Low Temperature Co-fired Ceramics Processing Parameters Governing the Performance of Miniaturized Force Sensors

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Abstract

For the development of miniaturized force sensors, built up in ceramics technology with piezo-resistive principle, the compatibility of the piezo-resistive thick-film paste with the substrate and termination paste has to be verified. This paper deals with the compatibility of the ESL 3414-A piezo-resistive paste on HTCC (high temperature co-fired ceramics) substrates (alumina as reference and the partially stabilized zirconia tape ESL 42013-A) as well as on LTCC (low temperature co-fired ceramics) substrates (HERAEUS AHT01-005, AHT08-047, CT707; and CERAMTEC GC) under different manufacturing conditions. The sheet resistance at room temperature, the longitudinal gauge factor at room temperature and the temperature coefficient of resistance have been measured. The results are compared with microscope images showing cracks in distinct cases. Finally, the compatibility and thus applicability of the ESL 3414-A on the investigated substrates is evaluated.

Key words: *piezo-resistive thick-film paste, gauge factor, low temperature co-fired ceramics, force sensor*

1 Introduction

In order to carry out studies about the fingering behavior of musicians who perform on woodwind instruments, a special force sensor system has to be incorporated in the instrument's body. Such instruments possess a variety of different shaped keys and the space for sensor integration is restricted. A technology is required allowing the fabrication of this complex sensor system together with customized, space limited packages including signal processing. LTCC (low temperature co-fired ceramics) technology has been proven as suitable approach for the realization of such sensor systems [1].

The core of the sensor system is formed by miniaturized force sensors whereas beams (lateral dimension: 2×1 mm²) are acting as spring elements. Those beams are to be manufactured in LTCC technology or post-fired HTCC substrates. On that beams piezo-resistive layers are applied by screen-printing. The thicknesses of the beams depend on the maximum finger force during instrument playing (about 5 N) and the mechanical characteristics of the tape material.

Different kinds of LTCC-tapes and a partially stabilized zirconia (PSZ) tape were tested with regard to their mechanical performance and compatibility with the piezo-resistive layer. The ESL 3414-A piezo-resistive thick-film paste has been chosen because of its high gauge factor (14–15 on alumina following the datasheet) compared to other thick-film pastes. Since the piezo-resistive thick-film paste has especially been designed for alumina (Al_2O_3) substrates, sintered 96% alumina substrates were used as reference.

The sintering of the piezo-resistive thick-film pastes is carried out at around 850 °C. At these temperatures, diffusion processes between substrate, piezo-resistive paste and resistor termination become an issue. These diffusion processes during sintering may influence the performance of the piezo-resistive paste. In order to conclude from influences of the resistor termination on the performance of the piezo-resistor, different termination pastes have been applied on the alumina reference substrates in a first step. Next, the geometry of the piezo-resistors has been varied to distinguish the influence between resistor termination (mainly resistor width) and substrate (total resistor area) on the piezo-resistor.

Using these sample variations, the sheet resistance at room temperature, the longitudinal gauge factor at room temperature (GF) and the temperature coefficient of resistance (TCR) have been measured. The results of the measurements allow a statement about the applicability of the ESL 3414-A piezo-resistive paste on different kinds of LTCC and HTCC substrates.

2 Sample Preparation

The termination paste variations were carried out with following pastes: ESL 8836 (Au) pre-fired (termination and piezo-resistor fired consecutively), ESL 9695-G (Ag/Pd), HERAEUS RP 26001/59 (Au-resinate) pre-fired, and HERAEUS TC 7305 A (Ag) pre-fired and co-fired (termination and piezo-resistor fired concurrently). In all cases the piezo-resistors were sintered after the termination. An advantage of sintering the piezo-resistor after the termination is that the length of the piezo-resistor is constant over all samples. Also, the piezo-resistors have not to be sintered twice when termination and piezo-resistive paste are sintered consecutively. A disadvantage of that method is that the thickness of the piezo-resistors depends on the thickness of the terminations and the distance between terminations due to the screen printing process.

