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3-D STRUCTURATION OF LTCC FOR SENSOR MICRO-FLUIDIC APPLICATIONS

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Abstract: Low Temperature Co-fired Ceramic (LTCC) Technology is recently addressed to be a key approach among advanced packaging technologies, offering many advantages over its competitors. One of its most attractive features is the ease of 3-D structuration thanks to the LTCC green tapes. These tapes can be easily cut into desired forms in the way to realize both structural and electrical functions under a single system. Three dimensional structures such as membranes, channels can be achieved either by cutting and laminating LTCC tapes or by using a sacrificial layer. In this work, we demonstrate fabrication of an LTCC micro-fluidic sensor using carbon-black paste as the sacrificial layer. The results indicate extremely fine-sized structural dimensions without deformation, which are attractive for pressure or thermal sensor applications.

Keywords: LTCC, 3-D structuration, Carbon-black paste

1. Introduction

Although it has been used since 1980’s, LTCC technology has recently become a widely preferred approach for micro-packaging world due to its versatile character of benefits [1-8, 14]. It provides LTCC green (unfired) tapes, which serve as dielectric substrates to carry both the mechanical and the electrical functions in a single system by the possibility of integration of thick film inks of passive electronic components. Its outstanding dielectric properties (low dielectric losses at high frequencies), co-firable character with highly conductive thick film conductor pastes such as Au, Ag at low temperatures (<900°C) have made it attractive for the telecommunication sector for many years [1-3, 5, 14]. By the introduction of LTCC tapes of various producers into the market, the prices dropped and this opened new applications for this technology such as sensors and micro–packaging [4, 9-14]. In spite of many advantages, which make LTCC technology suitable and attractive for sensor applications, it is basically the ease of fabrication of three-dimensional structures using multiple layers -as a common trend- or a sacrificial layer to build mechanical features in a hermetic environment [4, 15-17].

In this paper, we suggest an efficient solution for obtaining deformation-free 3-D structures integrated in LTCC for realization of micro-fluidic sensors. We explain processing of a sacrificial layer, which is carbon-black paste, and demonstrate the structural features obtained by this method such as a 40µ-thick membrane with 16mm-diameter and 15µ spacing.

2. Materials characterisation

We selected the carbon-black paste as the sacrificial layer for our purpose. In order to prepare this special ink, we basically followed the same procedure as for thick film pastes. Thus, the processing steps required utilization of a functional element, which is the carbon powder (graphite), with a proper organic vehicle in order to retain the right viscosity and rheology for screen-printing.

Graphite was selected as a sacrificial layer because it is known to burn away in air above ca. 600°C, which is intermediate between the debinding and sintering temperatures of LTCC. In air (the atmosphere used to fire LTCC), the graphite powder is removed as CO_2 gas (1) from the system.

$$C(s) + O_2(g) \rightarrow CO_2(g) \qquad (1)$$

Moreover, graphite does not react with LTCC nor contaminate the surroundings. Finally, it can easily be applied as a thick-film paste.

Thermo gravimetric analysis (TGA, Mettler TG50) was used to check the temperature range of the carbon. From the resulting curve (Fig. 1), graphite removal occurs between 600 and 800°C.

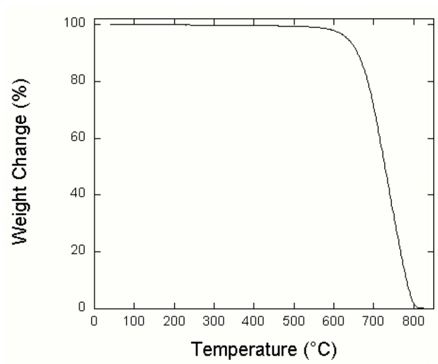


Fig. 1. Thermo gravimetric analysis (TGA) of graphite heated in air at 10°C/min.

