# Hybrid sol-gel-glaze planar optical waveguides on LTCC substrate – preliminary works

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Preliminary works on optical planar waveguides made from two different materials on LTCC (low temperature cofired ceramic) substrates have been presented. Sol–gel-derived silica and glaze layers were used to prepare the waveguide structure. This kind of component can be used in integrated optoelectronics devices, which implicates a higher scale of integration and reliability. Furthermore, thanks to this method, it is possible to connect passive components made in thick-film technology with planar optical waveguides on one substrate. Described structures can be used in many devices. The most interesting is their application in sensors with optical detection, telecommunication elements, *e.g.* optical switches or devices called "lab-on-chip". The main aim of the research was to develop a new method of producing optical planar waveguides. Choosing the best materials for both types of layers was made in the first part of the investigation. Very important is the compatibility of the materials and the interaction with LTCC substrate, especially during firing. This kind of test was made. The paper presents the results of experiments for choosing the best type of glaze from four low temperatures, and transparent types of them. Verification of interactions between a silica layer and a glaze determines the best process parameters. Optimization of a waveguide size and shape is described as well.

Keywords: low temperature cofired ceramic (LTCC), sol-gel, planar optical waveguide, integrated optics elements.

# 1. Introduction

Integration of passive optoelectronics with microelectronics devices is a well known issue [1]. Components such as micro-lenses or optical waveguides could increase the scale of integration, decrease the size of a device and open a new area of application [2, 3]. In microsystems based on ceramic technology, obtaining integrated elements is more difficult because high temperature in process flow is needed and some technologies, like doping, cannot be applied. Replacing external passive optics

(*e.g.*, optical fibers) with integrated components in ceramics devices enables the usage of active elements (*e.g.*, light source and detectors) added on the same substrate. This solution provides a lower number of connections, smaller distance between elements, more accurate alignment, especially in unfired ceramics, what all in all leads to higher efficiency of the designed system. Moreover, the elimination of additional materials and operations, like gluing, can increase device reliability.

It is worth to mention that the integration of optic and optoelectronic elements with microelectronic devices is always a difficult issue. The main problem is the size of elements because they must be very precisely placed. For integrated optical structures very important are also interactions between a substrate and optical element, especially at high temperature. In the LTCC technology during firing some particles (*e.g.*, lead) can diffuse from a substrate to optical material [4]. This interaction is unwanted and in some cases could disturb the proper work of final devices.

Surface roughness in optical applications is also a very important parameter. Every kind of surface unevenness can lead to a very high level of light dispersion. This problem can be neglected in substrates like glass or silicon, where surface roughness is in the range of single nanometres, but in ceramics it could be the main reason of light dispersion. For ceramics like DuPont 951, the roughness is 0.3  $\mu$ m according to the datasheet. For this reason, materials used in optical elements deposited on LTCC, beside their main task, should also decrease roughness.

The application opportunities for structures with integrated optics are very wide. One of the most obvious is optical telecommunication [5]. However, devices produced in this technology can be used not only in passive, like optical switches or multiplexers, but also in active advanced elements, like light amplifiers. One of the most interesting and promising applications are sensors with optical detection. Thanks to the use of planar optical waveguides, it is possible to transport light signal exactly to the measurement position. This leads to small devices where measurement is carried out precisely in the proper place. Moreover, this solution allows to make devices without electrical signals, which are proper for working in a dangerous, explosive environment (*e.g.*, methane detection in mines). These integrated optical waveguides could be connected with standard glass fibers, which gives opportunity for application in telemetric sensors.

Considering all these elements, new methods of producing passive optoelectronics elements are needed. Up to now planar waveguides on LTCC substrates are made only from organic materials like epoxy-based negative photoresist SU8 [6], which is suitable only for application below 150 °C and its stability in time is insufficient. The aim of this research was to obtain technology which allows to make optical elements for integration with LTCC devices. They should be resistant to high temperature and could be used in a wide range of applications. Very promising is a conception of using the sol–gel technique for thin optical layers deposited on LTCC substrates.

