

Thick-film resistors on zirconia substrates for possible strain gauge applications

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Abstract

Thick-film resistors that were developed for firing on alumina substrates have been evaluated in terms of their compatibility with ZrO₂. The characteristics of a number of 10 kohm/sq. resistors that were fired on 96% alumina and on tetragonal zirconia substrates were investigated. Possible interactions between the resistor material and the zirconia were studied with X-ray powder-diffraction analysis, with scanning electron microscopy and with energy-dispersive X-ray analysis. Sheet resistivities, temperature coefficients of resistivity, current noise and gauge factors were measured. The results indicate that the evaluated thick-film resistors are compatible with zirconia ceramics.

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1. Introduction

The change in resistance of a resistor under an applied stress is partly due to deformation, i.e. the changes in the dimensions of the resistor, and partly due to an alteration in the specific resistivity as a result of changes in the microstructure of the material.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 ($\Delta l/l$):

$$GF = (\Delta R/R)/(\Delta l/l) \quad (1)$$

Geometrical factors alone result in gauge factors of 2–2.5. Gauge factors higher than this are due to microstructural changes, i.e. changes to the specific conductivity. The GFs of thick-film resistors are mostly between 3 and 15. Due to their stability, temperature coefficients of resistivity (TCR) under $100 \times 10^{-6}/K$ and relatively low cost, strain gauges realised with thick-film technology offer advantages in some applications over both metal films (low GF, low TCR, expensive) and

semiconducting elements (high GF, high TCR, high expensive).^{2,3} Typically, four resistors connected in a Wheatstone bridge are used. For better sensitivity (greater change in the balance of the Wheatstone bridge), two resistors are normally under tension (an increase of resistance) and two under compression (a decrease of resistance). Within the same resistor series the GFs and the current noise indices of thick-film resistors increase with increasing sheet resistivity.^{4,5} Therefore, in most cases 10 kohm/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.

Thick-film resistors are printed and fired on a ceramic substrate (diaphragm), which is usually based on alumina. However, sensing elements made on partially stabilised tetragonal zirconia would have some improved characteristics as tetragonal zirconia has a higher mechanical strength and a lower modulus of elasticity than alumina.⁶ Therefore, the operating range and the mechanical strength of sensors are improved. Some mechanical characteristics for 96% Al₂O₃, which is mostly used for the substrates of thick-film circuits, and for tetragonal ZrO₂ (Y-TZP–Y₂O₃ stabilised tetragonal zirconia) are summarised in Table 1.

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Table 1
Some characteristics of 96% Al₂O₃ and Y-TZP (tetragonal ZrO₂)

| | Al ₂ O ₃ (96%) | Y-TZP |
|--|--------------------------------------|----------|
| Young's modulus E (GPa) | 250–330 | 200 |
| Flexural strength (MPa) | 300 | 800–1200 |
| Fracture toughness (MPa m ^{0.5}) | 3–3.5 | 6–8 |
| Poisson's ratio | 0.22 | 0.28 |
| Hardness (VH) (GPa) | 1600 | 1250 |
| Density (g/cm ³) | 3.6 | 6 |

By replacing an Al₂O₃ ceramic membrane with a ZrO₂ membrane of the same thickness the operating range can be extended or alternatively, thinner and therefore more flexible membranes could be used, which is important for low pressure sensors. This is shown schematically in Fig. 1 for a diaphragm with free edges and with fixed edges, respectively. The deformation of Al₂O₃-based and tetragonal ZrO₂-based membranes was calculated using the following parameters: membrane radius, 20 mm; thickness, 0.635 mm; and pressure, 1.5×10^4 Pa.

Thick-film resistor materials are a complex mixture of conductive particles, glass phase and added oxide modifiers. All the constituents of the resistor material react with each other and with the ceramic substrate during firing.^{7–10} The electrical characteristics of the resistors are the result of these reactions. As the thick-film resistors were developed for firing on alumina substrates, their compatibility with ZrO₂ needs to be evaluated.

Table 2
Conductive phase and summarised qualitative results of EDS microanalysis of elements detected in the glass phase of 10 k ohm² resistors^{11,12a}

| Resistor | Conductive phase | Main elements | Other elements |
|----------|------------------------------|---------------|----------------|
| 8039 | Ruthenate | Si, Pb, Al | Zr |
| 2041 | RuO ₂ + ruthenate | Si, Pb, Al | Mg, Zn, Ca, Ba |
| 3414-A | Ruthenate | Si, Pb | Al, K |
| 8241 | RuO ₂ | Si, Pb, Al | Ca |

^a Boron oxide, which is also present in the glass phase, cannot be detected in the EDS spectra.

