

Technological challenges in manufacturing of vacuum gauge thermionic cathode using thick-film technology

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This paper focuses on the development of a technological challenges of manufacturing the planar ceramic vacuum sensor based on the principles of hot-cathode ionization in the Bayard-Alpert configuration. The goal is to simplify the technological process by utilizing planar platinum structures with gold electrical paths instead of 3-dimensional structures. Various methods were tested, including the use of carbon-based SVM (Sacrifice Volume Materials) materials, but without success. Wet-etching using potassium hydroxide on Al₂O₃ substrates showed promise results. The findings highlight the challenges and progress made in developing the thermo-emitter component of the vacuum sensor.

Keywords: *alumina, wet etching, vacuum gauge, SVM, thick-film*

1. Introduction

The field of vacuum sensors overlaps with many areas, enabling crucial applications in various industries, from aerospace to semiconductor manufacturing [1–5]. Due to a number of advantages arising from the properties of the vacuum, its potential in various areas of science and industry was very quickly recognized. Advancing market and scientific needs result in pushing the limits of the minimum achievable pressure value. However, this trend is temporarily being frozen due to limitations of the volume in which the aforementioned vacuum conditions can occur. One of the answers to this demand is the design of MEMS (micro-electromechanical system)-type vacuum sensors (gauges) [6–8]; however, despite the undoubted advantages (small size, very high accuracy and repeatability of manufacture), this technology is quite expensive. Therefore, there is a need to search for other solutions.

According to the definition of vacuum, a vacuum gauge is applied to measure the pressure lower than atmospheric pressure [9]. Depending on the

measurement methodology, all types of pressure meters (including vacuum ones) can be divided into two main sub-sections – the direct and indirect meters/gauges [10]. According to this definition, direct meters measure the force resulting from the pressure difference or the effects of this force, e.g., the deformation of elastic elements or the change in liquid level. Hence, the great advantage of this group of vacuum gauges is the lack of influence of the type of gas measured on the value of the indication. Unfortunately, the gas type is relevant for indirect vacuum gauges, where the resulting measurement value is calculated on the basis of proportional quantities (e.g., thermal conductivity of the gas, ionic current intensity) described by appropriate physical relationships, defined under strictly defined conditions, among others, the composition of the gas mixture. Thus, these meters are scaled for one gas, e.g., nitrogen, and in the case of measurement in another atmosphere, the so-called proportionality coefficients are taken into account [10–15]. A detailed classification of vacuum gauges by operating principle/method of detection is shown in Figure 1.

Our goal is to develop technology accurate for designing sensors for high and ultra-high

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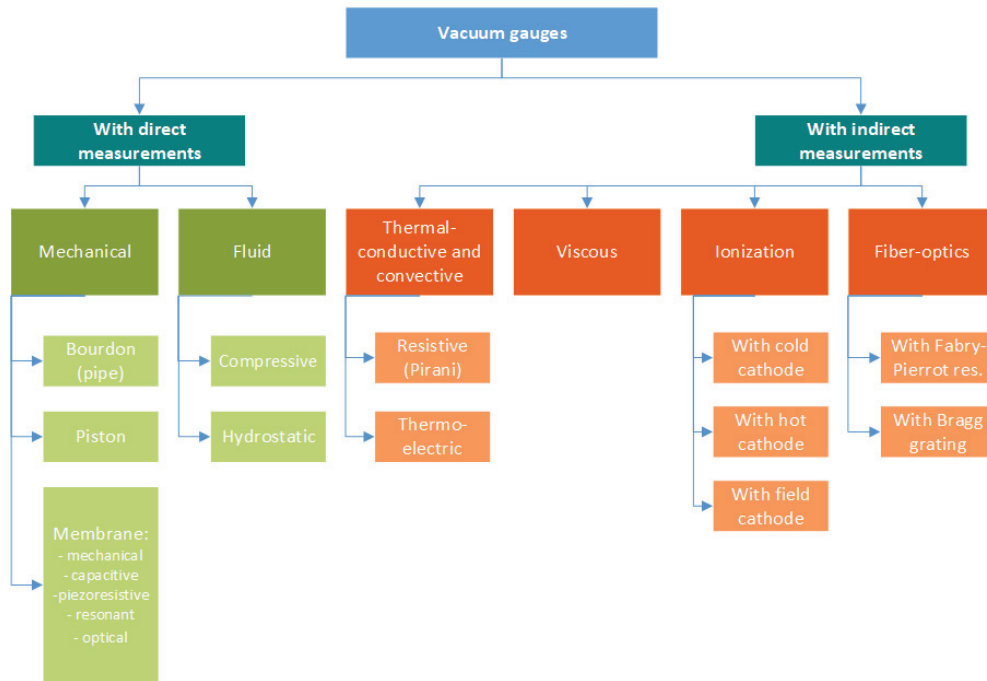


