# Influence of film thicknesses on the electrical properties of RuO$_2$-thick film resistors on aluminum nitride ceramics (AlN)

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## Abstract

*Aluminum nitride ceramics are very suitable as substrate material for thick film- / hybrid applications in the field of power electronics and microwave technology. Main features are high thermal conductivity, a thermal expansion coefficient close to silicon, good mechanical stability and dielectric properties. Thick film resistor pastes are usually composed of lead- and bismuth oxide free glass frit and ruthenium dioxide as electrical conductive component. In contrast to oxide ceramics like alumina ceramics, thermo dynamical calculations predict the reaction of ruthenium dioxide with AlN. Thus, resistor pastes for AlN are designed to build defined porosity in the films. In dependence of composition of the paste, substrate roughness and -quality varying interactions of the components and deviating film structures can be observed. On basis of different paste compositions resistors were built with varying film thicknesses. Electrical properties like resistance and its temperature coefficient as well as the power dissipation and short term overload voltage were correlated with geometrical data, chemical composition and substrate properties. Furthermore FESEM was used to characterise film properties like porosity and the interface resistor – ceramics. Users of thick film pastes are shown aspects under which conditions high quality resistors are maintained under maximum materials savings.*

Key words: aluminium nitride, RuO$_2$ thick film paste, resistors, film thickness

## Introduction

Aluminum nitride (AlN) is an excellent substrate material for thick film hybrid technology due to its material properties. Compared with alumina (Al$_2$O$_3$) or Low Temperature Cofiring Ceramics (LTCC), AlN shows a high thermal conductivity, thermal shock resistance and mechanical strength. Combined with a thermal expansion coefficient close to that of silicon it is predestined for highly sophisticated microelectronic applications with surface mounted Si-Chips for power electronics, RF- and microwave applications. Also, the requirements in terms of process control, stability and reliability are very high whereas the price should be low. Thus one way for optimisation could be to achieve lower film thicknesses to save precious metal containing pastes. Therefore, the influence of film thickness on RuO$_2$-based thick film resistors is investigated. Thereby the effect that the conductive phase RuO$_2$ can react with AlN [1] after the formula

$$4AlN + 3RuO_2 \xrightarrow{Glass} 2Al_2O_3 + 3Ru + 2N_2 \uparrow \quad (1)$$
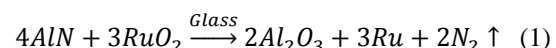
becomes more noticeable at smaller thicknesses and so deviating film properties could become a problem. Which influence the film thickness has on the deviation was investigated by determination of electrical properties (R$_{sq}$, TCR, STOL) in accordance with film thicknesses and substrate properties. Especially the roughness of the substrate is supposed to create an influence due to the fact that with increasing value more surface are is provided where the reaction can take place.

### Experimental procedure

*Paste formulation*

The paste was formulated according to FK9621 of IKTS AlN paste system FK9600 containing a glass phase, a conductive phase

(RuO$_2$), a TCR modifier and an inorganic additive dispersed in an organic vehicle which is created out of a solvent and a polymer. Powders were mixed in a mill and brought into an organic vehicle, which is added to achieve a screenprintability. Afterwards the paste was processed on a three roll mill to make sure the particles were fine-dispersed.

*Sample preparation and test methods*

The paste has been screen printed with an EKRA Microtronic II printer on different one by one inch sized AlN substrates from various suppliers.
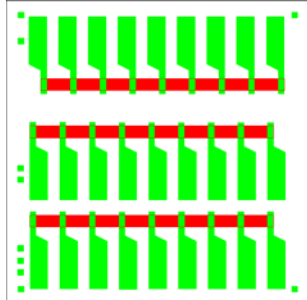


**Figure 1: Layout of Resistors**

The screen print layout is shown in Figure 1. One substrate contains 24 resistors (Fig. 1: red) with a geometry of 2x1 mm².

The contacts are build of an AgPd conductor paste FK1220 of Fraunhofer IKTS thick film paste system (Fig. 1: green) which was screen printed with a 200 mesh screen, levelled at room temperature for 10 Minutes, dried for 15 minutes at 150 °C and fired at 850 °C in an air atmosphere in a belt furnace with a dwell of 10 minutes and total cycle time of 60 minutes.

