bulk} × 10 ²⁴ (m ³)	13.4	16.2	20	11	12.8	22.4	13.1	17.7	26
						(PS)			(PS)

Table 3
Summary of the data for R344 series

Series	R344/ Au3612	R344/ PdAg3505	R344/ PtAg1130C	R344/ PtAuP304
<i>d</i> (μm)	12.4	12	12.1	12.1
ρ _{tot} (Ω cm)	11	10.6	10.5	12.3
ρ _{bulk} (Ω cm)	11	10.6	10.1	11.9
C _n (mm)	0.65	1.3	0.85	1.7
NR (%)	1.6 (PS)	2.8 (PS)	2.8 (PS)	6.2 (PS)
C _{bulk} × 10 ²⁴ (m ³)	236	209	232 (PS)	277

Table 4
Summary of the data for R343 series

Series	R343/ Au3612	R343/ PdAg3505	R343/ PtAuP304	R343/ PtAg1130C
<i>d</i> (μm)	13.6	14.4	13.3	14.7
ρ _{tot} (Ω cm)	1.32	1.33	1.47	1.33
ρ _{bulk} (Ω cm)	1.32	1.33	1.47	1.33
C _n (mm)	0.45	0.27	0.76	0.7
NR (%)	0.5 (PS)	1.4	0.5	4.4 (PS)
C _{bulk} × 10 ²⁴ (m ³)	26	25	26	22.5

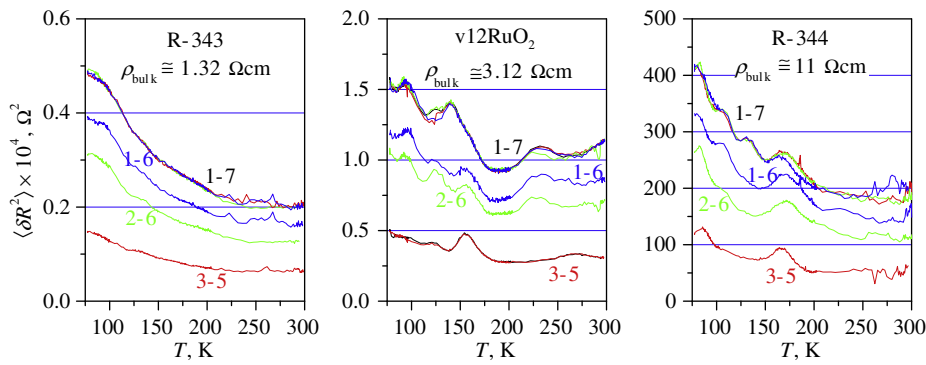


Fig. 2. Mean square resistance fluctuations in the band 10–100 Hz versus temperature measured between different pairs of terminations for samples made of pastes with different resistivity.

Table 5
Summary of the data for LTCC series

Series	DP 2021			DP2041		
	Au (PF)	PdAg (PF)	PdAg (CF)	Au (PF)	PdAg (PF)	PdAg (CF)
d (μm)	9.3	9	8.1	8.6	9.7	9.2
ρ_{tot} ($\Omega\text{ cm}$)	0.112	0.137	0.118	9.89	13.24	9.04
ρ_{bulk} ($\Omega\text{ cm}$)	0.112	0.135	0.118	9.67	11.71	8.89
C_n (mm)	1.25	0.89	0	1.3	0.14	0.44
NR (%)	48	31	6.9	36	5.5	3
$C_{\text{bulk}} \times 10^{24}$ (m^3)	0.035	0.033	0.030	1.5	3.6	1.5

sheet resistance $1\text{ k}\Omega/\square$. Data for R343 resistors are summarized in Table 4. Interesting observation is that bulk noise of RuO_2 -based composition is lower than this of $\text{Bi}_2\text{Ru}_2\text{O}_7$ -based. On the other hand the latter seems to be more susceptible to form well behaved low-noise interface with wider spectrum of conductive materials used for resistor terminations.

Fig. 2 shows the feature that cannot be seen from tabular data. Intensity and number of TANS increases with increasing resistivity. In noise versus temperature traces they appear as local maxima. They are almost absent in $\sim 1.3\text{ }\Omega\text{ cm}$ films of R343 series but became strong and frequent in $\sim 11\text{ }\Omega\text{ cm}$ R344 films.

Special attention should be paid to LTCC resistors. They were made from DP2021 ($100\text{ }\Omega/\square$) or DP204 ($10\text{ k}\Omega/\square$) pastes. Contacts were prepared from gold-based paste from ESL (product 8880H) or PdAg composition from Du Pont (product 6146 compatible with 951 Green Tape). Data for co-fired (CF) and post-fired (PF) LTCC resistors are collected in Table 5. First observation is that measured values of bulk noise are compatible with those reported in the literature [8] and are considerably lower than respective values for “on-alumina” resistors (see Fig. 3). Concerning layers interaction, note that Au contacts are worse than PdAg ones: both low- and high-resistive, Au-terminated samples have large non-stationary contact noise. PdAg composition designed for LTCC technology is much better for this issue, especially when fabricated in co-fire process. Co-firing, in general, seems to be critical parameter for switching phenomenon: in Table 5 all data for the CF resistors exhibit only little non-stationarity. Co-fired resistors are much better attached to the substrate than those made in PF process, and only little internal stress relaxes in CF resistors during temperature cycles. In line with this interpretation are also lower values of parameter NR observed for high-resistive DP2041 samples. These samples contain small fraction of conducting component and only little stress arise due to mismatch of temperature expansion coefficient of metallic grains and insulating matrix of resistive film.

