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# APPLICATIONS OF THE LTCC CERAMICS IN MICROPLASMA SYSTEMS

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**Abstract**

In this paper the current status of microplasma devices and systems made in the LTCC technology is presented. The microplasma characteristics and applications are described. We discuss the properties of the LTCC materials, that are necessary for reliable operation of the sources. This material is well known for its good reliability and durability in harsh conditions. Still, only a few examples of such microplasma sources are described. Some of them have been developed by the authors and successfully used for chemical analysis and synthesis.

Keywords: microplasma, Low-Temperature Co-fired Ceramics, microsystems.



## 1. Introduction

Cold plasma is typically created in electrical discharges at reduced pressure (below 13 Pa). Plasma becomes instable at atmospheric pressure and transfers into arc. Moreover, ignition requires voltage in a range of tens of kilovolts. However, when the dimensions of plasma are smaller than single millimetres, it is possible to create stable cold plasma, so-called microplasma [1].

It is necessary to create some sort of discharge reactor to ignite and sustain microplasma. It can be done with the means of micromechanical techniques. There are several materials used in microtechnology as substrates, such as semiconductors, glasses or polymers. Not all of them can be used for fabrication of microplasma devices. The material has to be very reliable and stable at reactive environment and at high temperature and voltage. Ceramic materials fulfil these requirements [2].

In this paper we present the current status of development of microplasma devices made in the *Low-Temperature Co-fired Ceramics* (LTCC) technology. We firstly describe similarities and differences between well-known low-pressure plasma and microplasma. Special attention is given on their numerous applications, because they are the reason of increasing effort put in this relatively new field. We also discuss advantages and limitations of the LTCC. Finally, LTCC microplasma generators are presented, among them ones developed in our group.

## 2. Microplasma

Plasma is an ionized gas, which is electrically quasi-neutral and exhibits collective behaviour. Only gases with sufficient ionization degree and dimensions fulfil this definition [3–6]. When these conditions are met, plasma can interact with electromagnetic field in the similar manner as metals. Therefore, plasma can be considered as an electrical conductor and has long-range interactions.

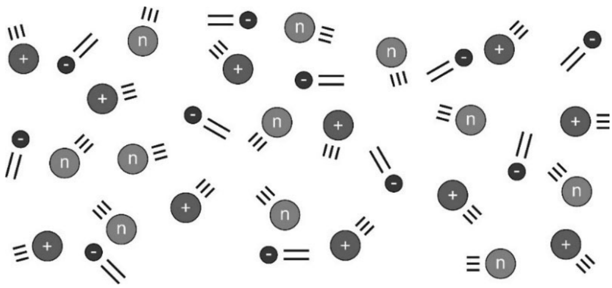

Fig. 1. Particles in plasma.

Plasma contains free electrons, ions and, in some cases, a neutral background. They have the same or different kinetic energies. These energies can be expressed by temperatures of ions $T_i$ and electrons $T_e$. If the $T_i$ and $T_e$ are equal, then plasma is referred to as a thermal (hot) one. However, it is possible to obtain plasma with ions having lower energy than electrons. Such plasma is non-thermal (cold). It is typically generated in discharges at reduced pressure.

Plasma consisting of molecules can also be used to distinguish other forms of gathering internal energy. Molecules can increase their rotational ($T_{rot}$) and vibrational ($T_{vib}$) temperature. Also, electrons in atoms can be excited to higher energy levels. This is called excitation energy $T_{exc}$ and is related to the electron energy distribution. A typical relation between the temperatures in cold plasma is [7]:

$$T_e \geq T_{exc} > T_{vib} > T_{rot} \approx T_i \approx T. \tag{1}$$

### *2.1. Definition and properties*

If at least one dimension of plasma is smaller than one millimetre [1, 8], it is referred to as microplasma, sometimes also called microdischarge. It is typically generated in strong electric field. Theoretically, the minimum dimension of micro-plasma is limited by the Debye length (approximately 0.35 μm) [3]. For the glow discharge at atmospheric pressure the smallest achieved distance between electrodes was approximately 0.5 μm [9]. For such small dimensions bigger role takes surface phenomena.