In this paper the characteristics of some 10 kohm/sq. thick-film resistors, fired on alumina (see Refs. 11 and 12) and on zirconia substrates, will be compared. The tested resistors were 2041 (Du Pont), 8241 (Heraeus) and 3413-A (ESL). Electro Science Labs. The 3414 resistor series was specially formulated for high gauge factors.¹³ Data for 8039 resistors, fired on ZrO₂ substrates which were published elsewhere,¹⁴ are also included. The conductive phase in the 8039 and the 3414-A resistors is based on ruthenates, in the 2041 resistors on a mixture of RuO₂ and Pb₂Ru₂O_{6.5}, and in the 8241 resistors on RuO₂. Data on the conductive phase and the qualitative results of an energy-dispersive X-ray analysis (EDS) of the glass composition of the thick-film resistors are summarized in Table 2.^{11,12} All glasses contain, as main elements, lead, silicon and aluminum 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_α line during EDS analysis in the glass matrix.

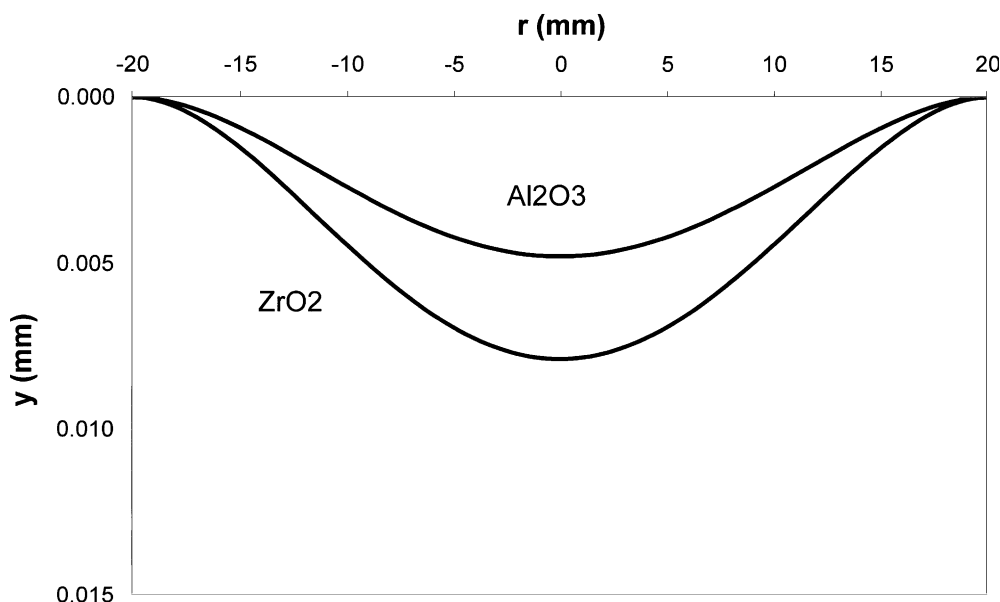


Fig. 1. The deformation of Al₂O₃-based and tetragonal ZrO₂-based ceramic membranes with fixed edges under a pressure of 1.5×10^4 Pa.

2. Experimental

Thick-film resistors, terminated with a prefired Pd/Ag conductor, were printed and fired at 850 °C for 10 min on 96% alumina and on tetragonal ZrO₂ (Y-TZP) substrates. The zirconia substrates were obtained from CoorsTek, CO, USA. For the electrical measurements the resistors (1×1 mm²) were terminated with a prefired Pd/Ag conductor. The dimensions of the resistors for microstructural analysis and X-ray diffraction (XRD) analysis, which were printed and fired without conductor terminations, were 12.5×12.5 mm².

Cold (from –25 to 25 °C) and hot (from 25 to 125 °C) TCRs were calculated from resistivity measurements at –25, 25, 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 ceramic substrate with a printed-

and-fired thick-film resistor in the middle of the substrate 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), *t* is the substrate thickness (m), *X* is the distance from the supported edge of the substrate (m) and *L* is the distance between the support edges (m).