Resistors itself were screen printed with varying screens (200 mesh, 250 mesh, 325 mesh, 510 mesh), with levelling, drying and firing comparably to the contact material. One exception was made to achieve highest thicknesses: For that, the film was obtained by two printing-levelling-drying-steps with a 250 mesh screen before the substrates were fired. All film thicknesses were measured with an auto focusing laser scanning profilometer of UBM.

Resistance was measured with a quasi four point method at 30 °C for precise results. From all 24 resistors an arithmetic average was calculated for each substrate. The temperature coefficient of the resistance (TCR) was obtained by measuring the resistors at varying temperatures between -55 and 150 °C on the fly. For better comparison of different results each curve was fitted by method of least squares and a linear (AQ) and quadratic (BQ) coefficient was obtained. With these the results could be extrapolated. To compare temperature-resistance-plots directly, a relative change of resistance was calculated by normalising the measured values to the resistance at 25 °C by

$$\frac{R}{R_{25}} = 1 + AQ \cdot (T - T_{25}) + BQ \cdot (T - T_{25})^2 \quad (2)$$

The TCR itself is defined by

$$TCR = \frac{R - R_0}{R_0} \cdot \frac{1}{T - T_0} \cdot 10^{-6} \quad (3)$$

For characterisation of samples under electrical load, the short term overload (STOL) voltage and its accompanying parameter maximum rated power dissipation (MRPD) were calculated from results. For this, the substrate was thermally coupled with a cooling device by a heat-conducting paste. Then the resistor in the middle of the substrate was connected to a power source and an infrared pyrometer was used to determine the temperature of the resistor. Initially the resistance at room temperature is measured. Then electrical load is applied for 5 seconds. At the end of this pulse the resistance R$_{hot}$ and temperature T are measured. Then a 60 seconds break is followed for cooling the substrate to room temperature. At the end of the break the resistance R$_{cold}$ and T are measured before applying another pulse of load. With every load pulse the power is increased by 5 Watts. The main principle of the measurement is also shown in Figure 2.
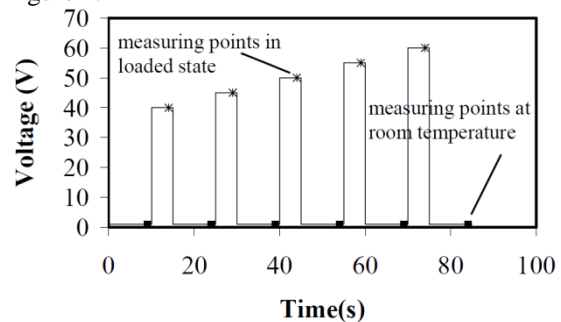


**Figure 2: Principle of STOL measurement**

STOL voltage is reached when the change of resistance $\Delta R_{cold}/R_{cold}$ exceeds 0.1%. The maximum rated power dissipation (MRPD) is then calculated by

$$MRPD = \frac{(0.4 \cdot U_{STOL})^2}{R_{sq}} \quad (4)$$

Surface and cross sections of the films of each substrate were characterised by field emission scanning electron microscopy (FESEM) in SE and BSE mode with an NVision 40 of Carl Zeiss AG.
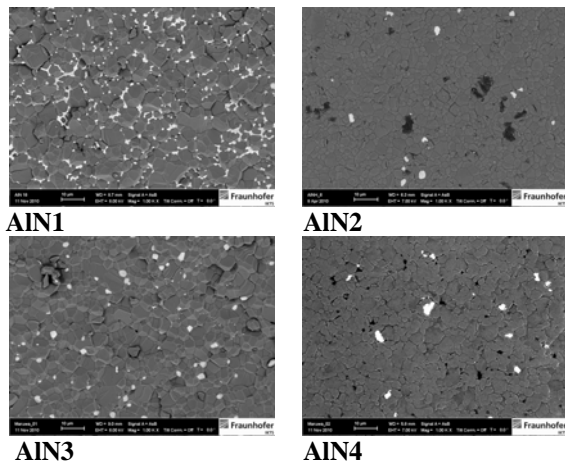
**Results and Discussion**