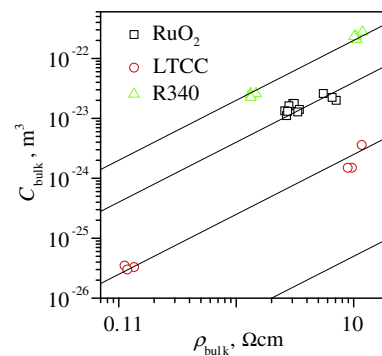


Fig. 3. Noise intensity versus resistivity of various thick resistive films. The most bottom line show relation found for gold, poly-Si and poly-SiGe layers found in Ref. [9].

5. Conclusions

The use of low-frequency noise spectroscopy reveals interesting properties of conductive/resistive film interface of TFR. These properties can be used for evaluation of the quality and practical performance of various systems of resistive/conductive pastes used in fabrication technology of these passives. At least three parameters should be taken into account when making the rank of compatible systems. These are size effect characterized by the ratio $\rho_{\text{tot}}/\rho_{\text{bulk}}$, and two noise parameters, C_n and NR. Of them, parameter NR seems to be more important for reliable applications as it is connected with rapid microstructural changes that may lead to device failure. For this reason any criterion should not evaluate systems that have values of NR larger than few percent. The other two parameters describe the impact of resistors interfaces on devices utilities. They are not critical for reliable purposes but large value of $\rho_{\text{tot}}/\rho_{\text{bulk}}$ and C_n mean that resistance and noise of the resistor are far from their nominal (designed) values. Circuits containing such components have lower performance characteristics or even are malfunctioning. Moreover, the well-known conjecture between noise level and device stability, lifetime and reliability [3,14–17] predicts that large C_n values are connected to accelerated drifts and enhanced probability of failure. Therefore valuable (compatible) systems of resistive/conductive pastes should keep both $\rho_{\text{tot}}/\rho_{\text{bulk}}$ and C_n as low as possible. When all these factors are taken into account, the compatible systems of pastes can be evaluated basing on the experiments performed in this study:

- RuO_2 resistive pastes are compatible with Au-based terminations. Metech product 3612 forms low-size-effect, low-noise, fairly stable contacts. Ag-containing conductive pastes should

not be used with RuO₂ pastes. Firing conditions have little influence on interface characteristics.

- Bismuth ruthenate (Bi₂Ru₂O₇) pastes form well matched contacts with Au conductive pastes. PdAg compositions are acceptable, although they form less noise-stable contacts, especially for high-resistive devices. PtAu contacts are unacceptable, probably due to glass composition incompatible with the glass used in the resistive paste.
- LTCC systems of compatible pastes and green tapes (e.g. DP2041/6146/951) are optimized for co-firing process and work better for low-conductive resistors containing lower fraction of metallic component.

Note eventually, that the data gathered in Tables 1–5 can be used not only to evaluate quality of thick-film resistor interfaces, but also to evaluate quality indicator of the resistive film itself. It was proposed to consider the so-called *reduced noise mobility*, μ_{zH} as such an indicator [16,17]. From our bulk parameters mobility μ_{zH} can be calculated as $\mu_{zH} = C_{\text{bulk}}/\rho_{\text{bulk}}q$, q is the elementary charge. Also there is simple relation with parameter C_{us} defined for layered materials, $C_{\text{us}} = C_{\text{bulk}}/d$ [9]. This parameter is thickness dependent but useful when studying noise intensity as a function of sheet resistance R_{\square} of the films. For gold, poly-Si and poly-SiGe layers Vandamme and Casier [9] have found linear relation $C_{\text{us}} = KR_{\square}$ with the value of $K = 5 \times 10^{-13} \mu\text{m}^2/\Omega$. As shown in Fig. 3 our data also show linear dependence $C_{\text{bulk}} = K'\rho_{\text{bulk}}$ with $K' = 2.5 \times 10^{-11} \mu\text{m}^2/\Omega$ for resistive films of LTCC devices, $4 \times 10^{-10} \mu\text{m}^2/\Omega$ for RuO₂ films and $2 \times 10^{-9} \mu\text{m}^2/\Omega$ for Bi₂Ru₂O₇ films of R340 series. As $R_{\square} = \rho_{\text{bulk}}/d$ coefficients K and K' can be directly compared. Our resistive films are thus from 2 to 4 decades noisier than layers studied in Ref. [9] (see Fig. 3).

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