According to the Paschen rule, the breakdown voltage for a certain gas is a function of the product of pressure $P$ and distance $d$ between electrodes. The Paschen curve has its minimum at a specific value. For micro-plasma sources it corresponds to the number of particles in a range from $10^{24}$ to $10^{30}$ m$^{-3}$. Therefore, even for a low ionization degree it is possible to obtain high electron densities. As a result, collisions between electrons and neutral particles occur more often than in low-pressure plasma. That makes micro-plasma a very useful tool in chemistry. Finally, according to smaller dimensions, the role of surface phenomena in microplasma is more important than in "typical" plasma [3].

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In science and technology, plasma is most often used at reduced pressure (below 13 Pa). It is significantly different from micro-plasma. First of all, micro-plasma is of course smaller. Therefore, the treatment of some surface can require some additional processes. However, micro-plasma does not need vacuum pumps. As a result, some processes can be performed online, which can significantly improve their time efficiency. Differences appear also at the microscopic scale. For a similar frequency of the excitation source the rate of collision in micro-plasma is about 100 times higher [10].

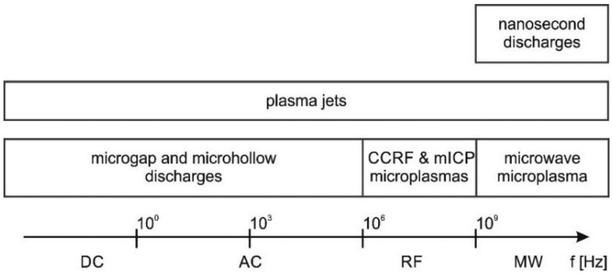


Fig. 2. Classification of micro-plasma sources (based on [11]).

### *2.2. Measurements of microplasma*

The methods of micro-plasma characterization are closely related to the type of generator. The most popular classification of micro-plasma sources is based on frequency of the excitation source [11]. *Direct Current* (DC) discharges are different from plasma generated in microwaves. The basic reason is energy transfer – with increasing frequency of electromagnetic field ions, having bigger mass than electrons, are not able to follow these changes. Therefore, their kinetic energy is much smaller than the energy of electrons. Obviously, there are other phenomena that create these differences. A separate group is constituted from *Atmospheric Pressure Plasma Jets* (APPJs), which can be designed for a certain frequency range [12].

Conventional low-pressure plasmas are often examined with the means of probes, *e.g.* Langmuir ones. These methods are impossible to apply to micro-plasmas, because they affect the generation conditions and, as a result, the properties of micro-discharge. The methods used for micro-plasma characterization can be divided into several groups, namely electrical, optical and chemical. The last group involves characterization of micro-plasma products and surface of the reactor.

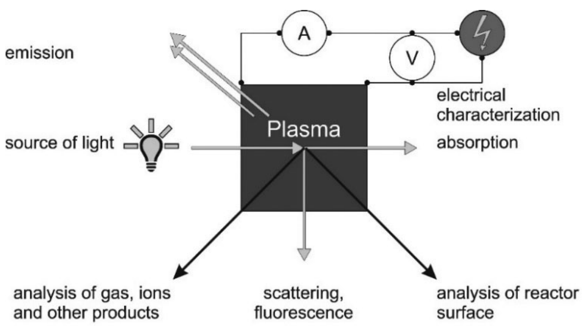


Fig. 3. Methods of micro-plasma characterization (based on [11]).

The current-voltage (electrical) characterization enables to determine power delivered to plasma and describe its general properties. For DC plasmas the slope of I–V curve shows the character of discharge (*i.e.* abnormal, normal or subnormal). For AC discharges these measurements become challenging. Voltage can be measured relatively easily using high-voltage probes, but current is usually measured either using the Rogowski coil or via HV capacitors. As frequency increases, there are higher requirements for the measurement equipment [11].

