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Green-tape ceramics. New technological approach for integrating electronics and fluidics in microsystems

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The miniaturization of analytical systems for different applications is currently a very active field of research. The inherent advantages of micro total analysis systems (µTASs) are well known. Although the fluidic platforms and the development of suitable miniaturized detection systems have been studied extensively, the integration in a single substrate of the electronics that is needed to manage the whole system using a single technology is still a sizeable challenge.

In this overview, we discuss the role of the low-temperature co-fired ceramics (LTCC) as a potential alternative for miniaturizing analytical systems, since a single technology can easily combine fluidics and electronics to produce a number of novel chemical microanalyzers.

Keywords: Electronics; Fluidics; Integration; Low-temperature co-fired ceramics; LTCC; Miniaturization

1. Introduction

The miniaturization of total analysis systems (TASs) in analytical chemistry aims to solve some common drawbacks found in macro analyzers, which, although they provide sensitive, accurate and traceable results, require a relatively high volume of reagents and sample and the operation by highly skilled staff. Additionally, since they are not portable, there is a delay between taking the sample and reporting the results, and this delay can be unacceptable in some applications.

The micro TAS (µTAS) concept began to emerge not only to address these problems but also to provide analysis with temporal and spatial resolution, in line with current trends in several research fields, especially in chemistry, environmental monitoring and medicine [1].

The advantages of µTAS are well known: low production costs; small sample volumes; low consumption of reagents; and, portability. µTASs comprise miniaturized devices able to integrate most of the stages involved in an analytical process [2,3]. The wide variety of key components needed to design µTASs has hindered their full development. Among the functional elements needed are microfluidic structures and their connections [4], pumps [5,6], valves [7,8], injection elements [9,10], reactors [11,12], filters [13], separation and/or preconcentration devices [14], microsensors (physical and chemical) [15,16].

Most of the work in the literature has focused on microfluidics, as it is the basic platform needed for µTAS development. Glass and silicon have been the most widely used materials for this purpose, in part because they are versatile and chemically robust, with relatively straightforward fabrication and the easy integration to optical detection systems [17,18]. Recently, plastic devices have shown a large number of advantages over those manufactured in glass or silicon, including speed of manufacture, the multilayer approach and lower cost of fabrication [19].

However, the procurement of a real, complete µTAS is limited by a number of factors:

- the limited research on miniaturized key elements for fluid handling (e.g., micropumps, microvalves and other microactuators);

- there are no complete solutions to packaging problems at the interface of the device and its environment and the issues of implementation in three-dimensional (3-D) are also still partly unsolved [20–22]; and,
- miniaturization and integration of the electronics needed to control the actuators and the detection system should also be considered, as many of the current microfabrication techniques and materials have proved incapable of easy, fast and low-cost implementation.

In this review, we present low-temperature co-fired ceramics (LTCC) technology as an alternative for the fabrication of μTASs. Using a multilayer approach, the fast prototyping of complex 3-D structures without liquid leakage can be easily attained using a relatively simple infrastructure. Moreover, since this material has been commonly used as an electronic substrate, it is perfectly compatible with screen-printing techniques, allowing the integration of electronic circuits, along with surface-mounted devices (SMDs). Hence, by using a single technology, fluidics and electronics can be integrated to achieve a total miniaturized system.

2. LTCC technology

The ceramic tape in a green state mainly comprises 45% filler (mainly Al_2O_3), 40% glass and 15% organic components (solvent, plasticizer and binder). They are known as ‘‘green’’ tapes because they are mechanized in the ‘‘green stage’’, when they are still soft and malleable, as opposed to hard and cured. Gongora-Rubio et al. gave details about the most remarkable characteristics of LTCC, their common fabrication techniques and some applications, mostly focused on physical sensors [23].

When compared to conventional microfabrication techniques (e.g., using glass, silicon or polymers), LTCC technology shows some additional advantages, including:

(i) rapid prototyping, which allows quick modifications;
(ii) low costs of fabrication;
(iii) no need for special and expensive fabrication facilities, such as clean rooms; and,
(iv) sealing elements, such as epoxies, are not needed, since, after the sintering process, the ceramic layers fuse solidly.

Nowadays, the study of different green-tape compositions to meet several needs is an active field of research for many ceramic manufacturers. Table 1 summarizes some of the most remarkable features of two types of green-tape ceramics.