### 2. Technologies

#### 2.1. Low temperature cofired ceramics (LTCC)

LTCC is included in the group of thick film microelectronic technologies. The main advantages of this material is the possibility to obtain the ceramic structures (also 3D) without using very high temperature (above 1000 °C). This technology enables to make buried layers with passive components (resistors or capacitors) [7], 3D elements like microreactors and channels, and some sensors. Moreover, LTCC is cheaper than other microelectronic technologies, does not need a very clean environment (*e.g.*, "clean-room"), and it is cost-effective for small series of devices. For these reasons, LTCC is one of the best methods to make multilayer, hybrid microelectronic structures.

The LTCC process uses, as a starting point, a thin unfired ceramic tape sheet. After cutting sheets to the demanded shape (most often by laser or mechanical punching), the passive and dielectric components are made by the screen printing method which is typical of thick film technology. If the structure is a multilayer, then interconnections through a single layer are made by vias, which are punched holes filled with conductive paste. Separate layers are stacked, laminated and cofired at 875 °C in order to form a monolithic multilayer structure. In post process operations, active or passive components can be added to the top or bottom part of the fired structure using various assembling methods (Fig. 1).

#### 2.2. Sol-gel technique

The sol-gel processing is a chemical production method of glass and ceramic materials from liquid phase (Fig. 2). Hydrolysis and alcoholic or water condensations of metal alkoxides are the main reactions proceeded in the sol-gel process. Usually, as a precursor of silica, tetraethoxysilane (TEOS) and tetramethoxysilane (TMOS) are used. To ensure faster hydrolysis of alkoxide, acid can be used as a catalyst [8].

The consistency of sol allows coating the substrates by various techniques. Dip-coating and spin-coating are the most popular methods for thin film preparation.



Fig. 1. LTCC processing.



Fig. 2. Sol-gel process.

After deposition, an optical layer should be annealed at high temperature depending on the kind of the material and its form of application. In our case it was 550 °C in order to obtain a dense and crack-free silica layer.

Silica layers made from sol-gel-derived material and annealed at 550 °C were less than 1  $\mu$ m thick. Thicker layers could be prepared using organically modified silicates (*e.g.*, 3-(glycidoxypropyl)trimethoxysilane, GLYMO) during synthesis. Unfortunately, these films could not be used at high temperature applications.

Small thickness of films made them unsuitable for a waveguide core because of difficult coupling of optical signal. For this reason introducing of a new material was needed. Two main desirable properties of this material were: the refractive index higher than in sol–gel-derived silica and the possibility of thick films production. The glaze, used in the thick film technology as an overglaze material, fulfilled these requirements. It had a character of paste and could be deposited in the standard screen printing technique. That means that the waveguides could have different sizes and shapes.

## 3. Experiments and results

As it was mentioned, an interaction between used materials is very important in hybrid devices. The negative effects could occur especially at high temperatures during drying, firing, *etc.* In our case this implicated that the glaze should be fired at as low temperature as it was possible. Thanks to that, particles diffusion between materials could be limited. Moreover, high temperature could negatively affect the quality of a sol–gel-derived film used as a first layer on the ceramic substrate.

Additionally, temperature firing profile, especially time and temperature gradient, also was important. If the firing cycle was too short or the sample was cooled too fast, air bubbles could be held in the glaze. Moreover, if the slope of a temperature profile after maximum value was too rapid, the glaze material could get cracked.

Most commercially available glaze materials were optimized for firing at the temperature near 850 °C, the same as LTCC, so they could be cofired. Furthermore, they were made for about 10–20  $\mu$ m layer thickness (single layer after screen printing). In our solution this temperature had to be as low as possible and for this reason the first part of the research was testing different types of glaze fired at different temperatures. It was necessary because the datasheet temperatures for standard thicknesses were usually smaller than in presented devices. It is caused by effects in glaze volume. The aim task was to choose a type of glaze which gave the best quality of a waveguide core fired in the lowest temperature as it was possible. Four types of commercially available glaze were tested: IP02, IP123, SGL-683 from Heraeus and G-483 from ESL. They were chosen because of the low firing temperature. For each type of glaze, fourteen substrates of LTCC DuPont951 were made. Seven of them were square shaped and four green tapes thick. Remaining seven were in a rectangle shape and each one contained three square cavities, one green tape (147  $\mu$ m) deep.