**1. AlN substrate characterisation**

The surfaces of AlN substrates were characterised in terms of roughness and content of sinter aid phase. Roughnesses differ for the varying substrates. The highest roughness observed has AlN1 while AlN4 has lowest substrate roughness as stated in Table 1. This is a result of the manufacturing processes used by the substrate suppliers. A wide range of roughness is covered so
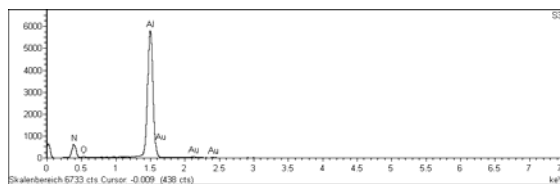
that possible impacts on film properties could be observed.
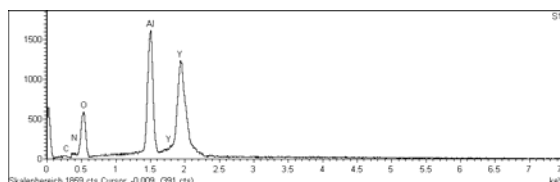
**Table 1: Substrate roughness**

| Ceramic | roughness $R_a$ [µm] | Manufactured quality |
|---------|---------------------|----------------------|
| AlN1 | 0,95 | lapped |
| AlN2 | 0,28 | asfired, brushed |
| AlN3 | 0,38 | lapped |
| AlN4 | 0,17 | asfired |



AlN1       AlN2

AlN3       AlN4

**Figure 3: substrate surfaces**



**Figure 4: EDX-spectrum of AlN4, dark area**



**Figure 5: EDX-spectrum of AlN4, light area**

Figure 3 shows FESEM images (BSE-Mode) of the AlN surfaces. Dark areas picture the AlN (compare Figure 4) whereas lighter areas remark the residual sinter aid yttrium aluminate (see Figure 5). On the surface of AlN2 to AlN4 is the concentration substantially lower than on AlN1. On AlN1 yttrium aluminate phases are obviously solidified out of the melt around the grain boundaries and partially well crystallised in the grain interstices (see Figure 3 AlN1).

The reason for that is that depending on the content of inevitable alumina in the AlN powder yttrium oxide is added as sinter aid. While sintering the AlN tapes $Al_2O_3$ and $Y_2O_3$ form various yttrium aluminates in dependence of available $Y_2O_3$ and of firing conditions: yttrium aluminium garnet (YAG, $Y_3Al_5O_{12}$), yttrium aluminium perovskite (YAP,

AlYO3) and yttrium aluminium monoclinic (YAM, $Y_4Al_2O_9$). Therefore it can be said that for AlN1 apparently a powder with higher oxygen respectively $Al_2O_3$ content was used.

## 2. Resistivity and its temperature coefficient

The resistance R is determined in dependence of the fired film thickness. Achieved thicknesses varied between 7 and 33 µm. Thereby the resistance is defined by following formula

$$R = \rho \cdot \frac{l}{w} \cdot \frac{1}{d} \tag{5}$$

and if length l is equal with witdh w, the so called sheet resistivity is then defined by

$$R_{sq} = \rho \cdot \frac{1}{d} \tag{6}$$

From transponsed formula (6) the resistivity ρ was calculated from measured results. It is shown in dependence of fired film thickness in Figure 6.
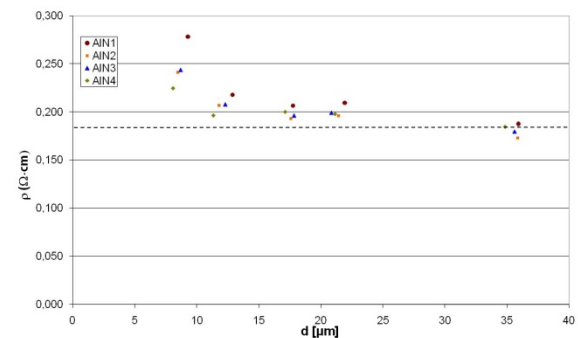