$$\varepsilon = \Delta l/l = (d^*t^*X^*12)/L^3 \quad (2)$$

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

$$\varepsilon = \Delta l/l = (d^*t^*6)/L^2 \quad (3)$$

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

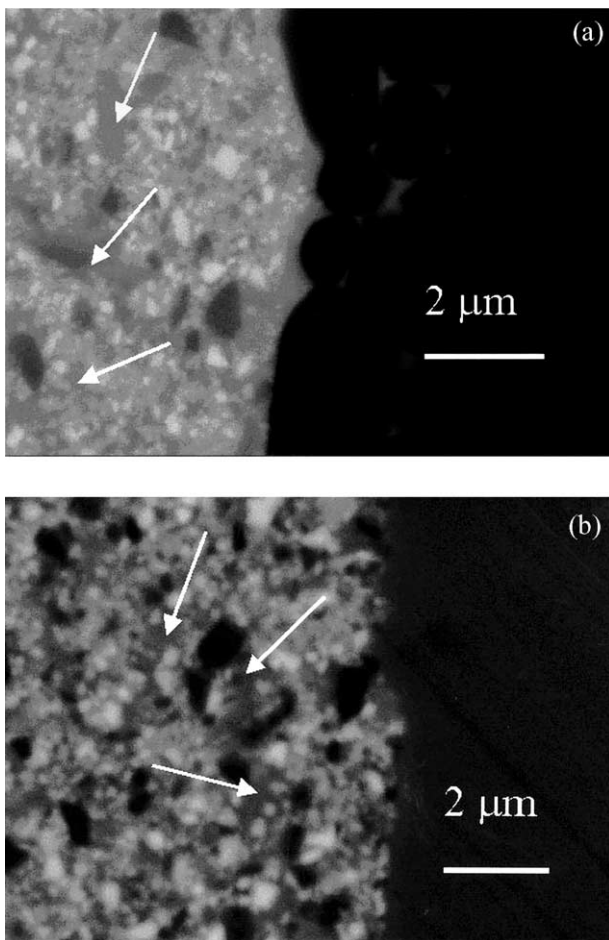


Fig. 2. (a) Microstructure of a cross-section of the 8039 thick-film resistor, fired at 850 °C on alumina. The substrate is on the right. Glass phase is indicated by arrows. (b) Microstructure of a cross-section of the 8039 thick-film resistor, fired at 850 °C on zirconia. The substrate is on the right. Glass phase is indicated by arrows.

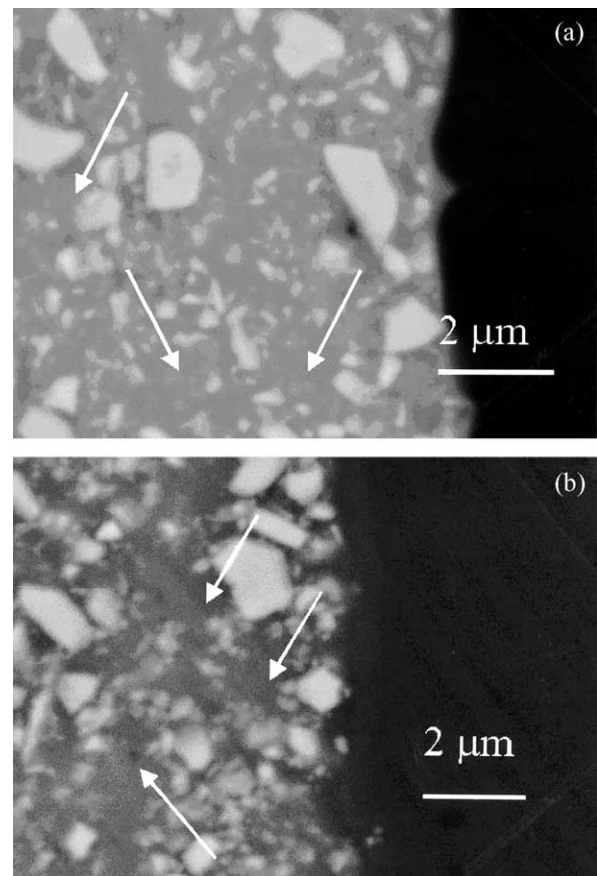


Fig. 3. (a) Microstructure of a cross-section of the 3414-A thick-film resistor, fired at 850 °C on alumina. The substrate is on the right. Glass phase is indicated by arrows. (b) Microstructure of a cross-section of the 3414-A thick-film resistor, fired at 850 °C on zirconia. The substrate is on the right. Glass phase is indicated by arrows.

For the microstructural investigation the resistors, which were printed and fired on alumina ceramics, were mounted in epoxy in a cross-sectional orientation and then cut and polished using standard metallographic techniques. A JEOL JSM 5800 scanning electron microscope (SEM) was used for the microstructural analysis. Prior to analysis in the SEM, the samples were coated with carbon to provide electrical conductivity and to avoid charging effects. The resistors, that were fired on alumina and zirconia substrates were analysed by X-ray powder diffraction (XRD) analysis with a Philips PW 1710

X-ray diffractometer using Cu K_{α} radiation. X-ray spectra were measured from $2\Theta=20^{\circ}$ to $2\Theta=70^{\circ}$ in steps of 0.04° .

