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The Characterization of Thick-Film Resistors on Dielectric-on-Steel Substrates for Strain Gauge Applications

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The compatibility of several types of $10 \, k\Omega/sq$, thick-film resistors (Du Pont QM-84, QM-94 and 8039, ESL 3414-A, and Heraeus 8241), which were developed for firing on alumina substrates, with insulated metal substrates, as well as the compatibility of some thick-film multilayer dielectrics (Du Pont QM-42, ESL D-4914 and Heraeus IP-222-SL) with stainless-steel substrates were evaluated. Sheet resistivities, temperature coefficients of resistivity (TCR), noise indices and gauge factors of the resistors fired on alumina substrates and on dielectric-on-steel substrates, were measured. The TCR values of most resistors, fired on the dielectric-on-steel substrates, are higher than the TCRs of resistors on alumina substrates, which is attributed to the higher thermal expansion coefficient of the stainless steel. Some "combinations" have very high and irreproducible gauge factor values, which is attributed to the incompatibility of the resistor and dielectric materials. At the interface between the dielectrics and the steel substrates, no interactions or evidence for new phases could be detected, which indicates the compatibility of the evaluated dielectric materials and the stainless steel.

1. Introduction

The change in resistance of a resistor under an applied stress is partly due to deformation, i.e., changes in the dimensions of the resistor, and partly due to alterations in the

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specific resistivity as a result of microstructural changes.⁽¹⁾ The gauge factor (GF) of a resistor is defined as the ratio of the relative change in resistance ($\Delta R/R$) and the strain (ΔUl):

$$GF = (\Delta R/R) / (\Delta U/l) \tag{1}$$

Geometrical factors alone result in gauge factors of 2–2.5. Higher gauge-factor values are due to microstructural changes which alter the specific conductivity. The GF values of thick-film resistors are mostly between 3 and 15. Due to their stability, low values of the temperature coefficient of resistivity (TCR) below 100×10^{-6} /K, and relatively low cost, strain gauges realised with thick-film technology offer advantages in some applications over both metal films (low GF, low TRC, expensive) and semiconducting elements (high GF, high TCR, inexpensive). Within the same resistor series, both the GFs and the current-noise indices of thick-film resistors increase with increasing sheet resistivity. Therefore, in most cases, $10 \text{ k}\Omega/\text{sq}$. resistors are used for the strain sensors as a useful compromise between sensitivity and relatively low noise, and also because of their relatively low power consumption.

Note, however, (though this is admittedly not directly relevant to the subject of this paper) that while the high GFs of thick-film resistors are desirable for sensors, materials with low GFs are obviously preferable for resistors in hybrid thick-film circuits. Resinencapsulating or fixing (e.g., gluing, soldering, etc.) thick-film hybrid circuits, made mainly on alumina substrates, to other supports and the possible mismatching of temperature expansion coefficients can result in a detrimental resistivity drift after the encapsulation or during use.

Pressure or force sensors can be realised with resistors on deformable membranes. The thick-film resistors are printed and fired on a ceramic substrate (diaphragm), which is usually based on alumina. However, sensing elements made on stainless-steel substrates would exhibit improved characteristics as the steel has a higher mechanical (flexural) strength, an intrinsic robustness, and a lower modulus of elasticity (around 70%) than alumina. (7-9) Furthermore, stainless steel is easily machinable and irregularly shaped substrates, large or small, can be made. Stainless steel generally contains more than 10% of Cr. The addition of Si and AI (and other elements in minute quantities) can also be incorporated. The inclusion of chromium in the steel leads to the oxidation of chromium to form a dense chromium-oxide "skin" over the steel surface and prevents further oxidation during high-temperature treatment. However, the conductive metallic substrate needs to be covered with an insulating dielectric layer to allow printing and firing of the thick-film materials on its surface. The dielectric is deposited as a paste using screen printing (DOS: dielectric on steel) or by laminating a LTCC (low-temperature cofired ceramic) tape on the surface of the steel substrate (TOS: tape on steel). (10)

Some mechanical characteristics for 96% Al_2O_3 , which is the most commonly used material for the substrates of thick-film circuits, and for steel (ferritic steel 1.4016 with 17% of Cr), are summarized in Table. 1.

However, as most thick-film resistors are developed for firing on alumina substrates, their compatibility and possible interactions with dielectric-covered stainless steel and the changes in their electrical characteristics need to be evaluated (see, for example, refs. (7,11) and references cited therein).