**Figure 6: resistivity ρ against thickness d**

At thickness values exceeding 15 µm a nearly constant behaviour can be seen, as expected. But the lower the film thicknesses become the more the results are deviating from calculated average of $\rho = 1.812 \cdot 10^{-1}$ Ω·cm (dashed line in Figure 6). A possible explanation for this behaviour would be the formation of an intermediate layer between resistor film and substrate due to the interaction of $RuO_2$ and AlN (1). To predict a thickness of this layer a model was raised which assumes that the interface layer and the bulk form two resistors connected in parallel (see Figure 7).
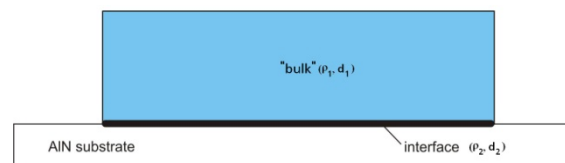


**Figure 7: model of resistors connected in parallel**

From that the sheet resistivity could be calculated by the formula

$$R_{sq} = \frac{R_{1,sq} \cdot R_{2,sq}}{R_{1,sq} + R_{2,sq}} \tag{7}$$

With $R_{1,sq} = \frac{\rho_1}{d_1} = \frac{\rho_1}{d-d_2}$ (8) and $R_{2,sq} = \frac{\rho_2}{d_2}$ (9) a fit has been carried out for the condition

$$\sum_i \left( R_{sq} - R_{sq}^{calc} \right) \xrightarrow{!} minimum \qquad (10)$$

for the five thickness values of each substrate series where the resistivity of the bulk was defined by the average value calculated above. This led to the following results stated in Table 2.

**Table 2: Results of fit**

| Substrate | $\rho_{"bulk"}$ [Ohm·cm] | $\rho_{"interface"}$ [Ohm·cm] | $d_{"interface"}$ [µm] |
|---|---|---|---|
| AlN1 | 0.1812 | 79.86 | 2.80 |
| AlN2 | 0.1812 | 35.50 | 1.75 |
| AlN3 | 0.1812 | 40.84 | 1.88 |
| AlN4 | 0.1812 | 13.96 | 1.29 |

It can be seen that the model predicts an intermediate layer thickness of 2.8 to 1.29 µm. Also the influence of substrate roughness is visible, roughest AlN1 has the highest interface thickness whereas smoothest AlN4 has lowest parameter.

For investigation of TCR behaviour, the resistance has been measured between -55 and 150 °C and it showed a quadratic dependence of the temperature with an resistance minimum at a temperature $T_{min}$. The TCR is the slope of the curve. It is usual to divide the TCR in so-called Hot-TCR (HTCR) from 25 °C … 150 °C and Cold-TCR (CTCR) from -55 ... 25 °C. An illustration of a measurement gives Figure 8.
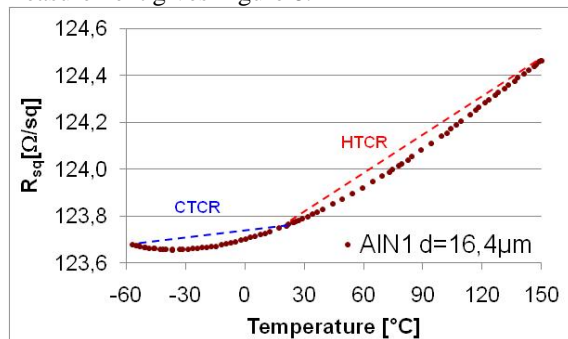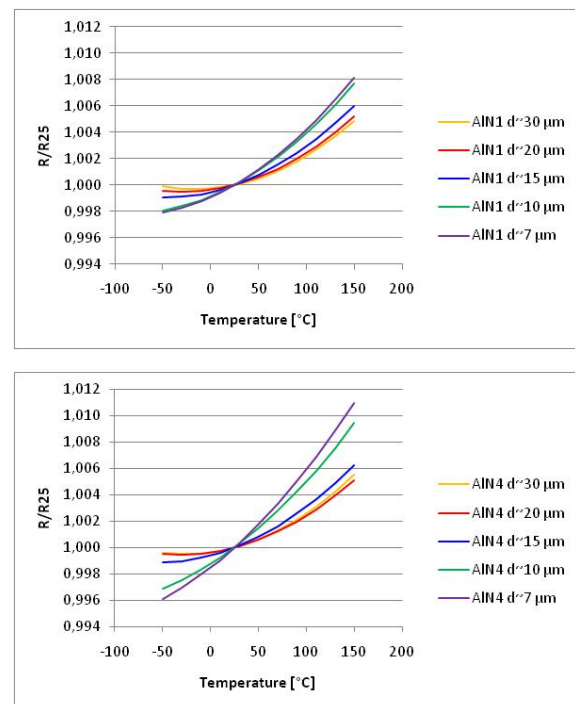
**Figure 8: TCR measurement**