Once fired, LTCC tapes have a chemical behavior similar to that shown by other common glasses when exposed to harsh conditions (e.g., concentrated NaOH solutions, HF).

Table 1. Physical and electrical properties of DuPont 951AX and Heraeus HL2000 green tapes

	Dupont 951AX	***Heraeus HL2000 5.3***
Physical properties		
Thickness (green state) (μm)	254 ± 13	133 ± 5.1
Shrinkage x-y plane (%)	12.7 ± 0.3	0.2 ± 0.04
Shrinkage z axis	15 ± 0.5	32
TCE (20–300°C), ppm/°C	5.8	6.1
Density (g/cm^3)	3.1	2.9
Thermal conductivity (W/mK)	3.3	–
Roughness (μm)	<0.34	0.7
Electrical properties		
Dielectric constant @ 3 GHz	7.8	7.4
Isolation resistance @ 100VDC, Ω	>10^{12}	10^{13}
Breakdown potential, V/μm	>1000/25	3000/thickness layer

Not only the great variety of tailored-made ceramics, but also their compatibility with other materials makes the ceramics suitable substrates for several applications. Some groups have focused their research on the compatibility of LTCC and piezoelectric thick-film layers to obtain sensors and actuators [24–26]. Hydrogenated amorphous silicon carbide (a-SiC:H) thin films have also been deposited onto LTCC ceramics, due to their excellent properties as passivation layers for microsensors and microactuators operating in harsh environmental applications [27]. The excellent compatibility between green tapes and thick-film technology has also been demonstrated by integrating other useful elements in microsystems (e.g., thermistors for temperature sensing [28]). Aerosol-deposition techniques can also be used to embed electric components to reduce the final size of the miniaturized device [29].

Fig. 1 shows the general fabrication procedure of an LTCC device.

Since this technology is based on a multilayer approach, the desired design must be decomposed in separate layers. Each of these layers contains a certain geometrical pattern. Because the entire layers overlap, a three-dimensional structure is achieved.

The layers are designed using computer-assisted design (CAD) software. After that, they can be mechanized using different methodologies (e.g., drilling, laser, jet-vapor etching or computer numerically controlled (CNC) machining). Due to their affordability, CNC machines have been widely used (Fig. 2). However, to attain smaller features in shorter times, an IR laser can be a better alternative.

The screen-printing stage (Fig. 3), used to integrate the conductive tracks and planar passive electronic components normally needed in detection and signal-