Product characterization involves chemical analysis of ions, gases and/or liquids, if the liquids are subjected to micro-plasma. Among the methods the main role plays the group of chromatographic methods. To improve their accuracy, they can be used together with spectroscopic methods, especially with a mass spectrometer for analysis of ions.

Plasma can have different impact on the surface of materials. It can be used for etching, modification and deposition. It is also valid for micro-plasma sources. The easiest method of basic inspection involves using an optical microscope. However, it is necessary to use other methods to obtain detailed results. Measurements of roughness and surface energy are relatively easy ones. More sophisticated methods are XPS, ATR-FTIR, AFM and SEM.

The most popular methods used for characterization properties of plasma itself are the optical ones. Absorbance bases on attenuation of light coming through plasma and is used to determine the amount of some specimens according to Lambert's law [13]. A different one is *Laser Induced Fluorescence* (LIF). Laser light excites atoms and molecules present in micro-plasma. Based on the fluorescence it is possible to characterize micro-plasma with a high resolution and detect non-excited specimens, such as ions or radicals [14, 15]. Finally, the easiest and most popular method is emission analysis. It can be combined with numerical simulation and give information about plethora of micro-plasma properties, such as temperatures [16].

### *2.3. Applications*

One of the most popular applications of low-pressure plasmas is deposition of different types of materials. The necessity of using vacuum systems extends time of the process. Because micro-plasma can be generated at atmospheric pressure, attempts are made to deposit materials in such conditions. The materials can be deposited either from the gas phase or from a solid, evaporated material. In both cases there are used APPJs. In the first one the *Capacitively Coupled Plasma* (CCP) sources are used, whereas in the second – *Miniaturized Inductively Coupled Plasma* (mICP) ones.

Cold atmospheric plasmas are used for surface modification of different materials. The modification can be defined as changing the surface energy or surface chemical composition. As a result, one may modify surface tension, chemical activity or mechanical properties. Local modifications using micro-plasma are applied to fabrication of microfluidic modules [17]. Because micro-plasma can be focused on a much smaller surface than the conventional one, it is possible to modify surface with a resolution of 200 µm [18].

Molecules and particles in plasma are susceptible to dissociation. Moreover, the presence of ions and radicals causes the use of plasmas in air cleaners and conditioners. Plasma removes bacteria, dust and bad smells. There are studies on using micro-plasma for dissociation of harmful nitrogen [19] and carbon compounds [20]. Products of dissociation can be also used for fuel production. For example, methanol can be used to obtain pure hydrogen [21].

Micro-plasma can be used for synthesis of nanomaterials. They have unique properties because of their size. There are reported results of synthesis of metals [22], metallic oxides [23] and organic nanostructures [24]. There are two main groups of synthesis methods. The first one involves etching and sputtering material on a substrate (the top-down method). In the second

– micro-plasma is the source of energy used for agglomeration of ions and molecules into nanostructures [22].

Probably the first field micro-plasma found its application in, was the analytical chemistry [8]. Micro-plasma gives the opportunity to use accurate analytical methods outside laboratories. The conditions are especially hard for conventional vacuum reactors. Micro-plasma sources were successfully employed in *Molecular Emission Spectroscopy* (MES), *Atomic Emission Spectroscopy* (AES), *Gas Chromatography* (GS) and *Mass Spectrometry* (MS). These methods were used to measure concentration of organic as well as non-organic compounds (especially metals) [25].

Plasma medicine involves using cold atmospheric-pressure plasma in medical applications [26]. They can be divided into direct and indirect treatments [27]. The direct treatment refers to in-vivo procedures. The first clinical research was already carried out. Micro-plasma has been used for curing wounds [26] and apoptosis of tumour cells [28]. On the other hand, the indirect treatment is applied in ex-vivo procedures. Micro-plasma can be used for sterilization of tools and solutions, for instance for E.coli disinfection [29].