The piezo-resistors have been printed with a length of 10 mm and a width of 1 mm to make differences in termination thickness negligible. Five samples were printed and measured each and the mean value of resistance was used for comparison. With an average film thickness of 13 µm, the sheet resistances of the piezo-resistors with different termination pastes are summarized in Tab. 1. All measurements were carried out at (23 ± 1) °C and (30 ± 5) % relative humidity.

Tab. 1. Sheet resistance of piezo-resistor paste ESL 3414-A with varying termination paste on 96% alumina substrates.

Termination paste	Sheet resistance in kΩ/☐
ESL 8836	6.7 ± 0.4
ESL 9695-G	6.1 ± 0.2
Heraeus RP 26001/59	6.9 ± 0.3
Heraeus TC 7305 A pre-fired	6.1 ± 0.2
Heraeus TC 7305 A co-fired	6.1 ± 0.2

The ESL 3414-A piezo-resistive thick-film paste has a sheet resistance of 8 kΩ/☐ ± 10 % at a film thickness of 11 µm. Because of the fact that the measured and scaled (13 µm/11 µm) resistances lied within this tolerance, no essential difference in piezo-resistor resistance among varying termination pastes could be observed. Anyway, for further investigations on different kinds of LTCC substrates, the ESL 8836 and ESL 9695-G are incompatible with LTCC due to substrate warping during co-firing. The Heraeus RP 26001/59 is also incompatible with LTCC because it is a resinate paste and thus dissolves in the glassy phase. The Heraeus TC 7305 A showed fewest warping on any of the considered LTCC tapes and therefore was selected for all subsequent experiments.

In the second step, the piezo-resistors where screen-printed with the geometries given in Fig. 1 (length×width): 1×1 mm² (R_1), 2×2 mm² (R_2), 2×1 mm² (R_3) and 1×2 mm² (R_4). Following substrates were used: HERAEUS AHT01-005, HERAEUS AHT08-047, HERAEUS CT707, CERAMTEC GC (all LTCC, as tape), ESL 42013-A (PSZ, HTCC, as tape) and 96% alumina (HTCC, purchased in fired state) as reference.

Since sintering involves tape shrinkage, this shrinkage had to be taken into account. Following combinations have been manufactured in all LTCC tapes:

- **Post-fired (A):** the tapes were scaled-up for cutting with respect to their shrinkage to approximately achieve equal-sized ceramic substrates after sintering. Afterwards, the resistor structures where printed and fired. This way was also used for the PSZ tape.
- **Co-fired, Pre-laminated (B):** the tapes were cut unscaled. Before printing of the resistor structures, the single tapes were laminated. Shrinkage has not been compensated.
- **Co-fired, Co-laminated (C):** the tapes were cut unscaled. After printing the resistor structures, the single tapes were laminated together with the termination/piezo-resistive pastes on top. Shrinkage has not been compensated.

The tapes were cut with a 120W Nd:YAG-laser, laminated at 7 MPa at 70 °C for 3 min. using an uniaxial press and sintered at peak temperatures of 850 °C (HERAEUS tapes, dwell time 28 min.), 870 °C (CERAMTEC GC tape, dwell time 20 min.) and 1450 °C (ESL 42013-A tape, dwell time 3 h) respectively. The dimensions of the sintered tapes of variant A and the unsintered tapes of variant B/C were 50 mm in length and 5 mm in width. The number of tapes and thus the substrate thickness was selected as a trade-off between effort and the prevention of warping during sintering. The sintered thicknesses are listed in detail in section 4.

To reduce statistical dispersion three samples of each R_1–R_4, every substrate and variation A–C have been manufactured and measured.