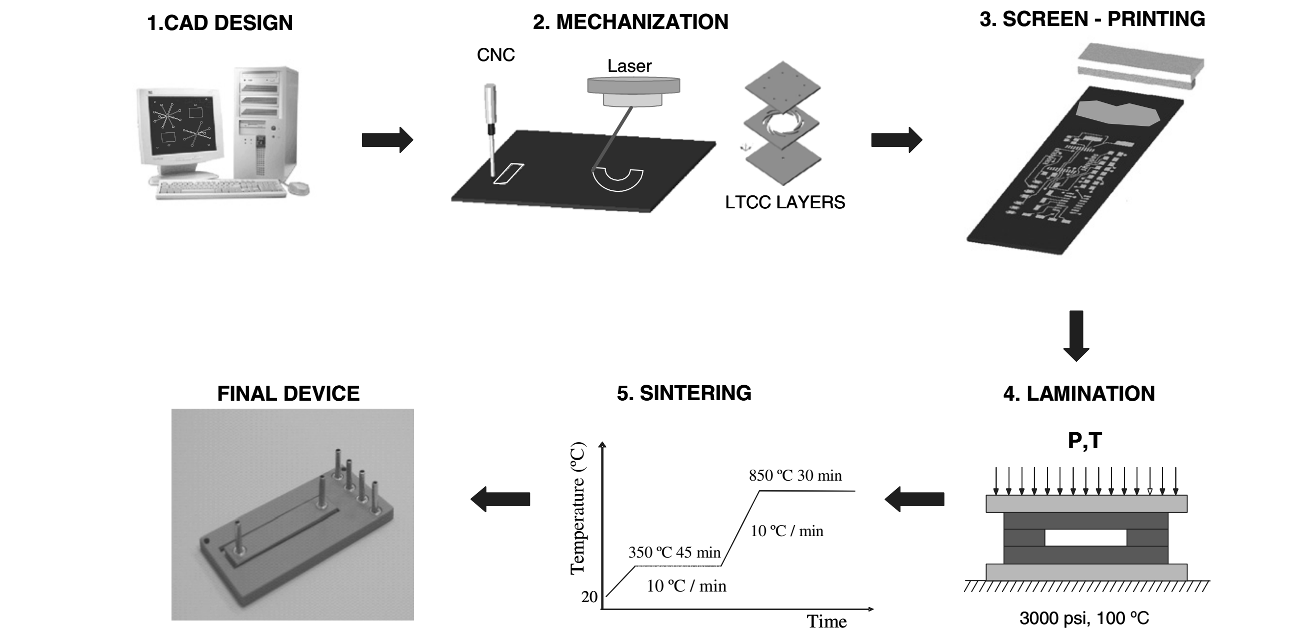

Figure 1. General fabrication procedure for a miniaturized LTCC device.

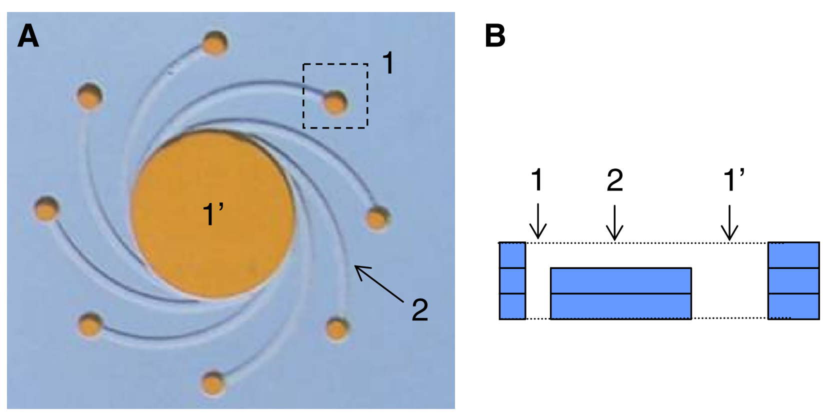

Figure 2. Ceramic layer mechanized by a CNC machine. (A) Mechanization of three laminated layers with two approaches: total removal (**1** and **1′**) and bass relief (**2**). (B) Cross-sectional view of the three laminated layers.

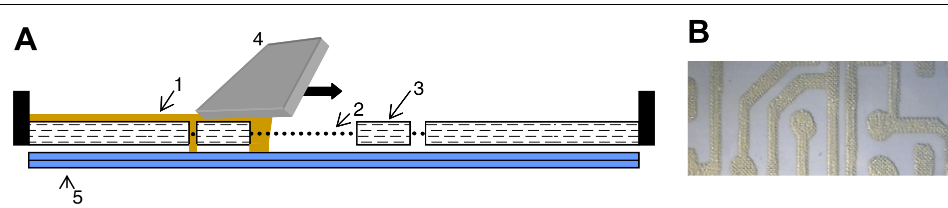

Figure 3. (A) Screen-printing process for three laminated LTCC. **1:** Screen-printing conductive paste; **2:** Metallic or polymeric mesh; **3:** Emulsion over the mesh delimiting the areas to be screen-printed; **4:** Squeegee; **5:** Laminated ceramics. (B) Part of a screen-printed circuit on LTCC.

processing systems, is usually done in more than one layer. Conducting tracks on each layer are connected by means of vials filled with conductive screen-printing paste. In this fashion, multilayer circuits are obtained in a more compact way, reducing their size, a desirable feature in miniaturized systems.

Once all the layers have been mechanized and screen-printed, and before they undergo the sintering process in a programmable box furnace, a lamination stage is required. A classical lamination procedure, used to bind the layers partly, involves applying temperature (100°C) and pressure (3000–5000 psi) for short times (3–5 min) [23].

Recent research has shown an alternative method that requires a lower pressure, so that deformations usually found in the patterns made are avoided. This process, known as cold and low-pressure lamination (CLPL), is based on the use of adhesives or organic fluids between layers [30]. These components provide a temporal binding between the ceramic layers that is strong enough and facilitates interpenetration of Al_2O_3 particles. The combination of both lamination methodologies can be a good option in order to avoid deformations arising from excessive pressure in complex ceramic structures.

