

Mechanical Properties of Low Temperature Co-fired Ceramics: Testing Methodologies for Strength Characterization

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Abstract: Functional components such as multilayer Low Temperature Co-fired Ceramics (LTCC) are examples of combination of a ceramic-based substrate with internal electrodes as well as surface features (e.g. metallization, contacting pads, cylindrical vias, etc) employed to provide the component with a given functionality. Due to miniaturization and design complexity, no standard methods for mechanical testing can be applied for component characterization. An experimental approach is here presented using the Ball-on-three-Balls (B3B) method, based on localized biaxial testing of metalized LTCC samples, at several locations under different environments, to quantify the effect of surface features or metallization (e.g. contacting pads or cylindrical vias) on the material strength distribution. Experimental findings show that the strength distribution of LTCC can be negatively affected by environmental degradation, where Subcritical Crack Growth (SCCG) phenomena can be enhanced in conditions of high relative humidity. In addition, metallization at the surface subjected to tensile stresses can even raise the strength of the component acting as a protective layer against environmental degradation, whereas cylindrical vias can become weak points in the design.

Key words: Functional ceramic components, Biaxial Strength, Fracture, Weibull Statistics, Environmental brittleness.

Mehanske lastnosti keramike z nizko temperaturo žganja: metodologije testiranja trdnosti

Povzetek: Funkcionalne sestavine, kot je večplastna keramika z nizko temperaturo žganja (LTCC), so primeri kombinacij substratov na osnovi keramike in internih elektrod, kot tudi površinskih lastnosti (metalizacija, kontaktne blazinice, cilindrične povezave itd.), ki dajejo komponenti željeno funkcionalnost. Standardne metode testiranja mehanskih lastnosti, zaradi miniaturizacije in kompleksnosti oblik, niso primerne za karakterizacijo elementa. V članku je predstavljen je eksperimentalen postopek na osnovi Ball-on-three-Balls (B3B) metode, ki temelji na lokaliziranem dvoosnem testiranju metaliziranih LTCC vzorcev na različnih lokacijah in okoljih in je namenjen kvalifikaciji vpliva površinskih lastnosti ali metalizacije (kontaktne blazinice, cilindrične povezave) na porazdelitev sil v materialu. Rezultati poskusov so pokazali, da ima lahko degradacija zaradi okolja negativen vpliv na razporeditev sil LTCC. Pri visoki relativni vlažnosti se lahko pospeši rast razpok (Subcritical Crack Growth – SCCG). Dodatno lahko površinske natezne sile zaradi metalizacije izboljšajo čvrstost in delujejo kot zaščitni sloj pred vplivi okolja, pri čemer pa cilindrične povezave predstavljajo šibke točke v konstrukciji.

Ključne besede: funkcionalne keramične komponente, dvoosna sila, frakcije, Weibullova statistika, okoljska občutljivost.

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1. Introduction

Many applications in microelectronics involve combinations of ceramic and metal constituents. Due to the different properties of the materials involved (e.g. thermal expansion coefficients, elastic constants, yield strength) components can be subject to internal stresses during fab-

rication, which may induce cracks that truncate the electrical performance of the device [1-4]. Other processes after fabrication (e.g. packaging) can also involve high tensile stresses which may damage the component. In addition, mechanical stresses occurring during service conditions (e.g. rapid temperature changes, mechanical vibration, bending) can affect the structural integrity of the device.

Functional components such as multilayer varistors (MLV), piezoelectric actuators (MPA), Low Temperature Co-fired Ceramics (LTCC) or semiconductors, among others, are examples of combination of a ceramic-based (or Si-based) substrate with internal electrodes as well as surface features (e.g. metallization, contacting pads, cylindrical vias, etc) employed to provide the component with a given functionality. For instance, LTCC consist of a complex three-dimensional micro-network of metal structures embedded within a ceramic substrate with a large content of glass. The low sintering temperature of the ceramics in LTCC (i.e. below 900 °C) can be achieved by using a glass matrix with a low melting point, allowing a vitrification of the glass ceramic composite material [5, 6]. This makes feasible the use of excellent electronic conductors such as silver, gold or mixtures of silver-palladium, arranged within and/or on the surfaces of the ceramic substrate, forming complex multi-layered structures. Today, they can be found in electronic devices (e.g. for mobile and automotive technologies) which have to operate under harsh conditions such as relatively high temperatures and mechanical shock under different environments.