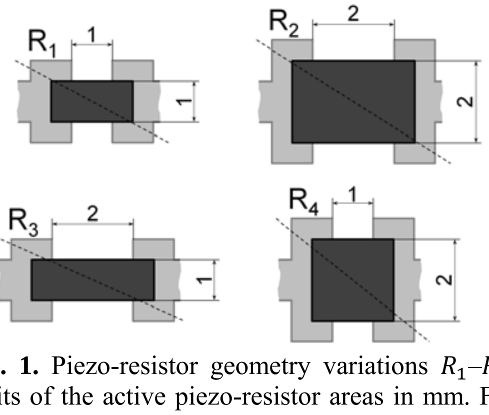

Fig. 1. Piezo-resistor geometry variations R_1–R_4. Units of the active piezo-resistor areas in mm. For the dashed lines see text.

3 Sheet Resistance

The measurement results of the sheet resistance of variation A–C are shown in Fig. 2, 3 and 4, respectively. Comparing the sheet resistances of R_1 to R_4 for a specific substrate, there could be observed a deviation of a factor of up to 3.2. This deviation can be explained by two reasons: diffusion processes between substrate and piezo-resistor and/or thickness variations of the screen-printed and fired piezo-resistors. The thicknesses of R_1–R_4 have been exemplarily measured on an alumina substrate sample (most planar substrate) using an optical profilometer (FRT MICROPROF with x–y-resolution of 1 µm, z-resolution of 20 nm). The thicknesses of the piezo-resistive layers were measured following the dashed lines in Fig. 1. The zoomed and x-scaled thicknesses are given in Fig. 5. The sheet resistance is inversely proportional to the layer's thickness. So, the ratios of $R_i\square/R_1\square \quad (i = 2,3,4)$ were compared with the ratios of thicknesses t_1/t_i. With this comparison a deviation of less than ± 4 % was calculated. This shows that the deviation between R_1–R_4 does not depend on different piezo-resistor areas.

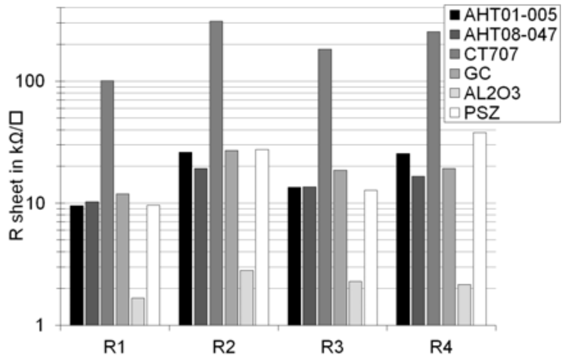

Fig. 2. Sheet resistance of post-fired (var. A) piezo-resistors R_1–R_4 on different substrates.

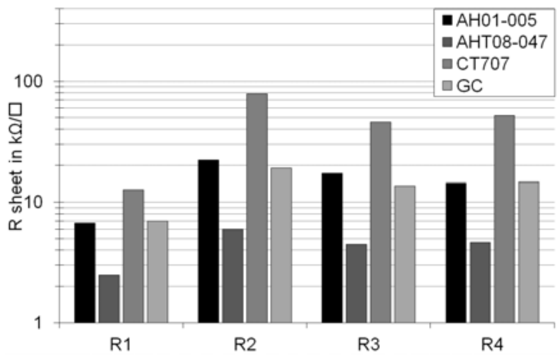

Fig. 3. Sheet resistance of co-fired, pre-laminated (var. B) piezo-resistors R_1–R_4 on different substrates.

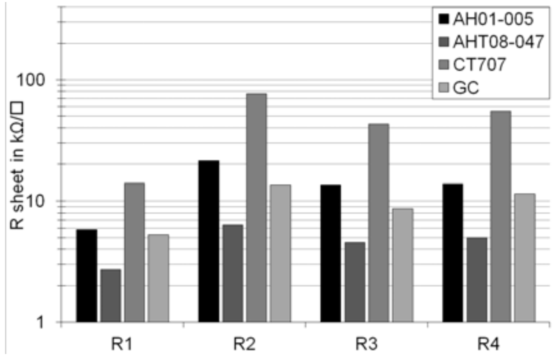

Fig. 4. Sheet resistance of co-fired, co-laminated (var. C) piezo-resistors R_1–R_4 on different substrates.