The final stage is sintering the laminated ceramics. This step can be performed in air or a nitrogen atmosphere. The temperature profile depends on the specifications of each manufacturer. It normally involves two temperature plateaus. In the first (200–400°C), all the organic components burn out. The second (600–900°C) is the temperature at which most glasses have their vitreous transition temperature. At this point, the alumina particles can interpenetrate the original ceramic layers so that, at the end of the sintering process, no difference between those layers can be observed [31]. One of the most important characteristics of green-tape ceramics is the shrinkage suffered by the layers due to the volatilization of the organic components in the sintering step. Some manufacturers (e.g., Heraeus) have developed zero-shrinkage ceramics. Furthermore, there are also some techniques to avoid this phenomenon, that include the use of modified ceramics (containing a higher amount of Al_2O_3 and BeO) [32,33], or sintering under pressure conditions or using sacrificial layers [34].

3. LTCC applications

Most LTCC applications found in the literature refer to the use of ceramics as substrates for electronic circuits (e.g., microwave and radiofrequency circuits, and physical sensors). Although information on the use of ceramics for fabrication of fluidic devices or miniaturization of whole measurement systems is scarce in literature [35,36], recent work, reviewed in this article, shows the versatility of this novel technology. For that purpose, in this section, we discuss LTCC applications in electronics and fluidics.

3.1. Sensing and microfluidic platforms for microanalytical systems

Sensing with LTCC devices has mostly been accomplished for physical parameters. Due to the high thermal and pressure resistivity of the ceramics, there are some examples of applications in the automotive field (e.g., Schmid designed a mass-flow sensor to control the fuel injection in an engine-control module (ECM) [37]). Wilde et al. [38] and Fonseca et al. [39] fabricated a pressure sensor capable of working at high temperatures in turbine engines. LTCC-based eddy-proximity sensors have also been used in the automotive industry to position metallic objects in anti-blocking systems (ABSs) and engines [38].

From a chemical point of view, there are also some papers about sensors that can be used in or that have been integrated into a miniaturized system. Teterycz et al. [40] and Pisarkiewicz et al. developed gas sensors to detect carbon monoxide, ethanol and ozone in air or hydrogen [36]. Hot-layer electrochemical sensors have been constructed to study the denaturalization of proteins [41].

Recent research has focused on the development of ceramic-based miniaturized analyzers aimed at the detection of environmental pollutants. All of them demonstrate two fundamental concepts:

- scaling down conventional continuous-flow analytical techniques, exploiting the advantages of the LTCC technology; and,
- the simplicity and the speed of prototyping using this technology, compared to silicon-based or polymer-based microdevices.

The time-consuming design and fabrication processes inherent in silicon-based devices have deterred widespread application of continuous-flow analytical systems using microelectronic materials and fabrication methods. The main advantages of silicon/glass or polymer technologies are that they are amenable to mass fabrication. However, their use for the production of a small number of tailor-made instruments with short production series is economically unfeasible. Using LTCC technology, almost all the practical scientific knowledge of continuous-flow systems can be deployed in a ceramic microsystem taking advantage of miniaturization. Using a multilayer approach, complex or multi-parametric continuous-flow microsystems can be easily designed. Fig. 4 shows that independent microfluidic platforms for specific analytes can be monolithically integrated in the same device. In addition, complex analytical procedures can be integrated into the same device by distributing the elements required (e.g., pretreatment steps, reagent mixers, detectors) throughout different ceramic layers.

Some of the examples found in literature include fabrication of a heavy-metal sensor using amperometric detection [35]. Although this particular system was not integrated into a fluidic platform, there are some examples of the combination of microfluidics (complex 3-D channels) with one or more detection systems. Most of the analytical methodologies used in routine determinations are based on optical detection, mainly absorption measurements. Scaling down this detection system would be of great interest. However, miniaturization of these