For each film thickness and substrate material, the data has been collected. As an overview all values for HTCR and CTCR are stated in Figure 9.
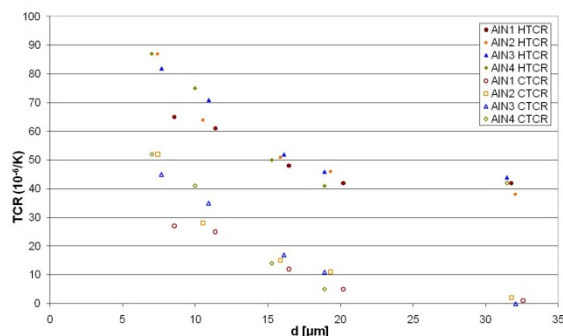
**Figure 9: Overview of Hot- and Cold-TCRs**

The behaviour of all substrates generally is comparable since HTCRs and CTCRs are increasing with decreasing film thickness for all substrate qualities and all CTCRs are lower than HTCRs. The only obvious characteristic in Figure 9 is that AlN1 stands out with lower values than the other three substrates. Nevertheless, all results meet the requirement that this property has to be less or equal than $100*10^{-6}$ K$^{-1}$ since it is specified so for the paste FK9621. Figure 9 in combination with Figure 6 shows a direct proportionality of TCR and resistivity.

For better comparison of TCR measurements the curves were normalised to resistance of T=25 °C and partially extrapolated by regression with the formula (2) to obtain $T_{min}$.

**Figure 10: relative resistivity compared for varying film thicknesses and substrates**

From the results of resistivity and TCR measurements it can be noticed that the resistivity is rising accordingly with TCR in dependence of decreasing film thickness. Parallely, $T_{min}$ is shifted to lower values. These effects are more clearly obtained for AlN1 than for AlN4. Thus, the assumption that resistor films interact more with rough AlN1 surface than with smoother substrate surface like AlN4 is underlined. An explanation can be that the chemical reaction (1) of $RuO_2$ and AlN is responsible for the formation of an intermediate layer between film and substrate. The thickness of the layer is constant for equal substrate qualities. In contrast, the substrate roughness has a direct influence of the thickness of this intermediate layer. The formation of ruthenium was proofed on model samples in [1] which also support this. The impact of the reaction products on the film properties are

complex and each product can influence them antithetic. At first, Ruthenium has got a higher TCR and lower resistivity. Additionally, ruthenium has a lower molar volume as ruthenium dioxide. These facts do directly influence conduction properties of the film. Also the incidental formed alumina acts as inert phase and can influence the glass composition and viscosity during firing which can respond in changed properties of the films. Properties of Ru and $RuO_2$ are drawn together in Table 3.

Table 3: comparison of material properties [2]

| Property | Ru | $RuO_2$ |
|---|---|---|
| Resistivity [Ohm·cm] | $6·10^{-6}$ | $4·10^{-5}$ |
| TCR [$10^{-6}$ $K^{-1}$] | 4200 | (5800*) |
| Molar volume [cm³/g] | 8.2 | 19 |

The value marked with * is obtained from single crystals of $RuO_2$ whereas the sintered powder material does have losses at grain boundaries.