3. Results and discussion

At the interface between ZrO_2 and the resistor no interactions or evidence for new phases could be detected by SEM and EDX. These results indicate the compatibility of the resistor material and the ZrO_2 . The examples of microstructures of the cross-sections of

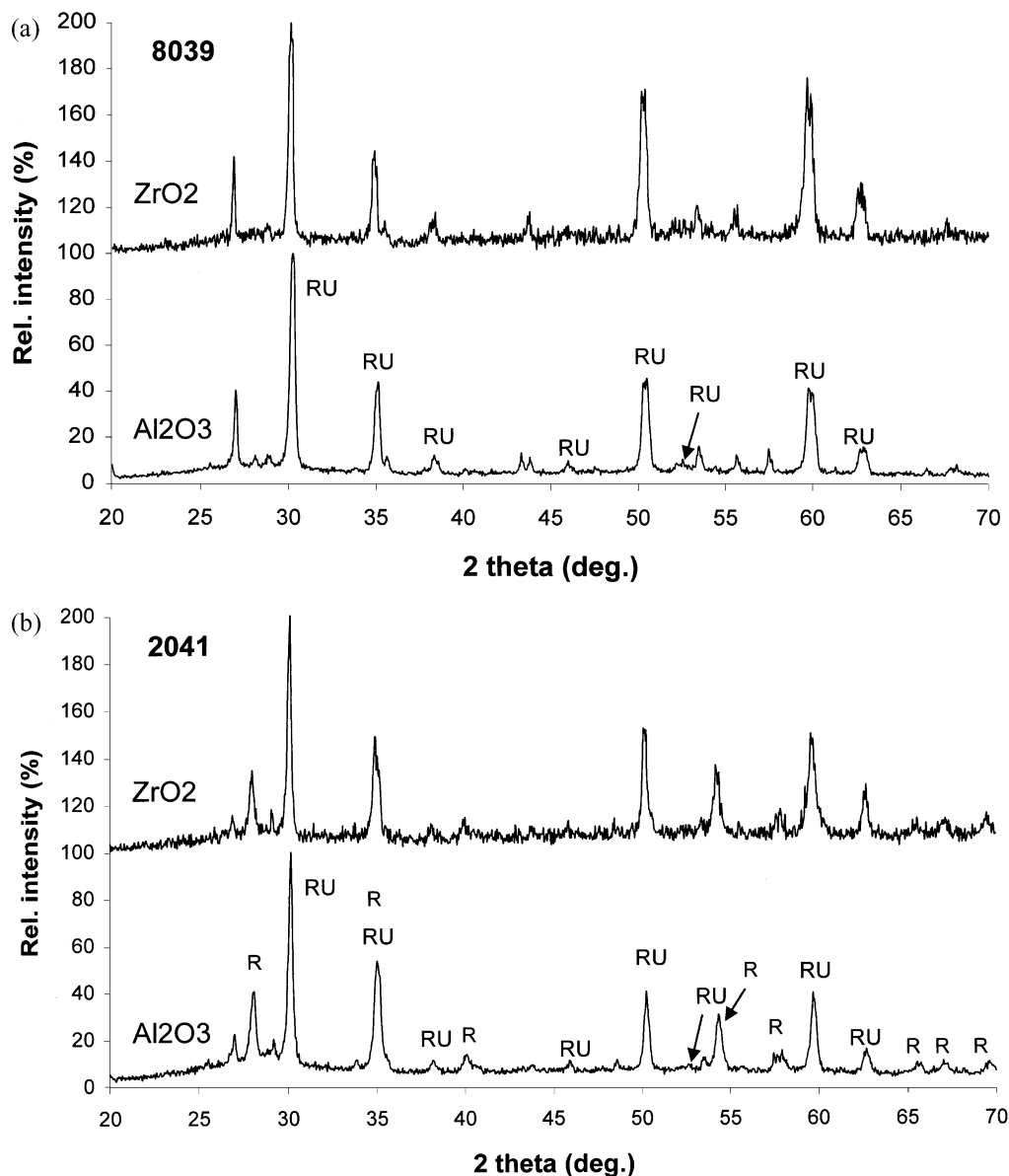


Fig. 4. (a) X-ray spectra of 8039 resistors, fired on Al_2O_3 and ZrO_2 substrates. Peaks of ruthenate phase are denoted "RU". (b) X-ray spectra of 2041 resistors, fired on Al_2O_3 and ZrO_2 substrates. Peaks of RuO_2 and ruthenate phase are denoted "R" and "RU", respectively. (c) X-ray spectra of 3414-A resistors, fired on Al_2O_3 and ZrO_2 substrates. Peaks of ruthenate phase are denoted "RU". (d) X-ray spectra of 8241 resistors, fired on Al_2O_3 and ZrO_2 substrates. Peaks of RuO_2 are denoted "R".

