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# Processing of graphite-based sacrificial layer for microfabrication of low temperature co-fired ceramics (LTCC)

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**Abstract**

The processing and application of graphite powder-based sacrificial layer for fabrication of microfluidic structures in LTCC is described. Such layers are produced as pastes, which are screen-printed in LTCC sheets to avoid sagging, by supporting closed, three-dimensional structures such as channels, membranes during firing. The aim of the paper is to highlight the selection of paste materials and the effects of processing conditions on the fabricated microfluidic components. It is seen that the complete burnout of graphite powder is the most critical stage as it is in kinetic competition with the open pore-elimination process of LTCC, which occurs at 785 °C in our system.


*Keywords:* LTCC; Fabrication of microfluidic structures; Sacrificial layer; Permeability of LTCC

## 1. Introduction

LTCC technology is based on LTCC tapes, which are cast from slurries of ceramic-filled glass systems those mixed with an organic vehicle [1–3]. They are produced by commercial suppliers at different thicknesses [4] varying from 30 to 350 μm. Low temperature and co-fired terms refer to firing temperatures below 900 °C and simultaneous firing of LTCC tape and thick-film pastes of electronic components, respectively [1–3]. Both of these keywords signify various benefits in microsystems engineering. In addition to processing feasibility, low firing temperatures permit the utilization of conductors with low resistances such as Au, Ag, Ag/Pd, which have low melting temperatures and can ideally be fired below 900 °C [5]. On the other hand these thick-films can be screen-printed and fired on LTCC substrates, which have low dielectric constants (*K*), creating an ideal platform for high-speed signal circuitry due to low losses [6]. Consequently, the technology has been used for long years in telecommunication sector for high-frequency applications and/or for highly reliable electronic army components [3,7,8]. Recently, applications in sensorics and microfluidics, which originate from other interesting properties of LTCC technology, have been demonstrated [8–11]. Thermal and chemical stability of LTCC tapes, high-packaging density with increased functionality capability, high yield-low cost production possibility in industry, combination of electrical and mechanical functions in a hermetic system can be counted as other attractive features.

However all of these different applications benefit from one great advantage of this technology, which is the ease of utilization of LTCC tapes. They can be cut by laser, punched, screen-printed with a large variety of conductors and passive electronic components, and then laminated up to 80 layers. This provides considerable flexibility in device fabrication.

For microfluidics, one additionally needs the ability to create controlled voids in order to fabricate structures such as membranes and channels. To this end, this paper aims to introduce a sacrificial paste, which is based on graphite-powder, to support the closed, three-dimensional, microfluidic components during firing. The selection of the paste materials, their characterization, and effects of processing conditions will be explained in details. Moreover a recently developed closed-chamber system for permeability analysis of the LTCC substrates will be presented. Finally the fabricated structures will be demonstrated in relation to the processing conditions. The methods of the study will be thermogravimetric (TGA) and dilatometry analysis, scanning electron microscopy (SEM) and electronic systems to measure the permeability of the LTCC substrates.

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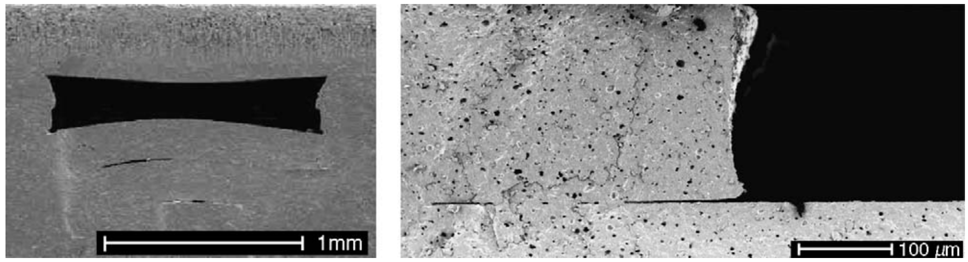


Fig. 1. Sagging (left) and delamination of unsupported samples during firing.

## 2. Preparation of sacrificial pastes

There are different methods to prevent deformation of LTCC structures during firing [12]. These can be classified as passive and active methods, which are based on manipulation of lamination conditions and utilization of sacrificial layers (graphite powder, fugitive phases, etc.), respectively. Although the common aim is the reduction of deformation, which is mostly observed as sagging, delamination, etc. (Fig. 1), the selection depends on the specifications and acceptable tolerances of the desired device. From this paper's point of view, the latter choice has become the ideal approach since fabrication of microfluidic components requires full integrity and well-defined dimensions free of deformation.