### 3. STOL measurements

STOL measurements were carried out for the two different substrate materials AlN1 and AlN2 with variable printed resistors thickness. The obtained values are shown in Table 4. The MRPD of all substrates are comparable around 4.5 W/mm². An impact of film thickness or substrate material to the power loss properties cannot be found.

Table 4: STOL values

| Ceramic | d [µm] | STOL-Voltage [V/mm] | MRPD [W/mm²] |
|---|---|---|---|
| AlN1 | 32.5 | 38.4 | 4.3 |
| AlN1 | 20.2 | 54.6 | 4.7 |
| AlN1 | 11.4 | 81.2 | 5.6 |
| AlN1 | 8.5 | 89.4 | 4.8 |
| AlN2 | 31.8 | 36.3 | 3.9 |
| AlN2 | 19.3 | 53.7 | 4.7 |
| AlN2 | 10.5 | 68.7 | 4.0 |
| AlN2 | 7.4 | 68.7 | 3.6 |

The development of resistance change against power is illustrated in Figure 11.
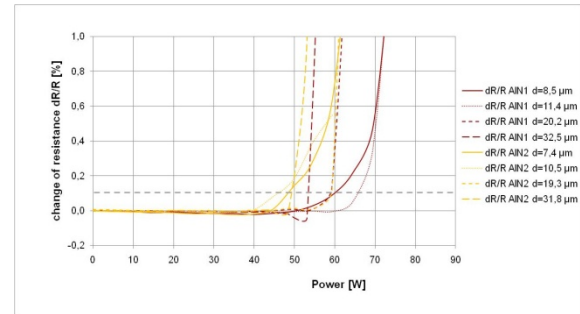


Figure 11: resistance change - power diagram

The dashed grey line marks the 0.1 % border where STOL –Voltage is calculated. Except for AlN1 with fired film thickness of 11.4 µm respectively 8.5 µm the graphs are close together. A systematic influence is not obvious. Corresponding to the resistance change, the temperature measured after a load pulse was drawn against the power load (Figure 12). These curves show for thinner films a shift to lower temperatures at same power level. This is understandable because in thick films, heat must be transferred through the film to the substrate whereas at thinner films the distance to the substrate is smaller and thermal dissipation rather fast. Thus, thinner films can be charged with higher load at same temperature level.
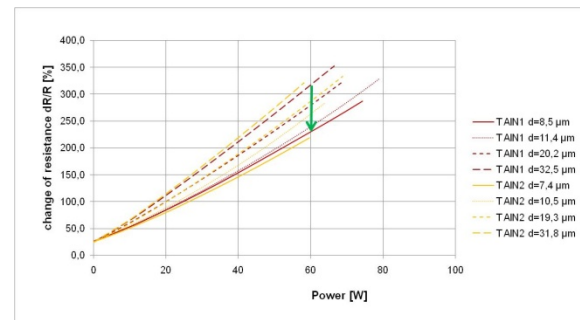


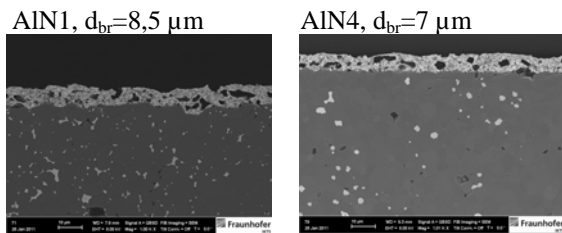Figure 12: resulting temperatures from STOL

### 4. Film characterisation by SEM

Cross sections of fired films were made and characterised by electron microscopy to investigate the supposed interface layer and film building behaviour in general.