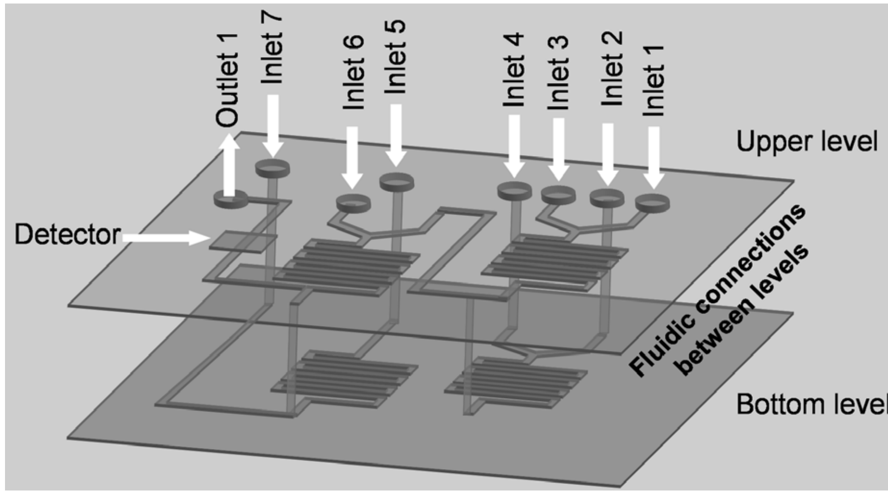

Figure 4. Multilayer microfluidic system with complex analytical procedures defined inside the same substrate.

systems involves scaling down the detection cell, which means a dramatic decrease in sensitivity. Despite this drawback, there are several reports regarding fabrication of LTCC microsystems with optical detection. Golonka et al. presented microsystems that included optical fibers [42,43]. Ibañez-Garcia et al. proposed a compact LTCC microsystem incorporating a monolithic integrated flow cell to carry out spectrophotometric detection [44]. Fig. 5A shows the design and the construction of all the layers that, after lamination and sintering, form the final device (Fig. 5B).

A specially designed flow cell with a vortex configuration, which allows optimized measurements of chemiluminiscence reactions, has been also proposed. This microsystem has been applied to determine several analytes, including cobalt and paracetamol [45].

Other detection techniques commonly used in microsystems include electroanalytical procedures, especially potentiometry and amperometry. Detection based on these techniques is simple, easy to miniaturize and highly sensitive, especially amperometric systems. An example of a potentiometric LTCC microsystem comprises a miniaturized analyzer to determine chloride ions in natural waters. This device integrates a potentiometric sensor, based on a solid $Ag_2S/AgCl$ disk and a 3-D mixer to enhance mixing between reagents [40]. Taking this further, Ibáñez-García et al. constructed an analyzer that integrated not only the potentiometric sensor (in

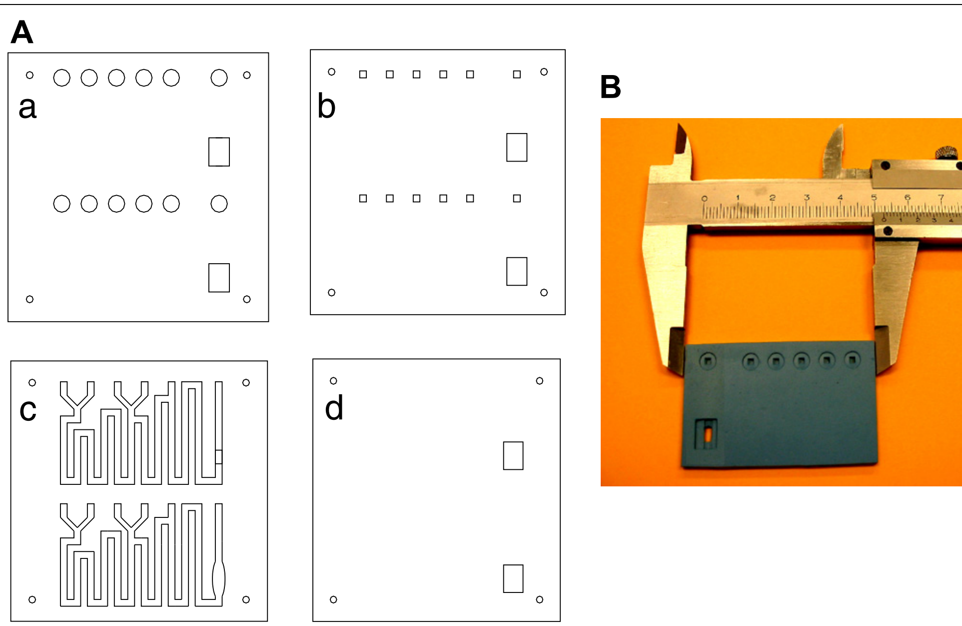

Figure 5. LTCC device to carry out spectrophotometric measurement. (A) Compounding layers from top (**a**) to bottom (**d**). Layer (**c**) shows the inner channels. (B) Final device. The optical window can also be observed.

this case, based on a polymeric ion-selective membrane) but also the reference electrode and the fluidic system in a single substrate. The analyzer was used to determine ammonium and nitrate ions [47].