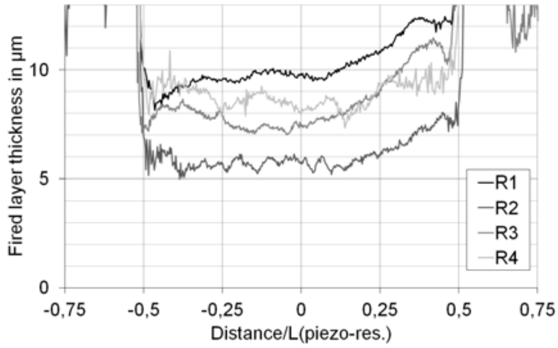

Fig. 5. Piezo-resistive layer thickness of R_1–R_4 on an alumina-substrate. The distances are related to the total length L of the piezo-resistive layer to allow comparison.

Comparing the sheet resistance of co-fired samples, no difference between pre-laminated (var. B, Fig. 3) and co-laminated (var. C, Fig. 4) samples could be observed.

The deviation of one particular resistor geometry on different substrates lies at a factor of up to 110 (e. g. R_2 on CT707 compared to R_2 on alumina). Therefore, the substrate has influence on the sheet resistance of the entire piezo-resistor volume, not just the substrate-resistor-interface. It can also be seen from Figs. 2 to 4 that the sheet resistance on different substrates deviates less from the alumina reference on co-fired (var. B and C) samples than on post-fired samples. In all cases the sheet resistance is higher than on the reference substrate. Two reasons may account on this phenomenon: micro cracks resulting from sintering or massive diffusion/phase changes into/in the piezo-resistor. Cracks are presented in section 4 below. [2, 3] did investigate, that no phase changes could be observed when sintering ESL 3414-B piezo-resistors on different substrates. However, it was not shown if the substrate has influence on the grain size or grain distribution. Due to the higher resistance values on post-fired substrates it is likely that the glassy phase of LTCC substrates has influence on the sheet resistance: the glassy phase is more present on the surface of post-fired substrates than of co-fired. For CT707, the high amount of SiO_2-glass may be responsible for the highest deviation of all investigated LTCC tapes. But this still has to be analyzed in detail.

4 Gauge Factor

First, the flexural strength σ_{break} of the post-fired substrates without piezo-resistors was determined. To get repeatable results, these measurements have been carried out following the ASTM C 1161-02c:2003 and EN 843-1:2008 standards [4, 5] for 3-point-testing of flexural strength of ceramics at ambient temperature. The flexural strength for 3 point bending is:

$$\sigma_{break} = \frac{3F_{break}l}{2wh^2},$$

where F_{break} is the force at break, l the length of the support span, w and h the width and height of the substrate, respectively (see Fig. 6). An H&P INSPECT MICRO with 10 N and 100 N force sensors has been used for measuring, fulfilling all prerequisites of the standards. Standard configuration B with *as-fabricated* was used with the restriction that the samples were thinner than required. This is due to efforts when manufacturing laminates. However, it was assumed that the substrates are isotropic, homogeneous and linearly elastic with average grain size less than 1/50 of the substrate's thickness.

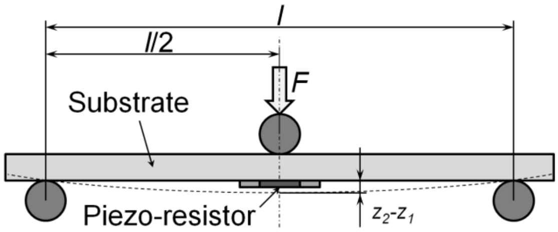

Fig. 6. Principle of 3 point bending test to measure $\boldsymbol{\sigma_{break}}$ and gauge factor.

The measurement results are summarized in Tab. 2. These results are mean values of five samples each. The value of the ESL 42013-A (PSZ) substrate are by far lower than expected for zirconia because perforations were laser-machined at the edges to prevent warping during sintering.

Tab. 2. Break force and flexural strength of post-fired substrates without piezo-resistors.