A limiting factor for the lifetime of glass- and ceramic-based components is associated with the subcritical crack growth (SCCG) phenomenon, especially in environments with high moisture content [7-9]. The lifetime is thus related to their mechanical strength and crack growth resistance during service conditions. It is also known that metallization as well as surface features (such as contact pads or metal cavities) can affect the strength of components (see for instance [10-13]). In summary, a combined effect of environmental degradation and geometry features can decrease the strength of brittle functional components.

In this paper, a testing method (the Ball-on-three-balls) is employed to measure the mechanical biaxial strength of commercial and sample LTCCs. Different loading conditions and environments are selected aiming to reproduce possible loading scenarios during service. In addition, an experimental approach combining both humid and relatively dry environments (i.e. water and silicone oil) is presented to assess the effect of different surface metallization on the strength of sample LTCC components.

2. Experimental

2.1. Material of study

The substrate (glass-ceramic) employed to fabricate the LTCC specimens of study is made of approx. 50%

of Al_2O_3 as filler and 50% of several glasses containing Ca, Na, Si, K, B and Al, where the crystallisation degree after sintering exceeds 90%. Figure 1 shows a SEM micrograph of a typical polished area on a LTCC of study. The bright phase corresponds to Al_2O_3 particles which are embedded in a glassy matrix (grey phase). Microstructural characterisation of the bulk material can be found elsewhere [14].

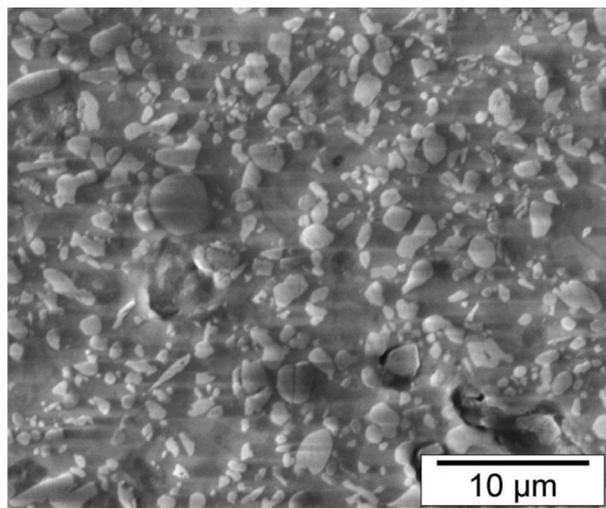


Figure 1: SEM micrograph of a LTCC microstructure showing alumina phase (bright) in a glassy matrix (dark).

2.1.1. Commercial LTCC components

The specimens used for the biaxial strength tests were cut from commercial LTCC-Tapes (panels of ca. 100 x 100 x 0.45 mm³), provided by the company TDK-EPC, Deutschlandsberg, Austria (Fig. 2).

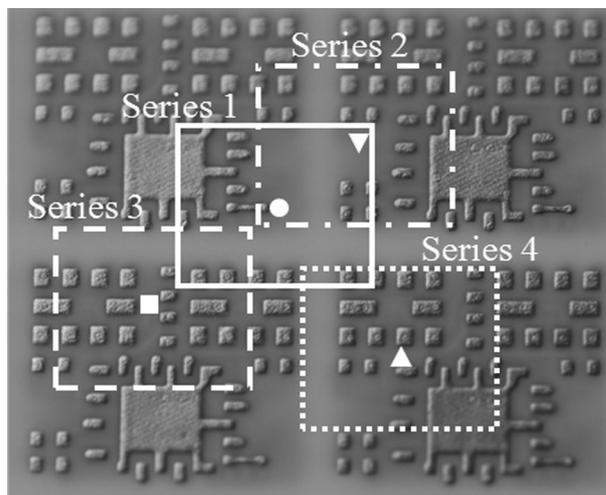


Figure 2: Scheme of a panel where the samples are cut out in such a way that the position to be tested lies in the centre of the specimen.

For the inner metallisation only Ag is used, whereas for the outer metallisation the silver pads are covered with a nanometric Ni/Au layer, which is employed to avoid corrosion and further guarantee good adhesion of possible soldered components. The thickness of the metal pads is approx. 7 μm . The processing route employed was tape casting and the co-sintering was performed in a furnace at 850 $^{\circ}\text{C}$. After sintering, four different samples of rectangular plates of ca. 11.0 x 9.7 x 0.45 mm^3 were cut from each panel, as indicated in Fig. 2. In each case, the centre of the plate contains a different region of the LTCC-unit. Bulk specimens of the same dimension were also tested as reference material.