On the square shape substrates, glaze paste was printed by the screen printing technique. In the other samples, the glaze has filled the cavities. Samples prepared by this method gave information about the glaze layer thickness after firing. It was important because during firing in the volume of the glaze other types of effects could occur than in the thin film.

The most important criterion of the final quality was transparency. If firing temperature was too low, material was milky and unsuitable as a core for a planar waveguide. The second criterion, especially in cases where material thickness was more than 50  $\mu$ m, was the verification of crashes or air bubbles. Each of these effects could make the glaze layer unsuitable for the optical waveguide, because of high optical attenuation on these defects.

Samples were fired in the temperature range from 550  $^{\circ}$ C to 800  $^{\circ}$ C with 50  $^{\circ}$ C steps. Afterwards, they were observed under an optical microscope to estimate their quality.

Experiments showed that no type of glaze could be fired below 700 °C. After heat treating at 550 °C, 600 °C, and 650 °C, all types of glaze had milky color (Fig. 3).

Moreover, in these temperatures the screen pattern was visible under microscope. For thin layers fired at 700 °C, three of four glazes were transparent. Paste from



Fig. 3. Samples on DP951 fired at 600 °C.

Fig. 4. Sample with glaze in cavities on DP951 substrates after firing.

ESL was still milky, that means it still had some unfired ingredients. Unfortunately, in the cavities where the material was thicker, all types of glazes were still unfired.

Next experiments showed that at first the transparent enough material in cavities was obtained at 800 °C from SGL-683 glaze. For this glaze, the results for thin films at 700 °C and 750 °C were also very good (Fig. 4).

Results for glaze IP-02 were good as well. These two materials were chosen for next experiments. The temperatures of 700 °C as the lowest for thin films and 800 °C for thicker films were chosen as well. These first tests showed which glaze was better for transparent thin and thick films. They gave also information about the lowest temperature which could be used to obtain good quality optical materials for light transmission. However, they did not inform about interactions between glaze and sol–gel-derived material during heat treatment, therefore the second experiment was performed.

Sol-gel-derived silica material was deposited on the LTCC substrate made of DuPont DP951 LTCC green tape, TMOS and hydrochloric acid were used as a silica precursor and catalyst, respectively. Films were deposited by the dip-coating method with the withdrawal speed of 30 mm/min [9]. After this, the gel was dried at 80 °C for 10 min and then heat treated in profile with max. temperature of 550 °C and temperature ramp-up 2 °C/min. These operations were repeated three times, so the final layer contained three single layers. This operation gave thicker films and decreased surface roughness more effectively.

The roughness of DuPont DP951 without any coating measured by profilometry was about 300 nm. The experiment confirms the effect of silica films on this parameter decrease. The roughness measured after triple deposition of the sol was about 90 nm. According to the literature, for a cladding in optical waveguide this value should be lower than 100 nm to obtain acceptable light attenuation.

On the substrates with sol-gel-derived silica films, a glaze stripe was made by the screen printing technique (Fig. 5). Glaze was printed twice, which gave the layer thickness of about 20–30  $\mu$ m. The sample was fired at 800 °C. This structure shows that in used glazes no unwanted effects like discoloration of the material were observed.

This structure could be treated as a planar optical waveguide. The simulation made in CamFR software for structure parameters showed that three modes of optical power



Fig. 5. Stripe made of glaze printed on a sol-gel layer.



Fig. 6. Profilometry measurement of glaze strip printed on sol-gel films.

could be transmitted. Unfortunately, the shape analysis done by mechanical profilometry showed that the core surface was very rough (Fig. 6). This effect introduced high attenuation and light was lost very fast. Therefore, the attenuation measurements could not be done yet.

# 4. Conclusions

The first works on the integration of sol–gel-derived materials with glaze as a planar optical waveguide on LTCC substrates were conducted. The subject of the research is complicated because of its novelty and the fact that these materials were not previously connected. For this reason, basic experiments must be carried out. However, first presented results showed that it was possible to connect these two technologies and materials in one advanced structure. The obtained structures are very promising and exhibit the expected properties. Therefore, the research will be continued and results will be presented in forthcoming papers. Optical planar waveguides which are possible to obtain by this method, could be used in integrated optoelectronics devices made in the LTCC technology.

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