Substrate	F_{break} in N	σ_{break} in MPa
Al_2O_3	10,81	342
ESL 42013-A	11,94	413 (see text)
HERAEUS AHT01-005	3,94	267
HERAEUS AHT08-047	5,28	331
HERAEUS CT707	4,22	267
CERAMTEC GC	3,03	319

Second, the gauge factor *GF* has been measured. Therefore, the substrates with piezo-resistors on the bottom side (expansion) were loaded with 10 % (F_1) and 90 % (F_2) of F_{max}, where F_{max} was selected to be half of F_{break}. The corresponding strains ε_1 and ε_2 and resistances R' (resistance at F_1) and R'' (resistance at F_2) were used to obtain the gauge factor [6]:

$$GF = \frac{\Delta R/R}{\Delta\varepsilon} = \frac{(R''-R')l^2}{R'[6h(z_2-z_1)]}$$

with z_i being the maximum displacement of the substrate at F_i. The load has been removed afterwards to exclude possible hysteresis. Then, the measurement was repeated five times, and the mean value of the last three measurements was taken to calculate the gauge factor. Fig. 7 presents the calculated gauge factors of the post-fired alumina, HERAEUS CT707 and ESL 42013-A substrates. The gauge factor for the remaining LTCC tapes, HERAEUS AHT01-005 and AHT08-047 and CERAMTEC GC, are not shown in the diagram since their calculated gauge factor was in 70 % of the post-fired cases greater than 80. The repeated measuring method did show, that the resistances R' and R'' did not stay constant; their value always increased with number of measurement cycles. This can be interpreted as sign, that the substrate or piezo-resistor tends to crack when loaded. Since the force and displacement did stay constant for all substrates over all measurement cycles, there is no evidence of crack formation in the substrate, that is, the piezo-resistor itself is to be damaged when loaded.

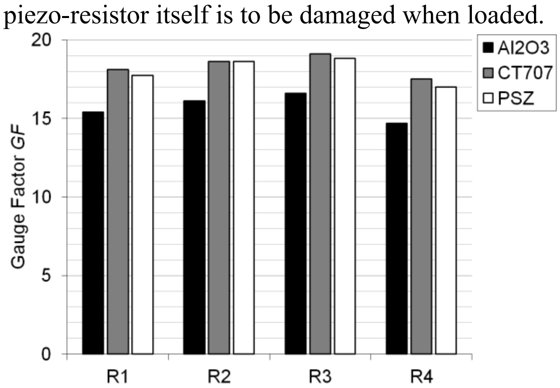

Fig. 7. Gauge factor of post-fired (var. A) piezo-resistors. For the omitted substrates see text.

The gauge factor of post-fired R_2 and R_3 was in all cases higher than of R_1 and R_4. The film thickness of R_2 and R_3 is lower than of R_1 and R_4 (Fig. 5) and therefore more volume of the piezo-resistor is exposed directly to the bending of the substrate.

Microscope images of fired piezo-resistors on post-fired substrates before bending are shown in Fig. 8 to Fig. 13. As expected, no cracks could be observed on the alumina and zirconia substrates (Figs. 8, 9). The piezo-resistors of the HERAEUS AHT01-005, AHT08-047 and the CERAMTEC GC tapes did show cracks spreaded over the entire piezo-surface (Figs. 10, 11, 13). The piezo-resistors on the HERAEUS CT707 did show cracks only at the intersection with the termination (Fig. 12).

The gauge factors of the co-fired piezo-resistors (var. B as well as var. C) did show damage in all cases. 30 % of the HERAEUS CT707 samples, 90 % of the HERAEUS AHT01-005 and CERAMTEC GC samples, and 100 % of the Heraeus AHT08-047 samples were damaged. All of these substrates did show cracks on the entire piezo-surface.