2.1.2. Sample LTCC

In order to distinguish the effect of inner structures and surface metallization on the biaxial strength, sample structures were fabricated using the same process as for commercial LTCC components. Rectangular testing plates of ca. 11.0 x 9.7 x 0.45 mm^3 were diced from each panel with a diamond saw. In addition to bulk material without metallization as reference material (Fig. 3a), three LTCC samples with different surface metallization were fabricated:

- (i) MT: Bulk LTCC with a full metallization layer of ca. 10 μm on top of the substrate (Fig. 3b).
- (ii) CP: Bulk LTCC with contact pads of 1.5 x 1.8 mm and ca. 10 μm thickness, covering the 75% of the top surface (Fig. 3c).
- (iii) CV: Bulk LTCC with cylindrical vias on the top with a diameter of 100 μm and a pitch of 300 μm , with a depth into the material of ca. 50 μm (Fig. 3d).

2.2. Mechanical testing: Ball-on-three-balls

The biaxial strength of the LTCC specimens was determined using a miniaturised Ball-on-three-Balls (B3B) fixture (Fig. 4a) especially built in-house to match the dimensions of the supplied components. In the B3B method, a rectangular plate (or a disc) is symmetrically supported by three balls on one face and loaded by a fourth ball in the centre of the opposite face (see Fig. 4b), which produces a very well defined biaxial stress field [15, 16]. The four balls had a diameter of 8 mm. A pre-load of 7 N was applied to hold the specimen between the four balls. The load is increased until fracture occurs, and the fracture load can be used to calculate the maximum tensile biaxial stress in the specimen at the moment of fracture.

For a bulk plate of an elastically isotropic material the equivalent maximum stress σ_{max} corresponding to the fracture load P can be calculated as follows:

$$\sigma_{\text{max}} = f \cdot P/t^2 \quad (1)$$

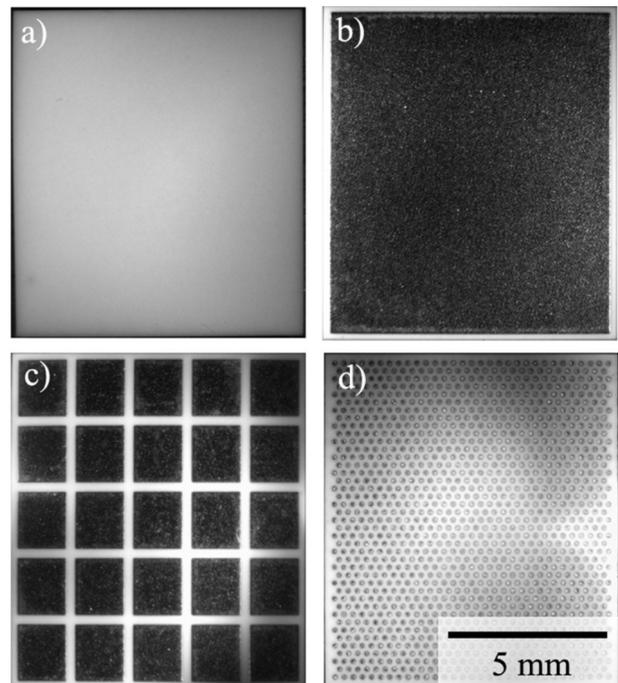


Figure 3: Samples with different metallization at the top surface of the substrate: a) bulk without metallization, b) bulk with full metallization layer, c) bulk with contact pads, and d) bulk with cylindrical vias of 100 μm diameter and 50 μm depth.

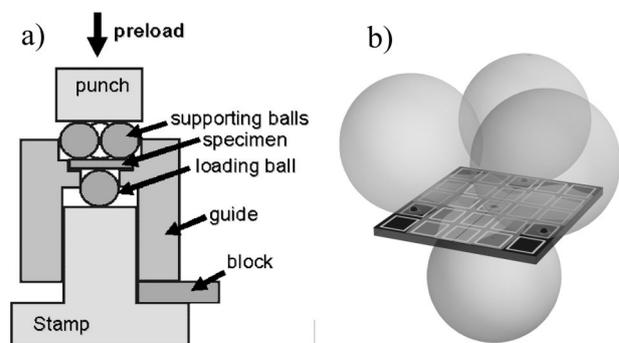


Figure 4: a) Schematic of the ball-on-three-balls test for biaxial testing and b) FE simulation of the stress distribution in the plate during loading.

where t is the specimen thickness, and f is a dimensionless factor which depends on the geometry of the specimen, on the Poisson's ratio of the tested material, and on the details of the load transfer from the jig into the specimen. Elastic properties were determined by means of the Resonant Beam Method [17] in a bulk LTCC sample, resulting in a Young's modulus of $E = 113 \pm 1$ GPa and a Poisson's ratio of $\nu = 0.2 \pm 0.01$. The factor f was calculated for the given geometry and Poisson's ratio as function of the thickness of the specimens. The resulting geometric factors corresponding to commercial LTCC and sample structures investigated here are reported elsewhere [13, 18].

