Informacije MIDEM Journal of Microelectronics, Electronic Components and Materials

Vol. 44, No. 1 (2014), 4 - 11

Characterisation of thick-film resistors as gauge sensors on different LTCC substrates

Marko Hrovat^{1,2}, Darko Belavič^{1,2,3}, Kostja Makarovič^{1,2}, Jena Cilenšek^{1,2}, Barbara Malič^{1,2}

¹ Jožef Stefan Institute, Ljubljana, Slovenia ² CoE NAMASTE, Ljubljana, Slovenia ³ HIPOT-RR d.o.o., Otočec, Slovenia

Abstract: A thick-film resistor can sense mechanical deformations in C-MEMS structures. The relatively low elastic moduli of LTCC ceramics, compared to alumina ceramics, would suggest an increased sensitivity of the sensing elements. Selected thick-film resistors (Du Pont 2041 and ESL 3414) were evaluated as piezo-resistors for force sensors. The resistors were screen printed and fired on various LTCC tapes (Du Pont 951, ESL 41020, Heraeus HL 2000, Ferro L8 and Heraeus CT 702) as well as on alumina substrates. The LTCC tapes were analysed by scanning electron microscopy, energy-dispersive X-ray analysis and X-ray powder analysis. The crystalline phases in the LTCC materials were determined. E electrical characteristics, i.e., sheet resistivities, noise indices and gauge factors of the thick film resistors, were measured. While there were variations in the other electrical characteristics of the resistors fired on different substrates, the gauge factors were rather independent of the substrate materials

This work is dedicated to the late Professor Marija Kosec

Keywords: thick film resistors, LTCC, X-Ray diffraction, micro-structures

Karakterizacija debeloplatnih uporov kot senzorjev sile na različnih LTCC substratih

Izvleček: Debeloplastni upori lahko služijo kot senzorji sile / deformacije na ali v večplastnih MEMS strukturah. Razmeroma nizki moduli elastičnost LTCC (Low temperature co-fired ceramics – keramika z nizko temperature žganja), če jih primerjamo s keramiko na osnovi Al2O3, vodijo do večjih občutljivosti senzorjev pri istih dimenzijah. Dva debeloplastna uporovna materiala – Du Pont 2041 in Electro Science Labs 3414 – sta bila tiskana in žgana na relativno inertnih Al2O3 in različnih LTCC podlagah. Testirani LTCC materiali so bili Du Pont 951, ESL 41020, Heraeus HL 2000, Ferro L8 in Heraeus CT 702. Z izjemo Du Pontovega 951 so vsi ti materiali brez svinca, medtem ko steklena faza v 951 vsebuje okrog 2 mol.% PbO. LTCC folije so bile analizirane z elektronskim vrstičnim mikroskopom, mikro anaslizo in rentgensko analizo. Določili smo faze, ki izkristalizirajo med žganjem. Izmerili smo električne karakteristike debeloplastnih uporov na različnih podlagah. Medtem, ko so plastne upornosti, temperaturne odvisnosti upornosti in indeksi šuma odvisni od podlag, na katerih so bili žgani upori, je faktor gauge – sprememba upornosti v odvisnosti od deformacije – v glavnem neodvisna od podlag.

Ključne besede: debeloplastni upori, LTCC rentgenska praškovna analiza, mikrostrukture

* Corresponding Author's e-mail: marko.hrovat@ijs.si

1 Introduction

Fired thick-film resistors basically consist of a conducting phase in an insulating matrix, usually a silica-rich, lead-borosilicate-based glass. The ratio between the conductive and the glass phases roughly determines the specific resistivity of the resistor. During the firing cycle the conductive phases of the resistor materials interact with the glass phase forming conductive networks through the sintered layers. In most modern thick-film resistor compositions the conductive phase is either ruthenium oxide or electrically conducting pyrochlores: mainly lead or bismuth ruthenates [1-3] with resistivities of 40 x 10⁻⁶, 150 x 10⁻⁶ and 270 x 10⁻⁶ ohm.cm for $RuO_{2'}$ Bi₂Ru₂O₇ and Pb₂Ru₂O_{6.5'}, receptively [2-4]. During the firing cycle the conductive phases of the resistor materials interact with the glass phase. The resistors are fired for only a short time at the highest temperature, typically 10 minutes at 850°C.