## 3. LTCC technology

The thick-film technology is based on screen-printing deposition on flat, non-conductive substrates. In the conventional applications the most popular substrate is alumina ceramics ($Al_2O_3$). Layers with different electrical properties are printed on its surface. Then, they are fired at a maximum temperature of 850 – 900°C.

The LTCC technology enables to fabricate microelectronic multilayer modules. The LTCC substrates are ceramic-glass composites with screen-printed elements on layer surfaces. These substrates are relatively easy to machine before firing. There have been also developed other techniques, which enable to create LTCC microsystems.

### *3.1. High-temperature properties of LTCC*

Due to its properties, the LTCC technology is commonly used for fabrication of high-temperature electronics and other devices [30]. In comparison with ceramics (*i.e.* alumina), LTCC substrates have a much lower thermal conductivity, which can be either advantage or disadvantage, depending on application. However, for ceramics this parameter significantly changes with temperature. For alumina the thermal conductivity decreases from 21.6 W/(mK) at room temperature to 7.6 W/(mK) at 800°C. LTCC substrates are much more stable – DuPont 951 has 2.75 W/(mK) at room temperature and 2.49 W/(mK) at 500°C [2].

Another important parameter is resistivity, which also varies with temperature. Jarosław Kita *et al.* compared resistivity of LTCC and alumina substrates [2]. As might be expected, the highest resistivity had pure alumina (99.99%), but 96% alumina (the most popular one) had higher conductivity than DuPont 951. Resistivity of these substrates decreases with temperature, but even in 800°C remains at a satisfying level.

These properties were used in several devices. The first group are pressure sensors that work at 400° [31] and 600°C [32]. Another group are miniature hot plates [33]. They are used in sensors, power generators, lasers and microfluidic devices. Such devices work at up to 600°C, when the "cold end" has temperature lower than 100°C. The maximum operation temperature is limited by the presence of glass phase in LTCC.

Table 1. Properties of ceramic and LTCC substrates [30].

| | **Thermal conductivity [W/(mK)]** | **CTE [ppm/K]** | **Flexural strength [MPa]** | **Breakdown voltage [kV/mm]** |
|---|---|---|---|---|
| $Al_2O_3$ | 26–35 | 6.8–9 | 300–400 | 10–20 |
| BN | 20–60 | 0.1–6 | 20–90 | 40–200 |
| AlN | 150–180 | 4.3–6.2 | 300–350 | 14–17 |
| $Si_3N_4$ | 20–30 | 2.6–3.6 | 500–800 | 10–14 |
| DuPont 951 | 3.3 | 5.8 | 230 | > 40 |
| Ferro A6 | 2 | 7 | 170 | > 29 |

### *3.2. LTCC micro-plasma devices*

According to the above described properties, the LTCC technology has been used for fabrication of different sensors and microsystems, including *Lab On Chips* (LOCs) with optical and electrical detection. However, the LTCC materials were relatively seldom used in microplasma sources. Before the 2013 only four of them were presented in the literature [34–38].

The first example of an LTCC micro-discharge device origins from the 2001 [34]. It was an MHCD device consisting of 5 layers of DuPont 951 ceramics with the total thickness of 500 µm. The stable micro-plasma was generated in a range of pressure from 0.25 to 1 atm in the neon atmosphere. A discharge was generated at DC voltage of 137 V and 1.1 mA current. A similar geometry was used in a CCP device [35]. It worked with a voltage amplitude of 1250–1350 V at 7.4 MHz frequency. In this case the electrodes were made of different pastes (conductive and resistive). Both of the devices, according to the geometry of electrodes, had limited applications.