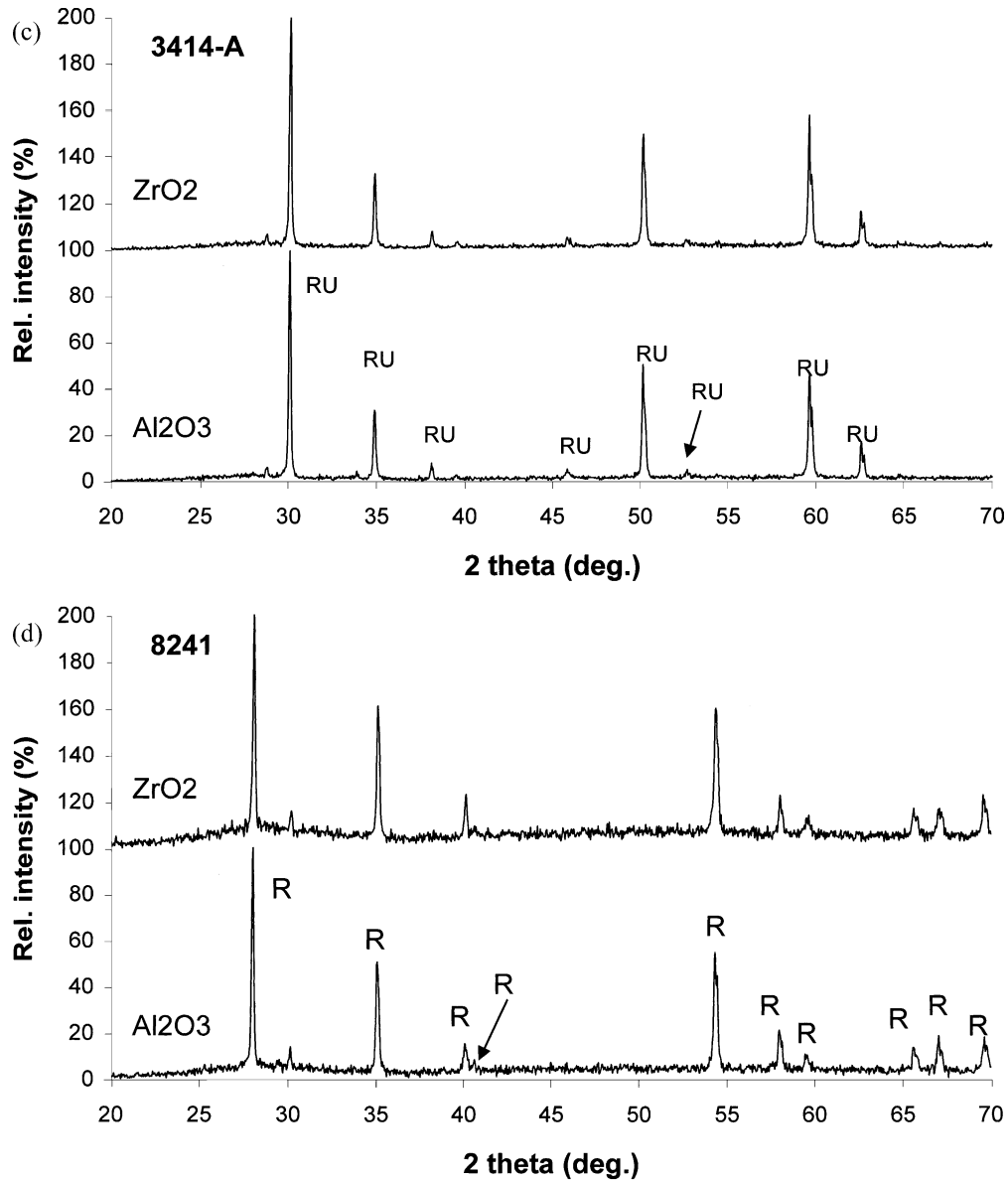


Fig. 4. (continued)