2.2.1. Testing of commercial LTCC

The tests on commercial LTCC were conducted under displacement control at a rate of 0.5 mm/min and a relative humidity (RH) of $23 \pm 2\%$ at $21 \pm 1\text{ }^\circ\text{C}$ (i.e. $H_{\text{abs}} \approx 4.3 \pm 0.4 \text{ g}_{\text{H}_2\text{O}}/\text{m}^3$), using a universal testing machine (Zwick Z010, Zwick/Roell, Ulm, Germany) with a load cell of 200 N. The load was increased until fracture occurred and the fracture load was used to calculate the maximum tensile biaxial stress in the specimen at the moment of fracture using Eq. (1). A total of 30 specimens were tested for each series.

2.2.2. Testing of sample structures

In order to discern the effect of surface metallization (e.g. contacting pads or cylindrical vias) and the effect of the environment on the strength of components, a novel experimental approach has been proposed. Biaxial strength tests were performed within two different environmental conditions: a) in water and b) in silicone oil bath, in order to reproduce high and low relative humidity conditions, respectively. In both cases, the samples (as described in Fig. 3), were placed in the B3B fixture and the corresponding fluid (i.e. water or silicone oil) was pumped into the specimen holder to fully cover the specimen during the entire loading. The temperature was maintained constant for all tests, i.e. $22 \pm 2\text{ }^\circ\text{C}$. The number of specimens tested for each sample ranged between 15 and 30, for statistical significance. The tests were conducted under displacement control at a constant cross-head speed (i.e. 0.1 mm/min) using a universal testing machine, (Zwick Z010, Zwick/Roell, Ulm, Germany).

3. Results and discussion

3.1. Strength of commercial components

Fig. 5 shows a Weibull diagram of the four LTCC series corresponding to four different tested regions of the LTCC-unit. Bulk specimens of the same dimension were also tested as reference material. Each distribution was collected on a sample of 30 specimens, which ensures statistical significance for the Weibull analysis. The nominal failure load, P , is represented vs. the probability of failure, F . The corresponding equivalent failure stress, σ_f , calculated with Eq. (1) for every tested specimen is also represented in the plot. The strength results from the bulk specimens are also shown for comparison.

It can be inferred from Fig. 5 that all series follow a Weibull distribution. Nevertheless, the strength distribution between some of the series with metallisation and the bulk series is different. This can be associated

with stress concentrations near metallization regions and/or residual stresses developed during co-sintering of metal-ceramic parts. In addition, some of the diagrams seem to follow a typical two-parameter Weibull distribution (e.g. series 1 in Fig. 5), whereas others show a so-called “concave banana-shape” (i.e. the strength distribution in the Weibull diagram deviates from a lineal trend, pointing downwards for lower failure stress values). In such cases (see for instance series 2 and 4 in Fig. 5) a minimum failure stress level (“threshold stress”) may be defined under which the material may not fail. This can be a result of metal electrodes arresting the crack in its propagation from the surface towards the bulk. A comprehensive study of the crack propagation can be found in [11, 13].

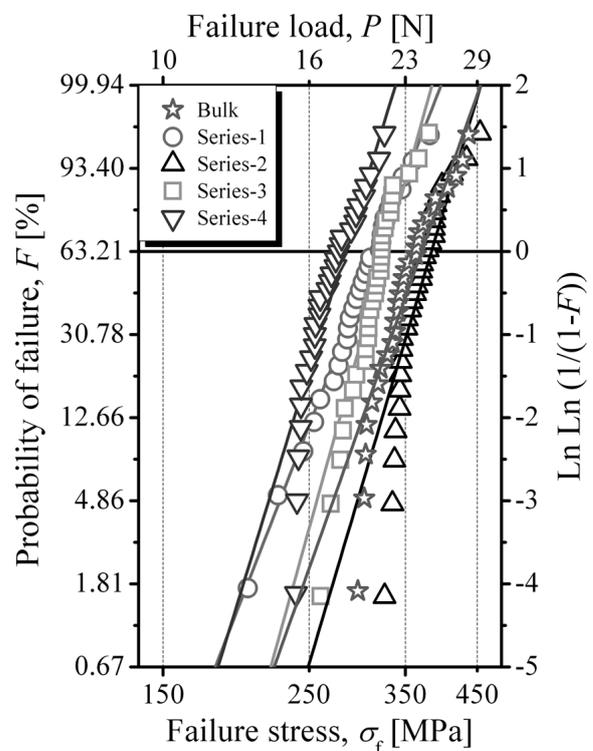


Figure 5: Weibull diagram of LTCC samples corresponding to four different tested regions of the LTCC-unit. A sample of bulk specimens is also shown for comparison.