Llopis et al. designed and developed a complex miniaturized system to determine a pesticide (carbofuran) using amperometric detection and enzyme inhibition. The detection system comprised two platinum sheets (acting as counter and working electrodes) and an integrated reference electrode (based on the Ag/AgCl pair) [48].

The integration of chemical separation techniques exploiting electroosmotic flow phenomena in microfluidic LTCC structures was proposed and realized by Henry et al. [49]. They demonstrated the capability of this methodology in designing lab-on-a-chip devices.

Table 2 summarizes a few remarkable examples of LTCC devices that have been constructed and their general characteristics.

In recent years, important improvements have been achieved in the development of microsystems that include fluidics and sensors (physical and chemical). However, current applications require more autonomous and complete analyzers that also include the associated electronics. This goal, approachable only through a multidisciplinary effort, can be achieved using LTCC technology.

3.2. Electronics for actuators and signal processing

LTCC technology has been widely applied in the production of printed circuit boards (PCBs) due to its excellent electrical, mechanical and thermal properties [50,51]. Its compatibility with thick-film technology and its fabrication process enable the aggregation of several layers to obtain multilayer circuits, which result in high-density boards of high complexity and small size. The multilayer approach also permits each layer to be inspected before being stacked and laminated, reducing or avoiding mistakes in the final device.

LTCC technology combines the dielectric properties of ceramic tapes with the high conductivity of pastes based on gold, silver and some alloys to fabricate high-performance electronic circuits that are applied in many research fields [23,52]. In addition, novel pastes with resistive and dielectric properties have been developed to produce planar passive components (e.g., resistors, thermistors, inductors and capacitors printed on the surface of the substrate or sandwiched between layers [53,54]). Discrete electronic components (e.g., transistors, operational amplifiers, and microcontrollers) can be soldered to produce electronic circuits capable of performing a wide variety of functions. Fig. 6 shows the distribution of several devices (discrete components and planar passive elements) in an LTCC substrate.

As an example of the ease of integrating passive components into ceramics, most electronic applications of LTCC focus on the development of microwave and radiofrequency devices [50,55]. The resultant devices provide examples of the reduction in fabrication costs attained with this technology.

Since the number of layers allowed for this technology is unlimited, ground planes and other noise-reduction components can also be included during the lay-out process. The insertion of ground planes between layers permits increase in signal speed in mobile communications and computer applications. Ground planes also

Table 2. Some remarkable features of LTCC devices that have been constructed

Target	*Remarkable features*	*Ref.*
Control of engines	Simulations of electromagnetic sensors based on the eddy current principle and the use of LTCC	[38]
Pressure sensor	A passive and wireless ceramic pressure sensor operated up to 400°C in a pressure range 0–7 bar	[39]
Gas sensors	Combination of thin and thick film technologies to detect reducing and oxidizing gases in air	[36,40]
Colored compounds	Integration of optical fibers and the use of transmittance and fluorescence measurements	[42,43]
Temperature control	Integration of a sensor/actuator (thermistor/resistance) pair to monitor temperature to carry out reactions at a certain temperature. Accuracy obtained: ±0.07°C	[62]
Nitrite ion in natural waters	Spectrophotometric determination of nitrite ion by means of the Griess-Ilosvay reaction. Limit of detection: 0.027 ppm	[42]
Paracetamol in pharmaceutical compounds	Determination of paracetamol by means of the inhibition of the luminol chemiluminiscence reaction. Limit of quantification:750 μg/l	[46]
Chloride ion in drinking waters	Potentiometric detection of chloride ions by means of a Ag_2S/AgCl sensor. Limit of detection: 6 (±3) ppm	[46]
Nutrients	Potentiometric detection of nitrate and ammonium ions by means of PVC-based ion-selective electrodes. Integration of the reference electrode	[47]
Phenolic compounds	Separation by electroosmotic flow and electrochemical detection	[49]
Pesticides	Determination of carbofuran by enzymatic inhibition and amperometric detection at the nanomolar level. Integration of working, counter and reference electrodes	[48]
Metals	Amperometric detection of mercury and copper by means of anodic stripping. Limits of detection of 0.9 μg/l and 0.45 μg/l, respectively.	[35]
Chloride-ion-detection microsystem	Monolithic device that integrates a potentiometric detection system and the electronics for data acquisition and processing. Limit of detection: 5.4 (±0.1) ppm.	[65]