On the other hand the motivation for selection of graphite powder-based paste is primarily due to its easy burnout, which ideally occurs according to Eq. (1). Thus, the paste supports the three-dimensional structure up to the burnout temperature of graphite powder, which is followed by gas removal and further densification of the LTCC tape.

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

Secondly, the paste can be screen-printed, which facilitates fine and easy structuring of desired patterns. Additionally it does not interact with the LTCC tape during firing.

We prepared the pastes in a similar fashion to those in thick-film technology: the functional material was blended with the organic vehicle in order to retain sufficient rheology for screen-printing. In our case, two types of graphite powder, which vary in particle size, were used as the functional element. From here on we will be referring to fine (2 μm) and coarse (15 μm) graphite powders as FG and CG, respectively, both of which were processed by the same procedure. This is to say the powder was initially blended with the previously prepared mixture of binder (ethy-cellulose) and solvent (terpineol), which was then followed by addition of dispersant (acetyl acetone) to increase the efficiency of mixing. The overall suspension was finally homogenized on a three-roll mill. The exact compositions by weight of the prepared pastes can be seen in Table 1.

Table 1
Composition of prepared pastes by weight percent

| Powder | $d_{50}$ (μm)[a] | Graphite | Binder | Solvent | Dispersant |
|---|---|---|---|---|---|
| FG | 2 | 27.4 | 3.1 | 64 | 5.5 |
| CG | 15 | 25.8 | 2.7 | 69.2 | 2.3 |

[a] Particle size of the powder.

Pastes based on FG and CG were then screen-printed according to the layout shown in Fig. 2, which has the large central area and inlet and outlet channels. The printed tapes (DuPont 951-AX) were dried for 10 min in air and at 120 °C, respectively. Lamination with thinner LTCC tapes (DuPont 951-C2), which was intended for improved device functionality (pressure sensing, reduced thermal dissipation) was carried out at 70 °C under 25 MPa. Finally the structures were fired according to a two-step firing profile in an LTCC oven (ATV-PEO 601). Samples were heated at a rate of 5 °C/min and kept at 440 °C for organic burnout of the tape. This step was followed by heating the samples at the same rate up to the peak firing temperature of 875 °C (peak dwell time of 25 min).

## 3. Results

The most important material properties and processing parameters, which directly influenced the fabricated membranes, can be summed as the chemistry and the particle size of the graphite powder used, heating rate, diameter of the membrane and the thickness of the overall module (number of thicker LTCC layers). Fig. 3 shows the membrane cross-section of a 7 mm-diameter membrane, which was prepared by FG-based

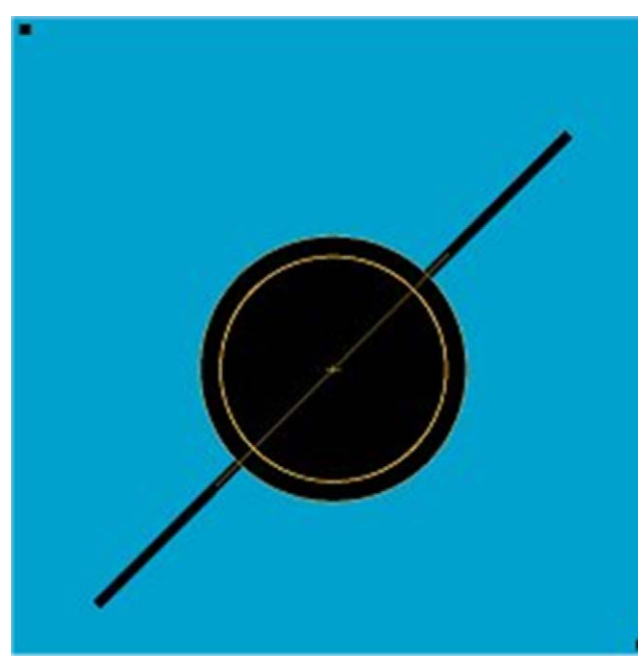

Fig. 2. Layout for the membrane of the microfluidic device.