Advanced microelectronic packages – ceramic modules – with a high density of interconnections and in-

tegrated electronic components are, in many cases, realised by Low Temperature Co-Fired Ceramics (LTCC) technology, which is considered as one of the more advanced technologies for the fabrication of these packages. LTCC materials are sintered at low temperatures around 850°C or 900°C. In order to sinter to a dense and non-porous structure at these, rather low, temperatures, LTCC materials contain some (or a great deal of) low-melting-point glass phase. LTCCs are mainly based on a mixture of crystallisable glass and ceramics: in most cases alumina [5-8].

Sensors for mechanical quantities are fundamental parts of MEMS, and screen printed and fired thick-film resistors can be used to sense the mechanical deformations in MEMS structures. The important characteristic of LTCC materials is a relatively low elastic modulus, which means an increased sensitivity of the sensing elements due to the larger deformation under same conditions as compared to alumina substrates. This is shown in Fig. 1. – calculated deflections of diaphragms with the same radii and thicknesses made with the alumina and the LTCC at an applied pressure of 100 kPa.



Figure 1: The deflections of diaphragms made with the alumina and the LTCC at an applied pressure of 100 kPa.

The change in the 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 [9]. The gauge factor (GF) of a resistor is defined as the ratio of the relative change in the resistance (Δ R/R) and the strain (Δ I/I):

$$GF = (\Delta R/R) / (\Delta I/I)$$
(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 of the specific conductivity. The GFs of thick-film resistors are mostly between 3 and 15.

Thick-film resistors are often integrated on the top of, or within, multilayer LTCC structures. However, most thick-film resistor materials are developed for firing on relatively inert alumina substrates. Therefore, the compatibility and interactions with the rather glassy LTCC substrates, leading to changes in the electrical characteristics, need to be evaluated [10-12]. Another factor could be the difference in the shrinking rates of resistors and LTCC tapes, if they are fired together on the surface or buried within green LTCC structures. This is shown in Figs. 2.a. and 2b. - sintering curves of the "normal" Du Pont resistor 5093 and the Du Pont LTCC 951, and the microstructure of the buried resistor after firing. In Figs. 3.a and 3.b the sintering curves and the microstructure of a buried resistor Du Pont CF-041 that was developed for compatibility with LTCC materials are shown [13]. The "normal" thick-film resistors start to shrink at temperatures around 100K to 150K lower than the LTCC materials, while in the case of resistors that are compatible with LTCC, both materials start to shrink at similar temperatures, i.e., at 650°C







Figure 2: a. Sintering curves of Du Pont thick film resistor 9053 and Du Pont LTCC 951; b. Thick-film resistor (Du Pont 5093) buried within LTCC (Du Pont 951) structure. Different sintering rates lead to deformation.

For strain sensors, two thick-film resistors, i.e., 2041 (Du Pont, nominal resistivity 10 kohm/sq.) and 3414-B (Electro Science Labs. nominal resistivity 10 kohm/sq.)





b.

Figure 3: a. Sintering curves of Du Pont thick film resistor CF-041 and Du Pont LTCC 951; b. Thick-film resistor (Du Pont CF-041) buried within LTCC (Du Pont 951) structure. Sintering rates of resistor and LTCC materials are comparable and the structure is not deformed.

were evaluated. The 2041 resistor was chosen because of its low noise [14], whereas the 3414-B was developed as a material with a high gauge factor, especially for use in strain gauges [15]. The resistors were screen printed and fired on five LTCC tapes, i.e., Du Pont 951, ESL 41020, Heraeus HL 2000, Ferro L8 and Heraeus CT 702. The resistors were also fired on rather inert alumina substrates.

As mentioned above, the electric characteristics of the resistors could deteriorate. Therefore, the main purpose of the paper will be an evaluation of the electrical characteristics of the resistors fired on alumina and different LTCC substrates. The sheet resistivities, noise indices, temperature coefficients of resistivities (TCRs) and gauge factors were measured.