Table 3

Sheet resistivities, cold (−25 to 25 °C) and hot (25 to 125 °C) TCRs, noise indices and gauge factors (GFs) of the 10 k ohm² resistors fired on the Al₂O₃ and ZrO₂ substrates

| Resistor | Substrate | R (k ohm ²) | Cold TCR (×10 ^{−6} /K) | Hot TCR (×10 ^{−6} /K) | Noise (dB) | GF |
|----------|--------------------------------|----------------------------|------------------------------------|-----------------------------------|---------------|----|
| 8039 | Al ₂ O ₃ | 7.15 | 50 | 95 | −14.6 | 11 |
| | ZrO ₂ | 9.20 | 115 | 150 | −13.8 | 10 |
| 2041 | Al ₂ O ₃ | 6.70 | −35 | 20 | −23.4 | 11 |
| | ZrO ₂ | 5.75 | 105 | 150 | −21.6 | 10 |
| 3414-A | Al ₂ O ₃ | 6.70 | −60 | 5 | 8.2 | 21 |
| | ZrO ₂ | 11.2 | 40 | 105 | 0.55 | 16 |
| 8241 | Al ₂ O ₃ | 5.45 | 25 | 65 | −6.0 | 15 |
| | ZrO ₂ | 1.44 | 245 | 275 | −9.2 | 13 |

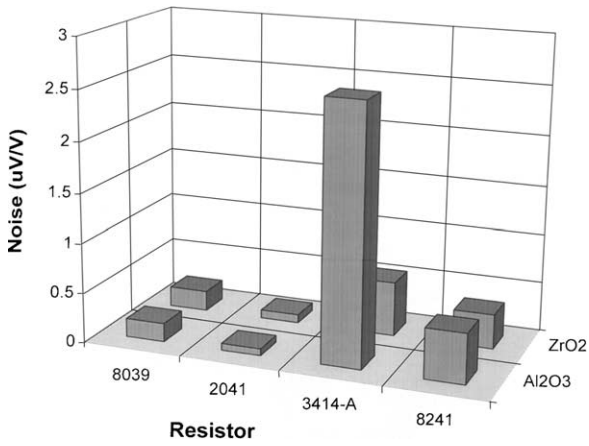


Fig. 5. Noise indices of resistors, fired on the Al_2O_3 and ZrO_2 substrates.

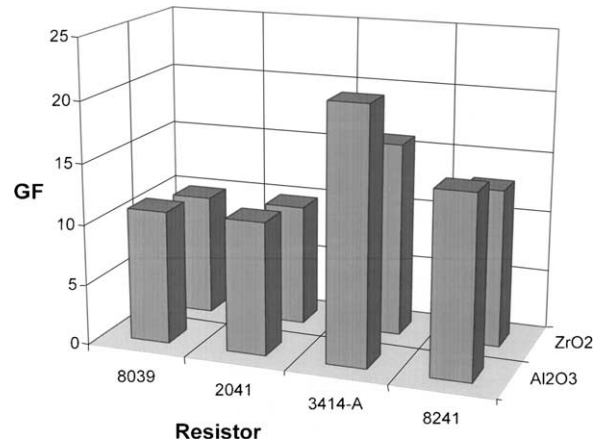


Fig. 6. Gauge factors of resistors, fired on the Al_2O_3 and ZrO_2 substrates.