Table 1 Some characteristics of 96% Al₂O₃ and stainless steel (1.4016).

Al ₂ O ₃ (96%)	Steel 1.4016	
250–330	220	
3.6	7.7	
$7 \times 10^{-6} / \text{K}$	10×10 ⁻⁶ /K	
20–24	25	
	250–330 3.6 7×10 ⁻⁶ /K	250–330 220 3.6 7.7 7×10 ⁻⁶ /K 10×10 ⁻⁶ /K

A number of 10 kW/sq. thick-film resistors were evaluated on "bare" ceramic alumina substrates, on alumina substrates covered with dielectric and on stainless-steel substrates covered with dielectric. The thick-film materials, i.e., the resistor and dielectric pastes, are listed in Table 2. The 3414-A resistors, Electro Science Labs., were developed particularly for use in strain gauges. The high gauge factors of this material are attributed to it's microstructure. In most thick-film resistors the grains are of sub-micrometer size. For 3414-A typical grain sizes are 1 micrometer or more. Tamborin *et al.* showed that, for the same sheet resistivity, the gauge factors increase linearly with the logarithm of conductive phase grain size.

Two Du Pont resistor series, QM 80 and QM 90, are intended for making resistors on the top of the Du Pont QM-42 dielectric layer. Resistors from the QM 80 series are designed for Pd/Ag termination, while those from the QM 90 series are terminated with silver. All other resistors were terminated with Pd/Ag-based conductors.

Some data on the conductive phase and the semi-quantitative results of the energy-dispersive X-ray microanalysis (EDS) of the glass composition of the thick-film resistors are summarized in Table 3.⁽⁶⁾ All glasses contain lead, silicon and aluminium oxides. Boron oxide, which is also present in the glass phase, cannot be detected in the EDS spectra because of the low relative boron weight fraction in the glass and the strong absorption of the boron $K\alpha$ line during EDS analysis of the glass matrix. However, most thick-film resistors use glasses with roughly equal proportions of PbO, SiO₂ and B₂O₃, since glasses rich in PbO, SiO₂ or B₂O₃ have high temperature expansion coefficients, high melting temperature or glass immiscibility, respectively.⁽¹⁴⁾

2. Experimental

Stainless-steel substrates (ferritic steel 1.4016) were prepared by printing (twice) the dielectrics on both sides of substrates and firing them at 850° C. Thick-film $10 \text{ k}\Omega/\text{sq}$ resistors (see Table 2) were printed and fired at 850° C for 10 min on 96% alumina substrates and on steel substrates covered with different dielectrics. Resistors were also printed and fired on alumina substrates, which were covered with dielectrics from the same producers, i.e., Du Pont 8039, QM-84 and QM-94 resistors on Du Pont QM-42 dielectric, ESL 3414-A resistors on ESL D-4913 dielectric and Heraeus 8241 resistors on Heraeus IP-222-SL dielectric. The dimensions of the resistors were $1 \times 1 \text{ mm}^2$. The resistors were terminated with a prefired Pd/Ag conductor with the exception of QM-94, which was terminated with a silver conductor.

Table 2	
Thick-film	materials.

Producer	Resistors	Dielectric
Du Pont	QM-84, QM-94, 8039	QM-42
ESL	3414-A	D-4914
Heraeus	8241	IP-222-SL

Table 3 Conductive phase and summarized semi-quantitative results of EDS microanalysis of elements detected in the glass phase of $10 \text{ k}\Omega/\text{sq}$, resistors. (6)

Resistor Conductive phase Main elements Other e		Other elements detected		
8039	ruthenate	Si, Pb, Al	Zr	
QM-84	RuO ₂ + ruthenate	Si, Pb, Al	Cu, Z r	
QM-94	RuO_2 + ruthenate	Si, Pb, Al	Ca, Mn, Cu	57
3414-A	ruthenate	Si, Pb	Al, K	
8241	RuO_2	Si, Pb, Al	Ca	χ.

Cold (from -25°C to 25°C) and hot (from 25°C to 125°C) TCRs were calculated from resistivity measurements at -25°C, 25°C, and 125°C. The current noise was measured in dB on 100 mW loaded resistors using the Quan Tech method (Quan Tech Model 315–C).