LTCC DP 951, which contains between 2 and 3 mol. % of PbO is, at least from the data in the open literature, the "workhorse" in electronic packaging. The HL-2000 is a well-known "zero shrinkage" material. "Zero shrinkage" does not mean that there is no shrinkage during firing. However, while "ordinary" LTCC tapes shrink in longitudinal and vertical directions, the HL-2000 shrinks only in "z" direction, while the "x" and "y" dimensions stay more or less the same.

The important "electrical" characteristics for all the evaluated LTCC materials (taken mainly from data sheets) are rather similar as, after firing, all the LTCC materials consist of a glass phase, a ceramic filler and a phase or phases that crystallise during firing. To summarise; the dielectric constants are between 7 and 8, the insulation resistivities are between 10¹² and 10¹³ ohm.cm and the break-down voltages are between 1000 V/25 um and 1500 V/um. These very similar reported results are reasonable, as more or less all LTCC materials are based on a mixture of glass and ceramic phase.

2 Experimental

The LTCC substrates were made by laminating three layers of LTCC tape at 70°C and a pressure of 20 MPa. The laminated green tapes were fired with the firing profiles required by the data sheets. The maximum firing temperatures were between 850°C and 900°C. The thick-film resistors printed on LTCC and alumina substrates were terminated with Pd/Ag conductors and fired at 850°C. The dimensions of the resistors for microstructural analyses and X-ray diffraction (XRD) analyses, which were printed and fired without conductor terminations, were 12.5 × 12.5 mm².

The samples - green and fired LTCC tapes - were investigated by X-ray powder diffraction analyses (XRD) with a Philips PW 1710 X-ray diffractometer using Cu Kα radiation. A JEOL JSM 5800 scanning electron microscope (SEM) equipped with an energy-dispersive Xray analyser (EDS) was used for the overall microstructural and compositional analyses of the LTCC samples and the cross-sections of the resistors fired on different substrates. Prior to an analysis in the SEM, the samples were coated with carbon to provide electrical conductivity and to avoid any charging effects. Note that the boron oxide, which is also present in the glass phase of LTCC tapes, 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 the EDS analysis in the glass matrix.

The sheet resistivities were measured. The current noise indices were measured in dB on 100-mW loaded resistors using the Quan Tech method (Quan Tech Model 315-C). TCRs were calculated from resistivities measured between 25°C and 125°C. The changes of resistivity as a function of substrate deformation (gauge factors) were measured with a simple device [16,17]. The ceramic substrate is supported on both sides. The load is applied to the middle of the substrate with a micrometer and this induces a tensile strain in the resistor. The magnitude of the strain is given by equation (2) [18]:

$$\varepsilon = \Delta I / I = (d * t * 6) / L^2$$
 (2)

d = deflection (m) t = substrate thickness (m) L = distance between support edges (m)

The gauge factors are calculated using equations (1) and (2) from the strain and resistivity changes. The test structures with resistors (the bending test) are shown in Fig. 4. For the measuring set-up used to obtain the results reported later, the distance between the support edges was 40 mm.



Figure 4: Test structure for resistivity vs. deformation evaluations. Five black squares are thick film resistors with dimensions 1.6x1.6 mm².

3 Results and discussion

3.1 LTCC materials

The X-ray spectra of the green and fired LTCC tapes Du Pont 951, ESL 41020, Heraeus HL-2000, Ferro L8 and Heraeus CT 702 are shown in Figs. 5.a, 5.b, 5.c, 5.d and 5.e, respectively. The unfired tapes are a mixture of glass and crystalline phase. In all the tapes the main crystalline phase is the alumina. The Heraeus CT 702 tape also contains a small amount of anatase, and the Ferro L8 tape small amounts of the anatase and the quartz. After the firing the peaks of other phases that crystallized from the glass phase can be observed. The main phases are the anorthite (calcium or barium alumo silicates) and the celsian (barium alumo silicate). The crystalline phases in unfired and fired LTCC tapes are summarized in Table 1.