Ideally, graphite removal should be complete below the temperature where the open porosity of LTCC is closed by densification. Therefore, the sintering of LTCC was studied by dilatometry (Setaram). For this experiment, LTCC pellets were prepared by pressing powder obtained by dissolving the organics of the tape using acetone in ultrasonic bath for 20 minutes and heating the resulting powder up to 250°C.

The dilatometric curve (Fig. 2) shows that shrinkage starts around 660°C and continues up to ca. 875°C (slightly above the range of Fig. 2), resulting in a final shrinkage of ca. 15%. An usual relative density of 85% to close the open porosity corresponds to ca. 10% shrinkage and 800°C.

Densification was also ascertained by scanning electron microscopy (SEM, Fig. 3). Liquid phase sintering leads to the wetting of the particles by melting of the glass phase starting from 700°C. Above, inter-particular regions are gradually filled. It is only after 800°C that the open porosity is no longer visible.

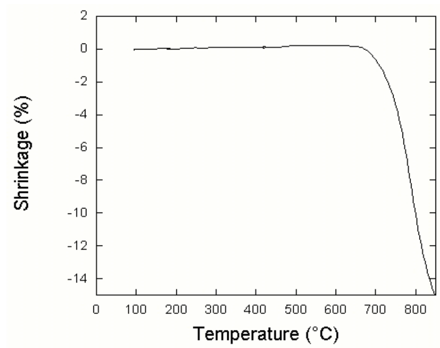


Fig. 2. Dilatometric analysis of a Dupont 951-AX tape.

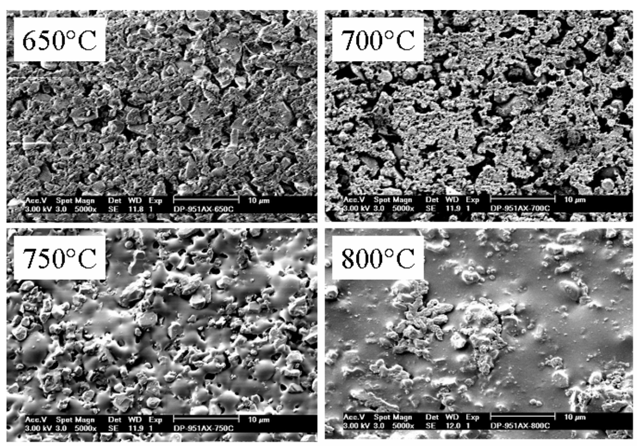


Fig. 3. SEM of the fired LTCC surfaces at different temperatures

The results of TGA, dilatometry and SEM indicate that the end of carbon removal and the closing of the open porosity occur at about the same temperature (around 800°C). As our estimation of the threshold for open porosity is not accurate, experiments are under way to determine it directly by measuring gas permeation through the LTCC.

In order to ensure full carbon removal at a lower temperature, it is possible to lower the heating rate.

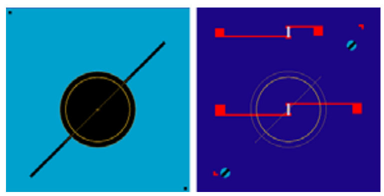
3. Fabrication of 3D LTCC structures

The following procedure and materials (Tab. 1) were used to fabricate the carbon paste. Processing route started by dissolving the ethyl cellulose in terpineol at 80°C for 10 minutes. The graphite was then gradually added to the mixture, together with acetyl acetone. The resulting paste, which is made at a ratio of 28:72 for functional element to organics, was homogenized in a three roll.

Product	Function	Specification	Supplier
Graphite	Functional element	1-2µ particle size	Aldrich, 28,286-3
Ethyl cellulose	Binder	control of rheology	Aldrich, 43,383-7
Terpineol	Solvent	slurry viscosity	Fluka, 86480
Acetyl acetone	Dispersant	dispersing additive	Sigma-Aldrich, P775-4

Tab. 1. Materials used for the carbon paste production.

The layout of the test structures, shown in Fig. 4, comprises a membrane with 2 inlet / outlet channels on opposite sides, with two resistors (measurement and reference).