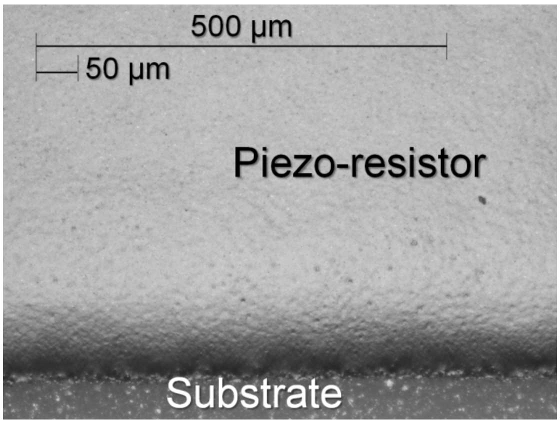

Fig. 8. Piezo-resistor ESL 3414-A on post-fired Al_2O_3 substrate.

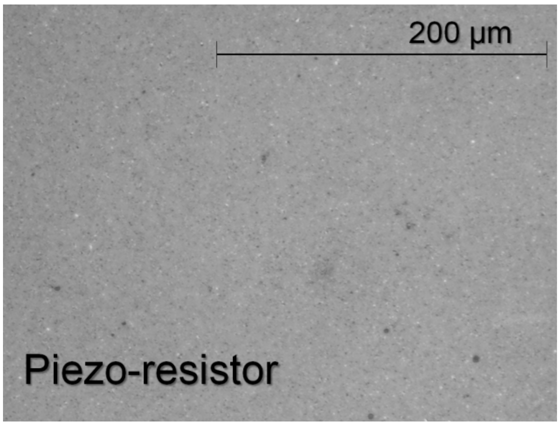

Fig. 9. Piezo-resistor ESL 3414-A on post-fired ESL 42013-A (PSZ) substrate.

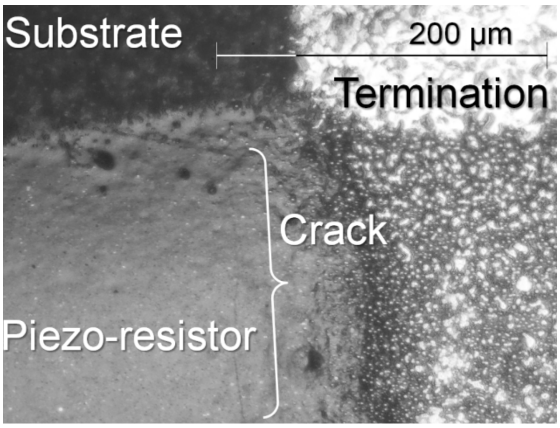

Fig. 10. Piezo-resistor ESL 3414-A on post-fired HERAEUS AHT01-005 substrate.

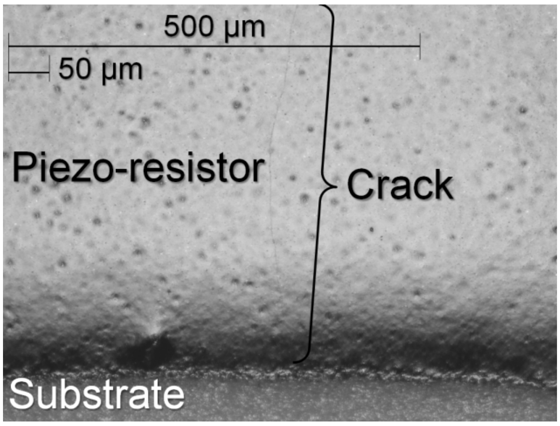

Fig. 11. Piezo-resistor ESL 3414-A on post-fired HERAEUS AHT08-047 substrate.

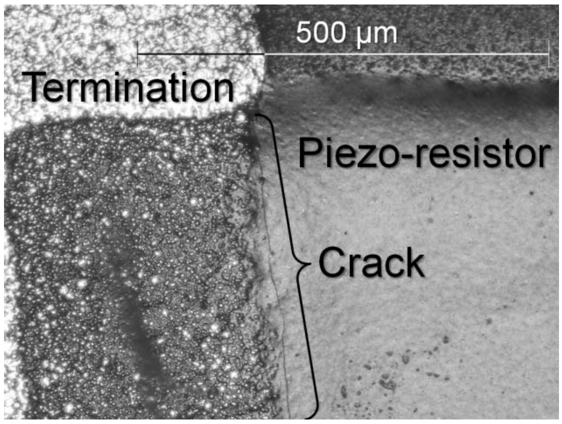

Fig. 12. Piezo-resistor ESL 3414-A on post-fired CT707 substrate.