The EDS analyses of the LTCC tapes are presented in Table 2. All the LTCC materials, with the exception of Heraeus HL-2000, also contain Co_2O_3 , which is added tp produce a blue colour in the fired tapes. As the concentrations of cobalt oxide are below 0.5 mol.% they



Figure 5: a. X-ray spectra of Du Pont 951 LTCC before and after firing. The peaks of alumina and anorthite are denoted "A" and "An", respectively; b. - X-ray spectra of ESL 41020 LTCC before and after firing. Unidentified peak is marked by "?"; c. X-ray spectra of Heraeus HL-2000 LTCC before and after firing. Unidentified peak is marked by "?";



Figure 5: d. X-ray spectra of Ferro L8 LTCC before and after firing. Al_2O_3 is denoted "A", TiO₂ "T", calcium alumo silicate "C", BaTiO₃ "B", quartz "S" and celsian (barium alumo silicate) "C"; e. X-ray spectra of Heraeus CT-702 LTCC before and after firing. Al_2O_3 is denoted "A", TiO₂ "T" and barium alumo silicate "B".

are not included in the Table. Most are lead-free, while the Du Pont 951 contains 2 mol.% (around 8 wt.%) of lead oxide. The main oxides are alumina (from 35 to 65 mol,%) and silica (from 25 to 40 mol.%). Heraeus HL-2000, Ferro L8 and Heraeus 702 also contain a small amount of titania, presumably to "assist" in the glass crystallization during firing. Table 2: Composition of LTCC materials (mol.%)

	DP 951	ESL 41020	Her. 2000	Ferro L8	Her. 702
1/2 Na2O	2				
MgO					4
1/2 Al2O3	53	39	52	65	34
SiO2	36	39	31	26	40
1/2 K2O			3		
CaO	4	12	8	1	4
TiO2			3	1	3
ZnO		2			5
BaO		8	3	7	7
PbO	2				

3.2 Thick-film resistors

Two typical microstructures of thick-film resistors fired on LTCC substrates are shown in Figs. 6.a (2041 on ESL 41020 LTCC) and Fig. 6.b (3414 on ESL 41020 LTCC). The resistor layers are on top of the LTCC substrates. The light grains are the conductive phase and the darker matrix is a glass phase. In both cases the light phase at the resistor/LTCC interface is rich in the PbO, which diffused from the resistor layer into the LTCC substrate during firing. However, this "reacted" layer is thicker in the case of the 3414-B resistor, which means more extensive interactions and therefore a more significant influence on the resistors' characteristics.

The electrical characteristics of the thick-film resistors on alumina and different LTCC substrates, i.e., sheet resistivities, TCRs, noise indices and gauge factors of 2041 and 3414 resistors are summarized in Table 3. The sheet resistivities, noise indices and gauge factors are graphically presented in Figs. 7, 8 and 9, respectively. Note that the noise indices in Table 3 are given in (dB), while in Figure 8 they are presented in (uV/V). These two units are related with a simple equation:

	$= 20 \log \text{ noise } (\text{uV/V}) \tag{3}$	noise $(dB) = 20$
--	--------------------------------------------------	-------------------

The sheet resistivities of the 3414 resistors are more "dependent" on the types of LTCC substrates than the resistivities of the 2041 resistors. I all cases sheet resistivities of resistors on LTCC substrates are higher, some-

Table 1: Evaluated LTCC materials and XRD analyses of crystalline phases in unfired and fired tapes.

	LTCC	Unfired tapes	Fired tapes	
Du Pont	DP 951	Alumina	Alumina, anorthite	
ESL	41020	Alumina	Alumina, celsian, gahnite	
Heraeus	HL-2000	Alumina	Alumina, anorthite	
Ferro	L8	Alumina, quartz, anatase	Alumina, quartz, anatase, celsian	
Heraeus	702	Alumina, anatase	Alumina, anatase, anorthite	