(a) (b)

Fig. 4. Layout for the carbon paste (a) and for the resistors (b, post-fired after sintering the LTCC).

The carbon paste was screen printed according to Fig. 4a on thick LTCC tape (Dupont). After drying at 70°C in a lab oven for 20 minutes, a 50µ-thick tape (Dupont) was laminated on it by uni-axial pressing at 25 MPa. This step was followed by firing the laminated structure in an ATV-PEO 601 oven with 875°C and 25 minutes being the peak firing temperature and time correspondingly.

4. Characterization

The cross section of the resulting structures were inspected by SEM. Fig. 5 shows a membrane that is a 40µ-thick with 16mm-diameter and 15µ spacing without sagging or any significant deformation. A comparison with a similar structure made by multilayering is given in Fig. 6. In the case, where an inner LTCC layer is cut and laminated to form the feature, there is a problem of incompletely bonded layers between the tapes in the regions close to the cut. This is not the case for regions away from the cut in the same material. However when a sacrificial layer is used, the material is well bonded up to the membrane edge.

Firing was followed by screen printing of thick film conductor and resistors, which were then fired (post) on the previously fired membrane structure at 850°C of peak temperature for 10 minutes in a belt furnace (Serratherm). Fig. 7. shows a complete micro-fluidic sensor with pipes glued onto its ports.

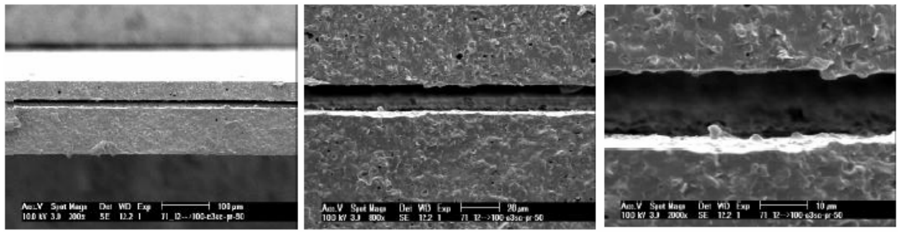


Fig. 5. Cross section of membranes by SEM.

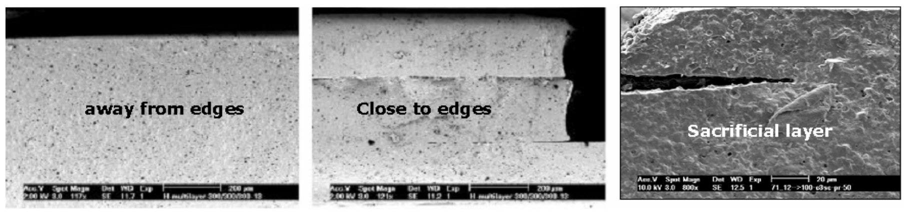


Fig. 6. Differences between the multilayered and sacrificial layer used structures.

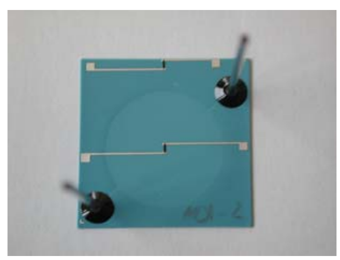


Fig. 7. Membranes with 40µ-thickness, 16mm diameter and 15µ-thickness.

5. Conclusions

In this work, it has been demonstrated that using carbon paste as a sacrificial layer for fabrication of 3-D sensor structures in LTCC, for example for flow and pressure sensing, can be achieved successfully and offers many advantages for realization of fine features.

- Membranes of 40µ thickness and 16mm diameter, with 15µ spacing can be obtained without significant deformation.
- The fired structure is a homogenous and well-integrated system, which can be post fired with thick film conductor or resistor pastes for device applications.
- The process is high yield and practical, once the ink properties are optimized and adopted to the LTCC material.

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