There are also examples of micro-plasma sources that worked at a reduced pressure. The first one was also the CPP type source [36]. It consisted of a matrix of micro-hollows, where plasma was extruded outside the discharge region. The device had 27.4 mm × 30.7 mm and the plasma region had 10 mm$^2$. A discharge was generated at a pressure of hundreds of Pa. The second one was the mICP type source excited in a microwave range (500 MHz to 1 GHz) [37]. A few types of sources were fabricated and the best performance was exhibited by coils with five turns with 1 mm pitch at 918 MHz. The main drawback of both devices was a reduced pressure of operation.

## 4. Applications of LTCC micro-plasma systems

### *4.1. Detection of selected elements*

OES methods are used for detection of selected elements. ICP is commonly used as an excitation source. However, it requires complex instrumentation and consumes a lot of power. An example is *Atmospheric Pressure Glow Discharge* (APGD) with *Electrolyte as a Liquid Cathode* (ELCAD). The principle of its operation is presented in Fig. 4.

The prototype of an APGD–ELCAD chip consisted of inlet and outlet of a discharge gas, an optical fibre groove, a discharge chamber, a microfluidic channel with inlet and outlet, and an aperture between the channel and the chamber. The channel and the aperture were designed in such a way that liquid analyte did not fill the chamber.

The chip was fabricated using DuPont 951 materials. It consisted of 18 layers. The discharge chamber was 1.2 mm high. The external electrodes were made of PdAg paste, the internal ones

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– of platinum. The multistage lamination was performed to obtain the designed geometry. Also, it was necessary to use SVM material and to modify the firing profile. The fabricated device is presented in Fig. 5.

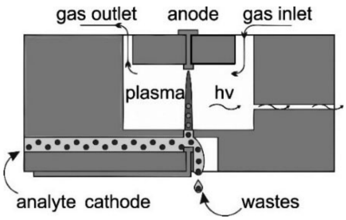


Fig. 4. The principle of operation of an APGD–ELCAD chip [38].

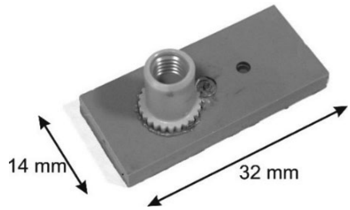


Fig. 5. An APGD–ELCAD chip.

The device was tested experimentally. A glow discharge was generated inside the chip in the helium atmosphere. The acquired spectra were typical for this discharge configuration. Also the elements added to the analyte were excited in micro-plasma, namely zinc and cadmium. Their DLs were 140 and 200 ppb, respectively. The device worked for more than one hour and the catastrophic failure occurred. It was a result of degradation of the cathode.

The device was modified in order to improve its performance [39]. The optical path was enlarged, the fluid outlet removed and the solid cathode moved out from the aperture. This chip was fabricated in a similar way as the first one. Moreover, the housing for the chip was made using 3D printing technique.

A discharge spectrum was similar to the first one. The differences were a result of changing an analyte. This time, copper ore was dissolved in nitric acid. The emission line from this element was observed. It enabled to determine the DLs. The line values were 27 ppb at 324.7 nm and 49 ppb at 327.4 nm. These values were comparable with the ones obtained in the conventional devices. In this case the anode was destroyed, as a result of mechanical stress introduced by the spring probes. Inspection of the chip interior revealed that some material was deposited in the sidewalls of the discharge chamber. It came from sputtering of the analyte.

### *4.2. Modification of water properties*

DBD generators consist of two electrodes and at least one of them is covered with a dielectric barrier. They are one of the most popular devices to generate cold atmospheric pressure plasma. It is a result of their simple construction, easy scalability and a power supply available in the market. A discharge is generated at an alternating voltage amplitude ranging from hundreds volts to hundreds of thousands volts.