The changes in resistivity as a function of substrate deformation were measured with the simple device described in ref.(15) The device is shown in Fig. 1. The substrate with a printed-and-fired thick-film resistor in the middle was supported on both sides. The load was applied to the middle of the substrate with a micrometer, which induced a tensile strain in the resistor. The magnitude of the strain is given by eqs. (2) and (3), $^{(16)}$ where "d" is the deflection (m), "a" is the substrate thickness (0.625 for alumina substrates and 1.05 mm for dielectric-on-steel substrates) (m), "X" is the distance from the supported edge of the substrate (m) and "L" is the distance between the supported edges (m).

$$\varepsilon = \Delta l/l = (d \cdot \mathbf{a} \cdot X \cdot 12)/L^3 \tag{2}$$

For X=L/2, i.e., in the middle of the substrate, where the strain is greatest, the equation transforms into:

$$\varepsilon = \Delta U = (d \cdot a \cdot 6)/L^2. \tag{3}$$

The gauge factors are calculated using eqs. (1) and (3) from the resistivity changes and the strain.

For the microstructural investigation the samples were mounted in epoxy in a cross-sectional orientation and then cut and polished using standard metallographic techniques.

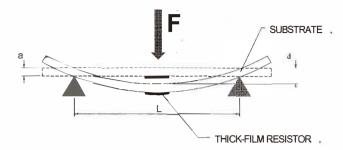


Fig. 1. A method for measuring the changes of resistivity as a function of substrate deformation (schematically). (15) The ceramic substrate is supported on both sides. The load is applied in the middle of the substrate with a micrometer and induces a tensile strain in the resistor.

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.

3. Results and Discussion

A semiquantitative EDS analysis of the dielectrics is shown in Table 4. The oxides are given in weight percent. All three materials contain around 20 wt.% of Al₂O₃ and between 30 and 40 wt.% of SiO₂. The D-4914 dielectric also contains over 35 wt.% of CaO. A small amount of cobalt, presumably added for blue colouring, is detected in all three dielectrics. The analysis of the stainless steel shows (as expected) 17 at.% of chromium and 1 at.% of silicon.

Microstructures of cross sections of the dielectrics/stainless steel interfaces are shown in Figs. 2a (QM-42), 2b (D-4914) and 2c (IP 222 SL). The steel substrate is on the right. The black grains imbedded in the dielectric matrix in the QM-42 and IP 222 SL dielectrics are alumina particles, which were added as the ceramic filler. The light grains in the microstructure of IP 222 SL are rich in Si-, Co- and Zn-oxides. At the interface between the dielectrics and the steel substrates, no interactions or evidence for new phases could be detected. These results confirm the compatibility of the evaluated dielectric materials and the stainless steel.

Sheet resistivities, cold (-25° C to 25° C) and hot (25° C to 125° C) TCRs, noise indices and GFs of the resistors fired on the alumina substrates, on the alumina substrates covered with dielectric and on the dielectric-on-steel substrates, are presented in Table 5. The experimental GF results are rounded up to 0.5, e.g., 9.0 or 9.5. The noise indices and GFs are also shown graphically in Figs. 3 and 4, respectively. On the graphs, the ceramic is denoted by "Cer." and the steel is denoted by "S." Note, however, that in Fig. 3, the noise indices are expressed in " μ V/V" while in Table 5, they are given as "dB". These two units are related with an equation:

noise (dB) =
$$20 \times \log \text{ noise } (\mu \text{V/V})$$
. (4)

Table 4 EDS semiquantitative analysis of Du Pont QM-42, ESL D.4914 and Heraeus IP-222-SL dielectrics (wt.%).

Oxide		Du Pont QM-42	ESL D-4914	Her. IP-222-SL	
MgO		1	1	2,7	
Al_2O_3		17.4	22.6	22.4	
SiO_2		32.4	39.6	42.0	
K_2O		/	/	1.2	
CaO =		2.2	36.9	4.1	
TiO_2		2.4	/	3.5	
Co_2O_3	67	1.3	0.9	2.4	
ZnO		16.1	1	5.4	.5
ZrO_2		8.3	1	/	
BaO		19.9	1	17.5	

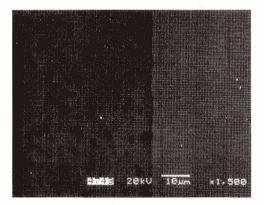


Fig. 2 (a) Microstructure of Du Pont QM-42 dielectric fired on a steel substrate. Dark grains are alumina particles. The steel substrate is on the right.