Figure 6: Microstructure of 2041 thick film resistor fired on ESL 41020 LTCC; b. Microstructure of 3414 thick film resistor fired on ESL 41020 LTCC

times significantly higher. The presumed reason for this is the diffusion of a glass phase from LTCC substrates into resistor films during firing resulting in a dilution of conductive phase in resistors. Also, the noise indices of the 3414 are significantly higher. However, the values of the gauge factors are for both resistors more or less independent of the substrate materials. The gauge factors of the 3414 thick-film resistors were higher (between 15 and 19) than the gauge factors of the 2041

20 18 16 14 R (kohm/sq.) 12 10 8 6 4 2 0 3414 AI2O3 DP951 2041 SL4102 HL2000 F L8 CT702

Figure 7: Sheet resistivities of 2041 and 3414 thick film resistors fired on alumina and LTCC substrates



Figure 8: Noise indices of 2041 and 3414 thick film resistors fired on alumina and LTCC substrates



Figure 9: Gauge factors of 2041 and 3414 thick film resistors fired on alumina and LTCC substrates

Table 3: Electrical characteristics – sheet resistivities, noise indices, TCRs and gauge factors – of the 2041 and 3414 thick-film resistors fired on alumina and different LTCC substrates.

	Resistor 2041				Resistor 3414		
Substrate	R	N.I.	TCR	GF	R	N.I.	GF
	(kohm/sq.)	(dB)	(x10-6/K)		(kohm/sq.)	(dB)	
Al2O3	9.0	-21.0	15	10.5	3.0	-4.2	19.1
DP 951	11.0	-20.0	20	9.8	9.3	5.0	18.8
ESL 41020	15.5	-15.1	55	9.7	14.7	7.1	17.9
HL 2000	15.8	-14.5	50	10.9	19.5	-7.9	17.5
Ferro L8	16.1	-18.7	-40	9.6	17.5	3.2	16.7
CT 702	14.9	-18.4	-15	8.2	6.6	-2.1	11.6

resistors (around 10). These experimental results indicate that both the evaluated thick-film resistors could be used as sensing elements in the LTCC structures if other electrical characteristics are taken into account.

4 Conclusions

Sensors for mechanical quantities are fundamental parts of MEMS, and screen-printed and fired thick-film resistors can be used to sense the mechanical deformations in MEMS structures. The important characteristic of LTCC materials is the relatively low elastic modulus, which means an increased sensitivity of the sensing elements due to the larger deformation under same conditions as compared to alumina substrates. Therefore, thick-film resistors are often integrated on the top of or within multilayer LTCC structures. However, as most of the thick-film resistor materials are developed for firing on relatively inert alumina substrates the compatibility and interactions with the rather glassy LTCC substrates, leading to changes in the electrical characteristics, need to be evaluated.

For strain sensors two thick-film resistors with nominal resistivity of 10 kohm/sq., Du Pont 2041 and ESL 3414-B, were tested. The resistors were screen printed and fired on five LTCC tapes, i.e., Du Pont 951, ESL 41020, Heraeus HL 2000, Ferro L8 and Heraeus CT 702. The resistors were also fired on rather inert alumina substrates. The LTCC materials were evaluated with XRD and EDS analyses. Results show that the unfired LTCC tapes are based on a mixture of glass phase and ceramic filler. After firing the crystalline phases crystallize from the glass component. The EDS microanalysis indicated that most of the LTCC materials, with the exception of Du Pont 951, are lead-free.

The sheet resistivities, temperature coefficients of resistivity, noise indices and gauge factors of the resistors processed on different substrates were measured. While there are variations in the other electrical characteristics of the resistors fired on different substrates, the gauge factors for both resistors are rather independent of the substrate materials.

5 Acknowledgment

The authors wish to thank Mr. Mitja Jerlah (IN.Medica) for fabricating the samples and measuring the electrical characteristics.