An LTCC DBD generator was made of DuPont 951 tape [40]. It consisted of two electrodes covered with a dielectric, the discharge chamber between them, the inlet and outlet of working gas. The structures were fabricated in the multistage lamination process from SVM materials. The electrodes were made of PdAg paste. The fabricated structures had $17.3 \times 17.3 \times 1.28$ mm$^3$.

After connecting the electrodes to a high-voltage supply, a quasi-homogenous discharge was observed. Its spectra were recorded for different working gases, namely air, argon and nitrogen. The influence of the flow rate and the amount of oxygen on the emission intensity was examined. Despite the structures were heated up to more than 200°, no degradation was observed even after long operation of the structures.

DBD was then used to fabricate so-called *Plasma Activated Water* (PAW). For this purpose two electrodes were fabricated from LTCC ceramics and polymer housing [41]. One of the electrodes covered with a dielectric barrier had direct contact with plasma, as presented in Fig. 6. The second one was made of gold paste and put inside the water. The housing was made in 3D printing technique.

A discharge inside the device is presented in Fig. 7. The discharge was characterized for three working gases (air, helium and argon). In all cases the discharge gas was mixed with ambient air. It was indicated by the rich emission of nitrogen. The water pH and conductivity were measured before and after the plasma treatment. Every time pH decreased and conductivity increased. The biggest changes were observed for the helium mixture. Since for this gas also the emission of nitrogen was the most intense, it might be assumed that the change of water properties was caused by the synthesis of the reactive nitrogen species.

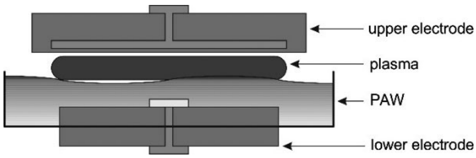


Fig. 6. Plasma activation of water with the LTCC device.

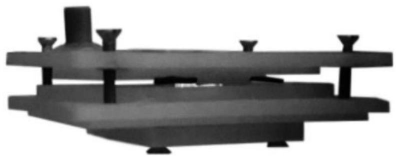

Fig. 7. The fabricated PAW device [41].

### *4.3. Synthesis of nanomaterials*

The APPJs are very popular sources of cold plasma, because the ionized gas is expanded outside the discharge region. They can be excited in a wide range of frequencies – from direct current up to microwaves. According to this the obtained micro-plasma has different properties. These jets find numerous applications, especially in treatment of thermal-sensitive materials.

An LTCC jet was obtained in MHCD configuration with AC excitement [42]. The discharge region was suspended on two arms in order to increase thermal resistance. The electrodes were made of gold and were not covered with a dielectric barrier. The micro-hollow had a 0.45 mm diameter and 0.85 mm height. After supplying a discharge, there was observed a big background noise, which could be a result of plasma thermalisation and sputtering of the electrodes. Furthermore, there was no tight gas connection and the structures were very susceptible to mechanical damages.

The modified APPJs had a solid structure and one of the electrodes was covered with a dielectric to improve micro-plasma stability. For the same purpose, the height of the micro-hollow was decreased to 100 μm. Also the housing was fabricated from thermoplastic materials for easier handling and improving gas connection. Jets were connected to HV AC supply with a modulated signal. The spectra were measured for discharges in nitrogen, helium and argon. They confirmed that the background noise was significantly decreased.

The APPJs had worked for tens of minutes without change and then micro-plasma became instable. Inspection under an optical microscope revealed that the uncovered gold electrode was etched and deposited on the dielectric barrier of the second electrode. It was confirmed by the measurements made using SEM–EDX. These changes also influenced the electrical properties of the jets measured by means of impedance spectroscopy.

It was decided to verify whether APPJ can be used for the synthesis of nanomaterials. This can be achieved in one of two ways. In the first one, a solid metal is etched and sputtered into

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the stabilizer. In the second method micro-plasma is used for reduction of ions. Because gold electrodes of jets were sputtered, both of the methods were tested.