The sheet resistivities of the 8241 resistors that were fired on the dielectric-on-steel substrates were very low, below 1 k Ω /sq. Sheet resistivities of the ESL resistors 3414-A on the same substrates were, on the other hand, rather high, between 15 and 30 k Ω /sq. For other resistor series, the sheet resistivities of resistors on alumina and on dielectric-on-steel-substrates are comparable. The noise indices of the 3414-A resistors, regardless of the substrate, were the highest. On the other hand, the GFs of the 3414-A resistors on alumina had the highest values of all the tested resistor materials. The TCR values of resistors, fired on the dielectric-on-steel substrates, are higher than TCRs of resistors on alumina substrates. The highest values, over 350 ×10⁻⁶/K, were measured for 8241 on D-4914 covered steel. This is presumably due to the higher thermal expansion coefficient of the stainless steel compared with alumina (see Table 1). As an example, the dependence of

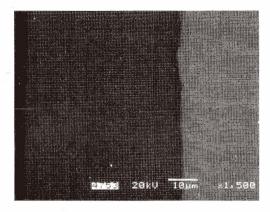


Fig. 2 (b) Microstructure of ESL D-4914 dielectric fired on a steel substrate. The steel substrate is on the right.

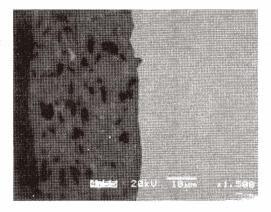


Fig. 2 (c) Microstructure of IP 222 SL dielectric fired on a steel substrate. Dark grains are alumina particles. The steel substrate is on the right.

the relative resistivity versus temperature for 8039 resistors, fired on different substrates, is presented in Fig. 5.

The irreproducible GF measurements in the case of 3414-A, fired on D-4914 covered alumina and of 8241, fired on QM-42 covered steel, as mentioned in the note for Table 5, were unrealistically high values for GFs. Measured GFs were between 250 and 350, which is more than one order of magnitude higher than the GFs of typical thick-film resistors, which are between 2 and 20. These results are, at this time, tentatively attributed to the incompatibility between the materials used in these "combinations," i.e., the 8241 resistor on QM-42 multilayer dielectric and the 3414-A resistor on D-4914 covered alumina substrate, and the resulting microcracks in the resistor film, as described by Prudenziati *et*

Table 5 Sheet resistivities, cold (-25°C to 25°C) and hot (25°C to 125°C) TCRs, noise indices and GFs of the resistors fired on the alumina ceramics and the resistors fired on the dielectric-on-steel substrates.

Resistor	Substrate	$R \\ (k\Omega/sq.)$	Cold TCR (×10-6/K)	Hot TCR (×10-6/K)	Noise (dB)	GF
QM-84	Al_2O_3	4.4	-60	15	-12.5	7.5 ± 0.3
	Al2O3 / QM-42	6.8	-105	-10	-13.7	8.0 ± 0.3
	Steel / QM-42	5.7	25	105	-14.2	7.0 ± 0.3
	Steel / D-4914	4.6	90	155	-13.7	7.5 ± 0.3
	Steel / IP-222-SL	3.3	100	165	-15.3	7.0 ± 0.3
QM-94	Al_2O_3	3.3	-20	60	-13.7	7.5 ± 0.3
	Al_2O_3 / QM-42	3.3	20	75	-17.3	10.0 ± 0.3
	Steel / QM-42	4.8	45	110	-12.5	8.0 ± 0.3
	Steel / D-4914	3.2	90	155	-15.1	7.5 ± 0.3
	Steel / IP-222-SL	3.2	85	145	-13.1	6.0 ± 0.3
8039	Al_2O_3	9.3	20	70	-10.2	9.0 ± 0.3
	Al2O3 / QM-42	14.5	-85	-10	-11.2	9.5 ± 0.3
	Steel / QM-42	9.8	95	165	-12.4	9.5 ± 0.3
	Steel / D-4914	7.2	145	195	-11.9	7.0 ± 0.3
	Steel / IP-222-SL	5.8	150	205	-12.3	7.5 ± 0.3
3414-A	Al_2O_3	6.7	-185	-95	9.0	20.0 ± 0.3
	Al ₂ O ₃ / D-4914	57	-11900	-2250	>30*	?**
	Steel / QM-42	26.2	-165	-85	3.0	12.0 ± 0.3
	Steel / D-4914	14.5	-135	-9 0	2.1	12.0 ± 0.3
	Steel / IP-222-SL	30.2	-210	-125	3.5	13.0 ± 0.3
8241	Al_2O_3	5.8	-20	-40	0.2	14.0 ± 0.3
	Al_2O_3 /IP-222-SL	2.0	40	75	-6.5	9.5 ± 0.3
	Steel / QM-42	0.5	275	305	-10.6	?**
	Steel / D-4914	0.6	335	380	-8.7	7.5 ± 0.3
	Steel / IP-222-SL	0.8	235	265	-9.1	6.0 ± 0.3