The financial support of the Slovenian Research Agency is gratefully acknowledged. Part of the research work was also included in the Centre of Excellence NA- MASTE, which is financially supported by the European Union (European Regional Development Fund), and the Ministry of Higher Education, Science and Technology)

6 References

- 1. J. W. Pierce, D. W. Kuty, J. L. Larry, The chemistry and stability of ruthenium based resistors, Solid State Technol., <u>25</u>, (10), (1982), 85-93
- 2. R. W. Vest, Materials science of thick-film technology, Ceram. Bull., <u>65</u>, (4), (1986), 631-636
- M. Hrovat, J. Holc, D. Belavič. J. Bernard, Subsolidus phase equilibria in the PbO poor part of RuO₂-PbO-SiO₂ system, Materials Letters, <u>60</u>, (20), (2006), 2501-2503
- 4. P. R. van Loan, Conductive ternary oxides of ruthenium, and their use in thick film resistor glazes, Ceram. Bull., <u>51</u>, (3), (1972), 231-233, 242
- W. K. Jones, Y. Liu, B. Larsen, P. Wang, M. Zampino, "Chemical, structural and mechanical properties of the LTCC tapes", Proc. 2000 Int. Symp. on Microelectronics IMAPS-2000, Boston, pp. 669-674, 2002.
- Y. Imanaka, Multilayer low temperature cofired ceramic (LTCC) technology, Springer Science + Business Media, Inc., 2005, 1-56
- C. J. D. Kumar, E. K. Sunny, N. Ranghu, N. Venkataramani, A. R. Kulkarni, Synthesis and characterization of crystallizable amortize-based glass for a low-temperature cofired ceramic applications, J. Am. Ceram. Soc., <u>91</u>, (2), (2008), 652-655.
- R. Muller, R. Meszaros, B. Peplinski, S. Reinch, M. Eberstein, W. A. Schiller, J. Deubener, Dissolution of alumina, sintering, and crystallization in glass ceramic composites for LTCC, J. Am. Ceram. Soc., <u>92</u>, (8), (2009), 1703-1708
- 9. K. Hoffman, "An introduction to measurements using strain gauges", Hottinger Baldwin Messtechnik GmbH, Darmstadt, 1989
- C. S. His, F. M. Hsieh, H. P, Chen, Characteristics of thick film resistors embedded in low temperature co-fired ceramic (LTCC) substrates, J. Eur. Ceram. Soc., <u>27</u>, (7), (2007), 2779-2784
- 11. M. Lahti, A. Vimpari, K. Kautio, Printable resistors in LTCC systems, J. Eur. Ceram. Soc., <u>27</u>, (8,9), (2007), 2953-2956
- M. Hrovat, D. Belacič, M. Santo-Zarnik, M. Jerlah, J. Holc, J. Cilenšek, S. Drnovšek, M. Kosec, Thick film sensing elements on LTCC structures, Informacije MIDEM, <u>41</u>, (4), (2011), 279-283
- M. Hrovat, D. Belavič, J. Kita, J. Holc, J. Cilenšek, L. Golonka, A. Dziedzic, Thick-film PTC thermistors and LTCC structures; the dependence of the elec-

trical and microstructural characteristics on the firing temperature. J. Eur. Ceram. Soc.., <u>27</u>, (2007), 2237-2243

- 14. M. Hrovat, D. Belavič, Z. Samardžija, J. Holc, A characterisation of thick film resistors for strain gauge applications, J. Mater. Sci., <u>36</u>, (2001), 2679-2689.
- S. Chitale, C. Huang, M. Stein, High gauge factor thick film resistors for strain gauges, Hybrid Circuits Technol., <u>6</u>, (5), (1989), 15-18
- 17. M. Hrovat, G. Dražič, J. Holc, D. Belacič. Correlation between microstructure and gauge factors of thick film resistors, J. Mater. Sci. Lett., 14, (1995), 1048-1051
- 16. M. Hrovat, D. Belavič, G. Dražič, J. Holc, S. Šoba. Investigations of thick film resistors with high gauge factors. Informacije. MIDEM, <u>25</u>, (1995), 108-114.
- C. Song, D. V. Kerns, Jr., J. L. Davidson, W. Kang, S. Kerns, "Evaluation and design optimization of piezoresistive gauge factor of thick film resistors", IEEE Proc. SoutheastCon 91 Conf., Vol. 2, Williamsburg, 1991, 1106-1109

Arrived: 09. 09. 2013 Accepted: 05. 02. 2014