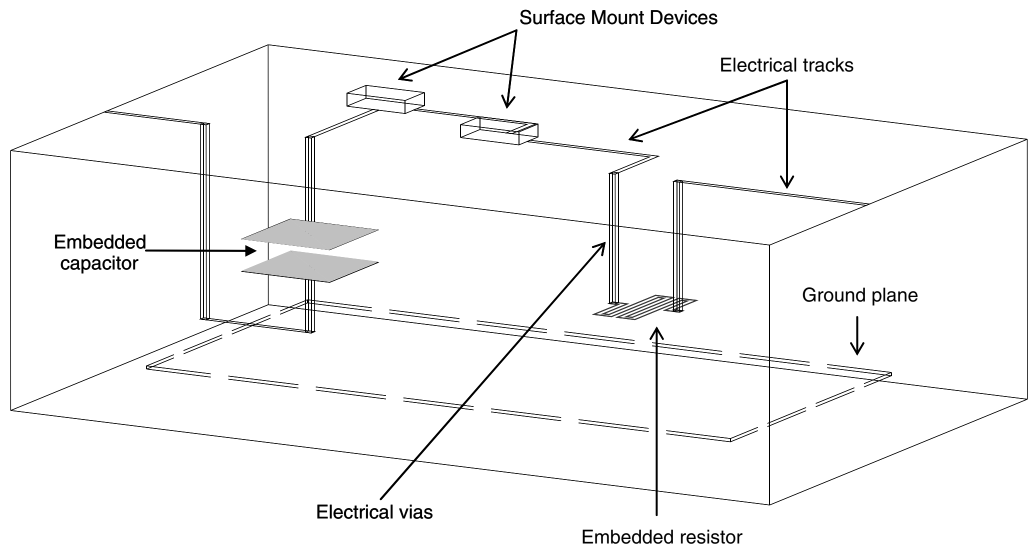

Figure 6. LTCC substrate with embedded passive components and discrete electronic elements mounted on its surface.

serve as electromagnetic isolators needed in highly sensitive electronic circuits. The LTCC-fabrication process only allows half or less of the surface to be metallized, so that tape-to-tape contact is not compromised, so ground grids, rather than ground planes, are used for this purpose.

Taking advantage of the versatility of this technology, some authors have also used green tapes as a substrate to fabricate actuators. Bau et al. [56] and Zhong et al. [57] constructed an electromagnetically driven micropump. Li et al. [58] fabricated omnidirectional wheels that can be used as actuators for vision cameras or robots.

Other interesting types of actuators are those aimed at temperature control: passive heating and cooling systems. The generation of perfectly controlled temperature gradients can be very helpful in polymerase chain reaction (PCR), as temperature needs to be controlled in different zones to ±1°C [59,60]. Moreover, a large number of enzyme reactions increase their reaction rates at 37 °C. Since temperatures higher than 45–50°C can inhibit the catalytic properties of proteins, accurate temperature control is essential in these cases (e.g., Bischoff et al. embedded metal sheets within the ceramic body so that heat could be spread across certain regions of the ceramic device [61]). Chou et al. fabricated a PCR system with air gaps that provided thermal insulation between the three reaction zones [59]. Martínez-Cisneros et al. studied different insulation geometries so that the good thermal conductivity of LTCC did not constitute an obstacle for precise attainment of the desired temperature [62].

The low dielectric losses in green-tape ceramics (dielectric constant at 3 GHz = 7.8), their hermetic sealing after the sintering process and the possibility of constructing complex 3-D structures enable their use as packaging materials for MEMS (micro-electro-mechanical systems) as well as assembly for flip-chip and ball-grid array (BGA) devices [63]. Ruso et al. presented a study about the hybrid integration of LTCC and silicon, where LTCC played the main role as substrate, fluidic circuit board, electrical interconnection and packaging [64].

3.3. Integration of electronics and fluidics

New demands often lead research towards the design of complete analytical devices that integrate all the elements required for specific applications. However, this goal can be reached only through the harmonic combination of several scientific disciplines as well as novel materials and new fabrication technologies.