3.2. Effect of surface metallization on strength distribution

In order to discern the effect of surface metallization (e.g. contacting pads or cylindrical vias) and the effect of the environment on the strength of components, an experimental approach has been proposed, based on localized biaxial testing of brittle samples at several locations under low and high humidity environments (i.e. silicone oil and water) respectively [18]. Figure 6 represents the corresponding Weibull diagram, where the equivalent failure stress,

σ_r (calculated for every specimen according to Eq. (1)) is represented versus the probability of failure, F , for the bulk sample tested in water and in silicone oil. It can be observed that the failure stress values follow a Weibull distribution, which is associated with the flaw size distribution in the sample. The Weibull parameters, σ and m were calculated according to EN-843-5 [19]. The full line represents the best fit of the strength data using the maximum likelihood method. The 90% confidence interval for the measured values is also shown in the referred figure as broken lines. It represents the range where the true Weibull parameter can be found with a 90% probability and reflects the influence of the sampling procedure. The characteristic flexural strength and Weibull modulus for all samples tested, with the calculated 90% confidence intervals, are given in Table 1.

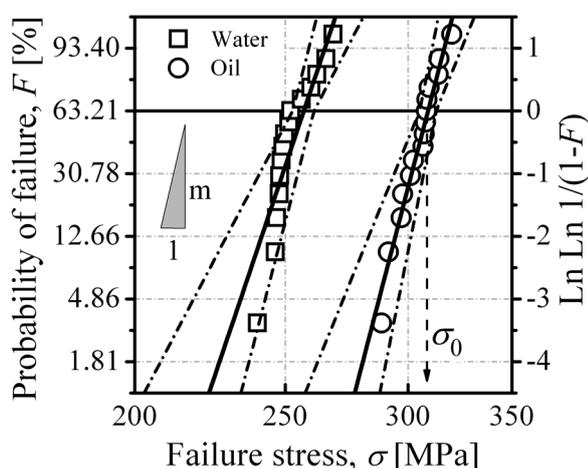


Figure 6: Weibull diagram of bulk LTCC samples tested in water and in oil.

Table 1: Characteristic strength, σ_0 , and Weibull modulus, m , of samples with different surface metallization, tested in water and in silicone oil.

Sample	Water		Silicone Oil	
	σ_0 (MPa)	m	σ_0 (MPa)	m
Bulk	257 (253–261)	32 (20–42)	309 (305–313)	41 (26–54)
MT	303 (295–311)	20 (13–26)	305 (300–311)	27 (17–35)
CP	296 (289–303)	22 (14–29)	298 (294–302)	38 (24–50)
CV	164 (161–167)	28 (17–37)	220 (217–222)	42 (27–54)

The effect of the environment on σ_0 can be clearly seen in Fig. 6, as has been reported elsewhere [14]. The SCCG of defects during loading in water diminishes the strength (ca. 20%) as compared to testing in dry conditions for the same loading rate. According to the results listed in Table 1, for the case of samples with a

metal layer on the top (tensile) surface (MT samples), no difference can be appreciated between both environments, yielding strength values similar to those for bulk substrate tested in oil (i.e. 309 MPa). This indicates that the metal layer “protects” the substrate from SCCG effects. The same applies for CP samples loaded both in water and in oil (see Ref. 18 for more details). A different situation is found for CV samples which reveal not only an effect of the environment (i.e. $\sigma_0 = 165$ MPa in water vs. $\sigma_0 = 220$ MPa in oil), but also a negative effect of the vias on the strength, compared to substrate (bulk) material (see Table 1); i.e. strength can be reduced up to 40% compared to bulk material.

4. Conclusions

In summary, it has been shown that localised biaxial testing using the ball-on-three-balls method performed in different environments can be used as methodology for reliability assessment as well as to quantify the effect of surface features on the strength of functional ceramic components. Whereas surface contacts such as cylindrical vias can become weak points in the design, metallization at the surface subjected to tensile stresses can even raise the strength of the component acting as a protective layer against environmental degradation.

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