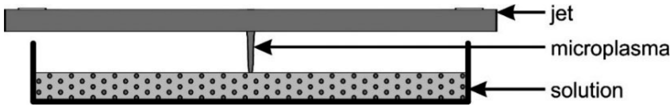


Fig. 8. The method of synthesis of nanoparticles with micro-plasma.

Micro-plasma was used to treat solutions of the stabilizer (1M solution of PEI) without and with precursors (chloro-auric acid). The jets were placed in a distance of 1 and 2 mm from the surface of the specimen. After a certain amount of plasma treatment the time specimens were characterized using UV–VIS absorption method. It appeared that the material of electrodes did not form any nanostructures. However, the plasma treatment improves the synthesis time in comparison with a reference specimen (stabilizer with precursors). Moreover, the plasma treatment improves dispersity of nanostructures, what was shown by DLS measurements.

### *4.4. Treatment of biological cells*

LTCC ceramics was also used in the field of bio-medicine [43]. The device was DBD-type with interdigital, buried electrodes (Fig. 9). A distance between them was 150 μm and a dielectric barrier was 50 μm thick. Microplasma was created in the hollows with a diameter of single micrometre. As a result of gas flow it was projected outside these hollows. Optimization of the chip geometry and supplying parameters enabled to ignite a discharge at atmospheric pressure with the power consumption in a range of single watts, and the temperature of the chip surface could be as low as 40°.

The authors also verified the impact of micro-plasma on living cells, as can be seen in Fig. 10. The experiments were carried out with fibroblasts. Different exposure times and tuning conditions significantly changed this influence. It was found that a longer exposure could be possibly used for disinfection of a surface, but the research was at a very early stage.

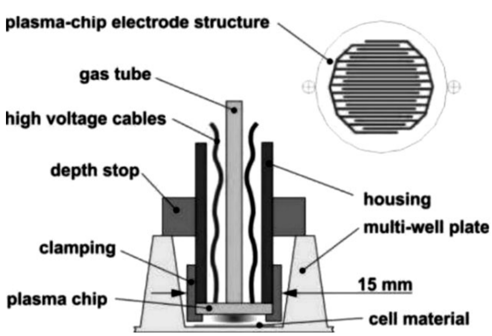


Fig. 9. The design of an LTCC chip for biomedical applications [43].

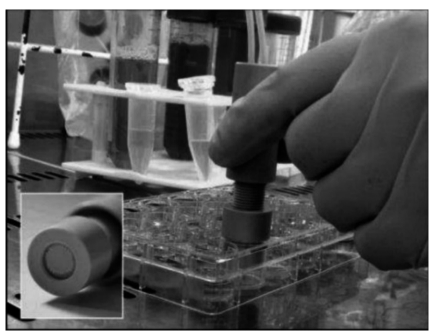

Fig. 10. An LTCC biomedical device during operation [43].

## 5. Conclusions

The paper presents atmospheric pressure generators, which can be described as plasma micro-generators. Low-pressure plasma is an ionized gas, which reacts to an external excitation in a collective way. As a result of decreasing dimensions between electrodes it is possible to generate stable plasma at atmospheric and higher pressure values. Micro-plasma sources found their application in analytical chemistry, engineering of materials, biology and medicine.

The generators can be fabricated in the LTCC technology. These materials exhibit very good electrical properties and endurance in harsh environments. Among numerous devices some of them are designed to work in high temperatures. They are used as packages of sensors, hot-plates and miniature calorimeters. Before the 2013 some LTCC micro-plasma devices were also presented, but their practical application was limited.

In the last few years the authors developed a few different constructions which were used in chemical analyses and syntheses. The elemental analysis was performed on a chip with one liquid electrode. DLs of selected elements were lower than 1 ppm. A device with DBD electrodes was used for modification of water properties. We also used APPJ for synthesis of gold nanoparticles. This paper also presents a device for the selective treatment of cell cultures, developed at the Ilmenau University of Technology.

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