AlN1, $d_{br}$=20,2 µm    AlN4, $d_{br}$=18,5 µm
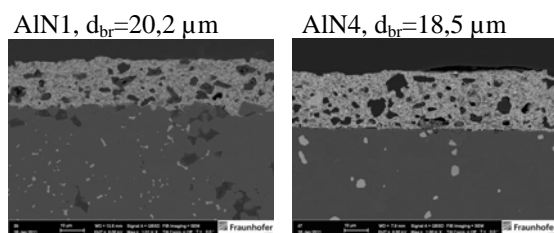


Figure 13: SEM-images of cross-section of fired films

AlN1, $d_{br}$=8,5 µm          AlN4, $d_{br}$=7 µm



**Figure 14: SEM-images of cross-section of fired films**

The images of Figure 13 and Figure 14 show high and low thicknesses in contrast with rough and smooth substrate surfaces. Generally, no noticeable differences can be described. The for the film formation necessary porosity is built in every film and there are no outstanding particles or agglomerations of any phase visible. Highly resolving pictures where prepared too.
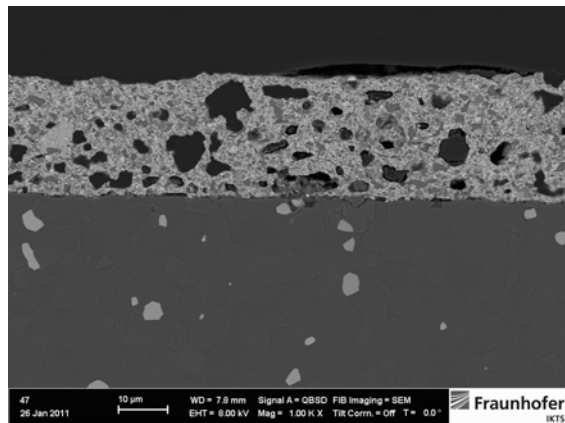


**Figure 15: overview of a resistor film (AlN4, d=18,9 µm)**

Exemplarily, Figure 14 shows that no intermediate layer could be visibly evaluated. Additionally, EDX analysis has been carried out near the substrate surface to investigate changes in composition.
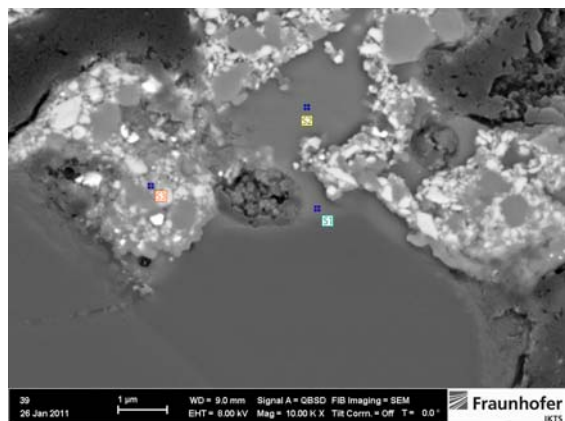


**Figure 16: cross section of fired film on AlN1 with EDX spots**

**Table 5: Analysis of EDX spetra**

| Point | Detected elements |
|---|---|
| S1 | Glass, conductive phase, TCR modifier, inert phase |
| S2 | Glass, TCR modifier |
| S3 | Glass, conductive phase, TCR modifier, inert phase |

An interface film showing traceable amounts of Ruthenium cannot be detected, as additionally can be seen in Table 5. Furthermore, changes between bulk and near the substrate surface are hardly visible.

**Conclusion**

It was shown that the specific resistivity and its temperature coefficient increase with decreasing fired film thickness of the resistors of paste FK9621. This effect is intensified when substrate material is used with high roughness (AlN1). Accordingly, smooth surfaces show less impact (especially AlN4). The reason for this behaviour is the formation of an intermediate layer where the composition of the resistor changes due to reaction products formed during firing. Model calculations showed that this layer is 1.3 to 2.8 µm in height. The intermediate layer could not be detected by FESEM and EDX investigations, although the formation of ruthenium was proved at test specimen by [1]. At last, resistors with varying film thickness tolerate comparable power loss values. At a constant power level, thin resistor films do not heat up that much like thick resistor films. Respectively, lighter resistors are able to dissipate more power at same temperatures.

**References**

[1] C. Kretzschmar et al., "The reaction between ruthenium dioxide and aluminium nitride in resistor pastes", J. Mat. Sci. 28 (1993) p. 5713 – 5716

[2] Pierce et al., "The chemistry and stability of ruthenium-based resistors"; Proceedings of 3rd European Hybrid Microelectronics Conf., Avignon, France 1981.