Silicon and glass technologies are the most widely used microfabrication techniques. They permit the production of electronics, fluidics, sensors and actuators on micro and nano scales. However, as previously mentioned, they require complex, time-consuming fabrication processes. Also, they do not permit integration of discrete electronic components into the same substrate and complete electronic systems needed for a total analysis system cannot always be included in the same chip. As with silicon, polymers allow fabrication of microfluidic channels, and incorporation of electronic components in the same substrate can be achieved through relatively complex processes. However, they do not always require complex facilities, such as clean rooms. But, there are reports in the literature about sealing problems leading to leakage between layers and making it difficult to integrate electronic circuits.

In this review, we have focused on electronics, microfluidics and their integration using LTCC technology to obtain complete miniaturized analyzers. The suitability of this novel technology arises from its easy implementation and low costs (capital and operational), leading to a flexible, cheap prototyping process, and also from the versatility of the ceramics themselves that allow operation of the resulting systems in a wide range of conditions. The integration of these two technological platforms (microfluidics and electronics) can be attained using a modular or a monolithic approach, depending on the final application and the functionality required by each microanalyzer [65]. Fig. 7 presents a monolithic chloride-ion microanalyzer, smaller than a credit card, integrating fluidics, detection and electronics for data acquisition and digital signal processing in a single substrate.

The rapid prototyping offered by LTCC technology permits fabrication of specific and dedicated microfluidic manifolds accurately adapted to each analytical problem. In this way, the modular approach is based on the use of a unique electronic module adapted to a specific detection system (e.g., potentiometry, amperometry or colorimetry) and a dedicated chemical module that can include all the stages involved in a classical analytical process (e.g., microfluidics, pretreatment steps, and detection system). The modular approach is a very versatile tool, as the same electronic platform can be attached to a wide variety of fluidic platforms (based on the same detection principle) to determine several analytes.

Figure 7. Chloride-ion microanalyzer developed with the "lab-on-a-credit-card" concept for integration using LTCC technology.

Following this modular concept, some miniaturized instruments with applications in areas such as potentiometry [65,66], amperometry [67,68] and colorimetry [69,70] have been developed using conventional materials and technologies, such as PCBs). However, these types of miniaturized instruments do not permit construction of monolithic devices (integrating electronics and fluidics in a single substrate). Until now, only a few authors have profited from LTCC properties to integrate both concepts. Moilanen et al. [71] reported multiplexed read-out electronics modules implemented in a LTCC substrate for a PbS detector array.

The intrinsic advantages of LTCC technology permit the monolithic integration of microfluidics, detection systems and electronics, as all of them can share the same substrate and the same fabrication methodology. In this sense, construction of compact, robust and portable microanalyzers with more autonomy can be easily attained.

The potential of LTCC technology to integrate fluidics and electronics monolithically has already been demonstrated [65]. However, the monolithic approach presents a limitation that needs to be considered; unlike the modular system, once the LTCC system has been built monolithically, its analytical application in terms of analyte and type of sample is fixed and it cannot be modified easily, so the monolithic approach can be selected only when microanalyzers are needed for a specific application. In this case, compactness, robustness, low power consumption and total portability, but not versatility, would be the features of interest.

Regardless of how compact the ceramic microsystem is, its robustness will depend on different factors that should be taken into account, e.g.:

- type of sample (e.g., suspended particles should be filtered in order to avoid occlusion of the microchannels);
- a harsh environment that could cause the microsystem to deteriorate;
- frequency of analysis that could shorten the lifetime of some fluidic or electronics components;
- type of detection system (e.g., optical systems should be more robust than PVC-based ion-selective membranes).

However, most of these requirements are not specific to LTCC microsystems, but apply to most equipment aimed at the analysis of real samples and naturally they are solved.

4. Future prospects

We have demonstrated that LTCC technology is an excellent alternative for combining microfluidics and electronics in a single substrate using one fabrication methodology. However, there are still a number of issues to be tackled to fulfill the potential of LTCC technology. The miniaturization of actuators (pumps and valves) for handling fluids remains one of the most challenging fields of research, for not only LTCC but also other technologies based on other materials.

When considering the great complexity of some sample matrices and analytes, it would be very interesting to investigate further the integration of chromatographic (i.e. electrophoresis techniques), non-chromatographic pretreatment steps and new detection principles to address as many analytical applications as possible. The study of the compatibility between materials, (e.g., silicon and LTCC) would also greatly increase the possibilities for novel miniaturized analyzers.

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