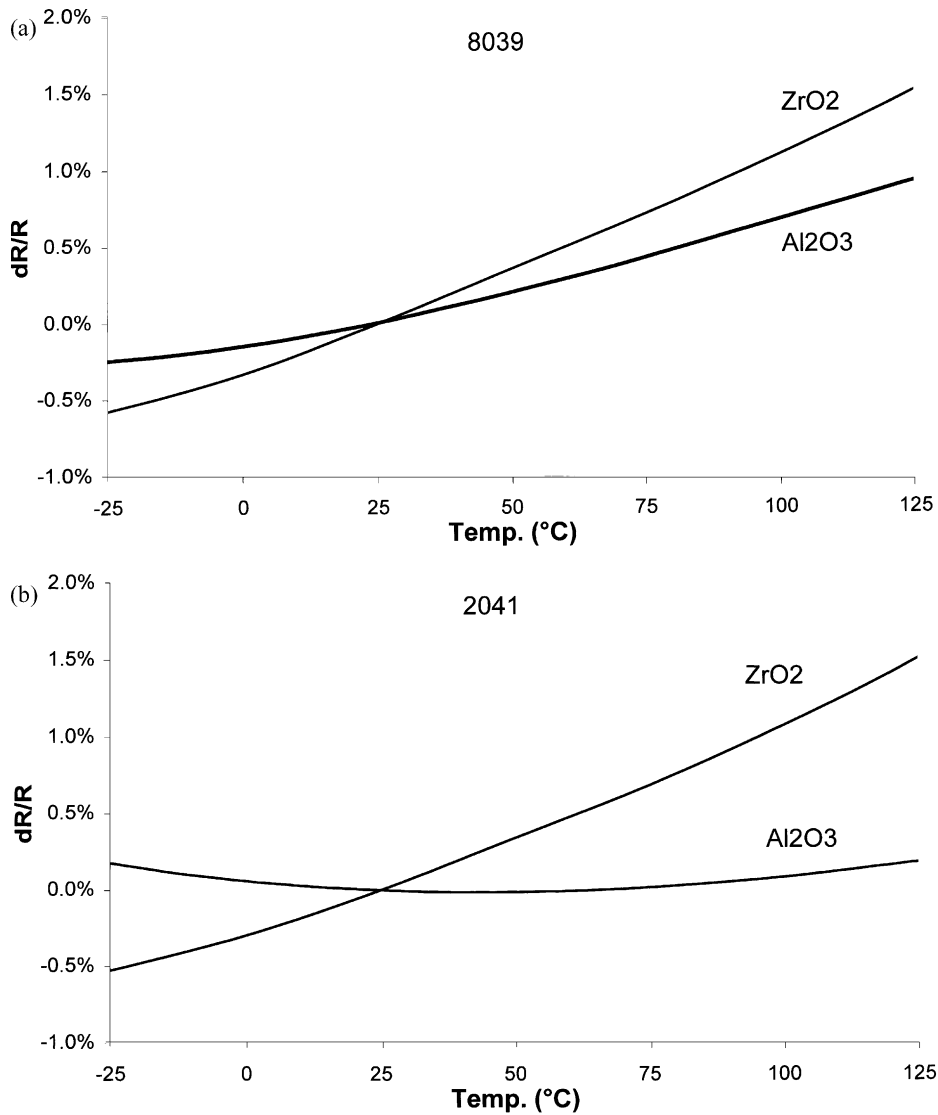


Fig. 7. (a) The dependence of relative resistivity on temperature for 8039, fired at 850 $^{\circ}\text{C}$ on Al_2O_3 and ZrO_2 substrates. (b) The dependence of relative resistivity on temperature for 2041, fired at 850 $^{\circ}\text{C}$ on Al_2O_3 and ZrO_2 substrates. (c) The dependence of relative resistivity on temperature for 3414-A, fired at 850 $^{\circ}\text{C}$ on Al_2O_3 and ZrO_2 substrates. (d) The dependence of relative resistivity on temperature for 8241, fired at 850 $^{\circ}\text{C}$ on Al_2O_3 and ZrO_2 substrates.