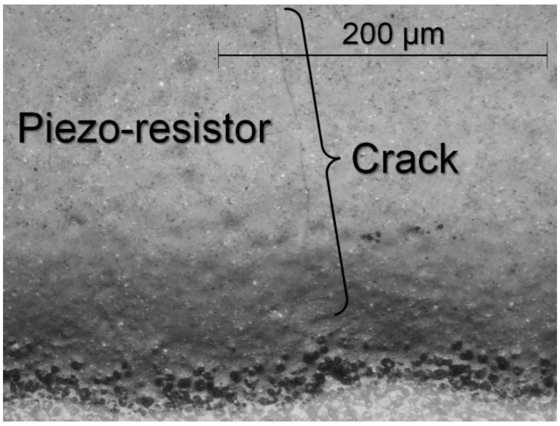

Fig. 13. Piezo-resistor ESL 3414-A on post-fired CERAMTEC GC substrate.

5 Temperature Coefficient of Resistance

The temperature coefficient of resistance (*TCR*) of the post-fired (var. A) samples has been measured after loading in the range of +30 °C to +125 °C, see Tab. 3. All LTCC substrates show a relative high *TCR* compared to the alumina reference substrate. The HERAEUS AHT08-047 as well as the CERAMTEC GC have high negative TCRs of about -1100 ppm/K. The *TCR* of the HERAEUS CT707 is lower but also about -531 ppm/K.

Tab. 3. Temperature coefficient of resistance of the piezo-resistors depending on substrate.

Substrate	TCR in ppm/K
Al2O3	+184 … +246
ESL 42013-A	-32 … +152
HERAEUS AHT01-005	-317 … -134
HERAEUS AHT08-047	-1 052 … -471
HERAEUS CT707	-531 … -360
CERAMTEC GC	-1 100 … -600

6 Comparison and Interpretation

Since all co-fired samples did show cracks. These cracks existed before bending and did increase during bending tests. Therefore, the ESL 3414-A piezo-resistive paste is incompatible when co-fired on any of the investigated LTCC tapes due to crack formation on the piezo-resistive layer. Crack formation was also omnipresent on the piezo-resistors screen-printed on the post-fired HERAEUS AHT01-005, HERAEUS AHT08-047 and CERAMTEC GC tapes. Only the HTCC substrates (alumina and PSZ) and the HERAEUS CT707 substrate possessed constant gauge factors. The CT707 did also show cracks at the intersection with the termination. Those cracks did not influence the gauge factor since they are located at the very end of the piezo-resistor. However, those cracks are responsible for the increased negative temperature coefficient of resistance. If the coefficient of thermal expansion of the piezo-resistive paste is higher than of the CT707 substrate, then the cracks are compressed during heating and thus the resistance decreases. This would also explain the high negative *TCR* of the remaining cracked LTCC substrates.

7 Conclusion

Within this paper the ESL 3414-A piezo-resistive thick-film paste has been applied on different kinds of HTCC and LTCC substrates under varying manufacturing conditions. It emerged that this paste was only compatible with the HTCC substrates 96% alumina (reference) and ESL 42013-A. On the post-fired HERAEUS CT707 a repeatable gauge factor could be measured but the piezo-resistive paste showed cracks at its intersection with the termination. This yields in an increased *TCR* of the piezo-resistor. Continuous loading of that substrate has to be carried out to characterize the long-term behavior of that cracks. It emerged that the ESL 3414-A paste is incompatible with the HERAEUS AHT01-005, HERAEUS AHT08-047 and CERAMTEC GC substrates, post-fired as well as co-fired.

Acknowledgement

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