>30 dB* noise too high to measure

al.⁽¹⁷⁾ However, it is interesting to note that the sheet resistivity and the noise index of 8241 on QM-42/steel are rather low, around 500 ohm/sq. and -10.5 dB, respectively, while the resistivity of 3414-A on D-4914/alumina is over 50 k Ω /sq. TCR values are large and negative and the noise index is very high and could not be measured as the upper range of the Quan Tech Model 315-C instrument is +30 dB. Therefore, it is not shown in Fig. 4.

4. Conclusions

The compatibility of various $10 \text{ k}\Omega/\text{sq}$, thick-film resistors (Du Pont QM-84, QM-94 and 8039, ESL 3414-A, and Heraeus 8241), developed for firing on alumina substrates, with insulated metal substrates as well as the compatibility of some thick-film dielectrics (Du Pont QM-42, ESL D-4914 and Heraeus IP-222-SL) with stainless-steel substrates

^{?**} irreproducible measurements

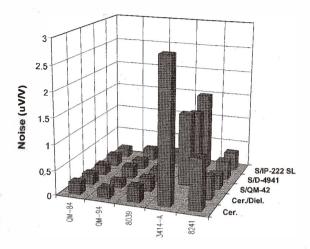


Fig. 3. Noise indices of $10~k\Omega$ /sq. thick-film resistors, fired on the alumina substrates, on alumina substrates covered with dielectric and on dielectric-on-steel substrates. The ceramic is denoted by "Cer." and the steel is denoted by "S."

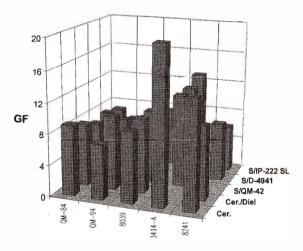


Fig. 4. Gauge factors of $10 \, k\Omega$ /sq. thick-film resistors, fired on the alumina substrates, on alumina substrates covered with dielectric and on dielectric-on-steel substrates. The ceramic is denoted by "Cer." and the steel is denoted by "S."

(ferritic steel 1.4016) were evaluated. At the interface between the dielectrics and the steel substrates, no interactions or evidence for new phases could be detected. This indicates the compatibility of the evaluated dielectric materials and the stainless steel, which was also confirmed by XRD analysis.

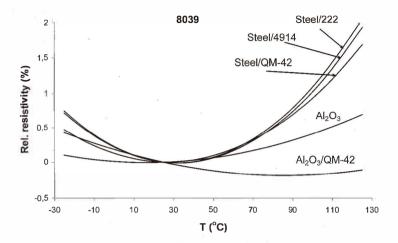


Fig. 5. Dependence of relative resistivity on temperature for 8039 thick-film resistors, fired on the alumina substrates, on alumina substrates covered with QM-42 dielectric, and on dielectric-on-steel substrates.

Sheet resistivities, TCRs, noise indices and gauge factors of resistors, fired on the alumina substrates, on alumina substrates covered with dielectric, and on dielectric-onsteel substrates, were measured. Sheet resistivities of QM-84, QM.94 and 8039 resistors on different substrates are comparable. Resistivities of 3414-A and 8241 resistors on dielectric-on-steel substrates, as compared with the values on alumina, are relatively high and low, respectively. The TCR values of most resistors, fired on the dielectric-on-steel substrates, are higher than the TCRs of resistors on alumina substrates. This is attributed to the higher thermal expansion coefficient of the stainless steel when compared with alumina. Some "combinations," i.e., the 8241 resistor on the QM-42 multilayer dielectric and the 3414-A resistor on the D-4914 covered alumina substrate, have very high and irreproducible gauge factor values (over 200), which is attributed to the microcracks in resistors and therefore could not be used. Other tested thick-film resistor materials could be used on the dielectric-on-steel substrates if an allowance is made for the changed sheet resistivities and increased TCR.

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