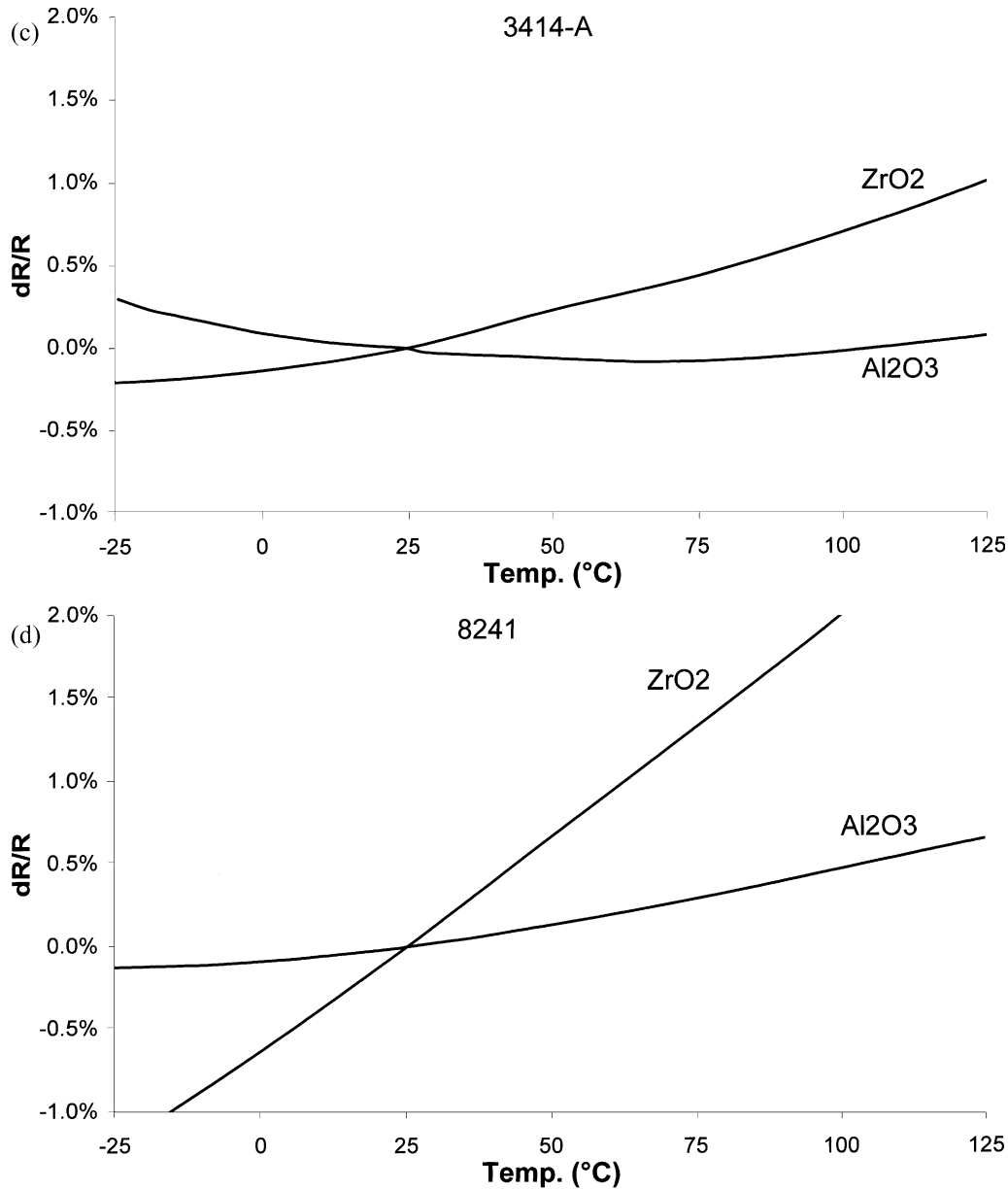


Fig. 7. (continued)

some $10 \text{ k } \Omega^2$. resistors that were fired at $850 \text{ }^\circ\text{C}$ on alumina and zirconia substrates, are shown in Figs. 2a and b, and 3a and b for and 3414-A, respectively. The ceramic substrate is on the right. The microstructure of resistors consists of light grains of conductive phase imbedded in the grey glass phase. The glass phase is indicated by arrows.

The X-ray spectra of the resistors, that were fired on Al_2O_3 and ZrO_2 substrates are shown in Fig. 4a–d. The peaks of RuO_2 and the ruthenate phase are denoted “R” and “RU”, respectively. The spectra are nearly the same, which confirms the compatibility of the tested $10 \text{ k } \Omega^2$. resistors with the zirconia ceramics.

Sheet resistivities, cold (-25 to $25 \text{ }^\circ\text{C}$) and hot (25 to $125 \text{ }^\circ\text{C}$) TCRs, noise indices and gauge factors (GFs) of the resistors fired on the alumina and zirconia ceramics are presented in Table 3. The noise indices and GFs are also shown graphically in Figs. 5 and 6, respectively. The dependence of the relative resistivity vs. temperature is presented in Fig. 7. However, note that in Fig. 5 the noise indices are expressed in “uV/V” while in Table 3. they are given as “dB”. These two units are related with an equation:

$$\text{noise (dB)} = 20 \times \log \text{ noise (uV/V)}$$

The sheet resistivities and gauge factors of the resistors on the Al_2O_3 and ZrO_2 substrates are comparable. The GFs of 8241 resistors on alumina are around 15. This value is slightly higher than the value reported in the literature (GF around 13).¹⁷ The noise indices are also comparable with the exception of the 3414-A that was fired on alumina which had a significantly higher noise of 2.75 $\mu\text{V}/\text{V}$. The noise indices of 2041 resistors have the lowest values. However, the TCRs of the resistors on the ZrO_2 are considerably higher. The hot TCR values are in all cases over $100 \times 10^{-6}/\text{K}$. The highest values, over $250 \times 10^{-6}/\text{K}$ were measured for 8241. This is probably due to the higher thermal expansion coefficient of the tetragonal zirconia when compared with alumina (see Table 1).

4. Conclusions

The characteristics of some 10 k ohm^2 thick-film resistors that were fired on alumina and on tetragonal zirconia substrates were investigated with the aim of determining the compatibility of the resistor materials which were developed for firing on Al_2O_3 , with ZrO_2 substrates. At the interface between the ZrO_2 and the resistor no interactions or evidence for new phases could be detected by SEM analysis. The X-ray spectra of the resistors fired on Al_2O_3 and ZrO_2 substrates are nearly the same. The sheet resistivities and noise indices (with the exception of the 3414-A resistors) of the resistors on Al_2O_3 and ZrO_2 are comparable. The high positive TCRs of the resistors on the ZrO_2 can be attributed to the higher thermal expansion coefficient of tetragonal zirconia. All the obtained results confirm the compatibility of the evaluated thick-film resistors with tetragonal zirconia ceramics. Therefore, the tested thick-film resistor materials could be made on tetragonal zirconia substrates if an allowance is made for the increased TCR.

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