Fig. 1. Classification of vacuum gauges by operating principle/structure, based on [10–15]

vacuum. This attempt required the development of new measurement methods that corresponded to very high gas dilution. A suitable approach was “molecule counting,” which can be realized by measuring the ionic current produced by the gas ionization process and capturing particles with a non-zero electric charge. There are two groups of vacuum gauges of this type – hot cathode gauges (HCG) and cold cathode gauges (CCG), known also as Penning gauges [16–20]. An HCG operates by emitting electrons from a heated filament (cathode) into the ionization space of the gauge to ionize the gas molecules, while a CCG typically works based on the penning discharge. In order to emit a free electron from the surface of a solid to a vacuum, it is necessary to overcome the work function, i.e., to supply a sufficient amount of energy. The most commonly used hot filaments are spirals formed from materials that are difficult to melt and have a sufficiently low work function. Among them, we can list oxide-coated tungsten and iridium [21]. The role of field emitters can be performed by an array of microscopic pointed or cone-shaped tips made from molybdenum, tungsten or silicon (Spindt-type microtip field emitter arrays) [22] or

layers of carbon nanotubes (CNTs) [19]. Both of the aforementioned emitter types are adaptable, for example, in the design of an ionization vacuum gauge in the Bayard-Alpert configuration. This is a specific variation of the triode, consisting of three main components: an emission cathode (thermal or field), an electron collector and an ion collector. The aforementioned modification between the triode head and Bayard-Alpert is a change in the location of the individual components. The classical triode solution has the ion collector surrounding the other two electrodes, which is associated with high X-ray irradiation of the ion collector. Thus, the BAG (Bayard-Alpert gauge) head has a laterally located emission cathode with the ion collector surrounded by a grid that accelerates and collects electrons.

Our idea assumes that the role of the thermo-emitter would be performed by a planar platinum structure with gold electrical paths applied directly to the Al_2O_3 substrate by screen printing. The motivation for developing the planar structures of the thermo-emitter (thermionic cathode) was the simplification of the technological process in comparison to the standard LTCC (Low-Temperature

Cofired Ceramics) process that allows the creation of the 3-dimensional structures but with a higher unit cost [23–25]. On the other hand, both LTCC and thick film technology are much more economically viable than MEMS, which is also an important aspect. This article focuses on the process of developing the technology for constructing the component of the thermo-emitter. A number of methods have been tested to examine the feasibility of making a complete structure and components of a vacuum gauge in a Bayard-Alpert configuration adapted to thick film technology. Preliminary studies were half-successful in testing the feasibility of a free platinum structure on a ceramic substrate acting as a thermionic cathode.

2. Materials and Methods

The development of the planned ceramic vacuum sensor design began with preliminary studies, which consisted of the determination of the technological and performance capabilities of the various components of the sensor design. However, the main focus was on developing a thermo-emitter design in the form of a platinum spatial structure isolated from the substrate. This approach is dictated by strength limitations on the side of thermal stresses introduced by the temperature gradient between the platinum thermo-emitter and the Al_2O_3 substrate. Moreover, parameters such as conductivity and heat capacity, as well as the brittleness of the ceramic substrate, limit the ability to heat the platinum thermo-emitter to a temperature required to achieve effective thermos-emission. Therefore, a number of attempts have been made with various techniques to aid in the fabrication of thick-film spatial structures. These methods include the use of sacrificial volume materials (SVM) and the removal and release of structures during the etching process [26–29].

2.1. Sacrificial Volume Materials on the Al_2O_3 substrates

The first stage was to choose the adequate temperature profile to ensure the burnout of the SVM. For this purpose, a layer of graphite paste (4440

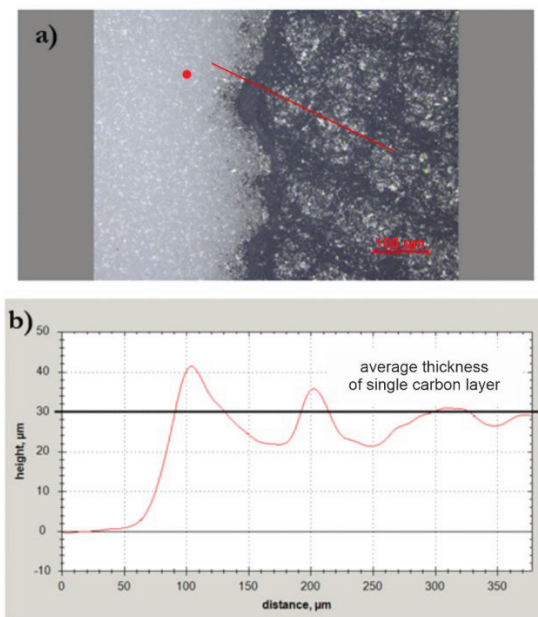


Fig. 2. Optical microscope imaging results: a) top view of the structure with the thickness testing trajectory (red line), b) thickness profile of the tested structure according to the set trajectory

from Electro-Science Laboratories) was applied to an Al_2O_3 substrate (96% Al_2O_3) using a screen-printing process (Aurel VS1520A). The printed layer was then dried for 10 minutes at 120°C in a convection oven. An optical microscope (Leica DM4500) was used to determine the quality of imprinted structures, and the results are shown in Figures 2a and 2b.

As can be seen in Figure 2a, the SVM layer was applied evenly on the substrate with the screen mesh, which is characteristic of unfired layers made by the screen-printing process. However, analyzing the thickness profile of the structure shown in Figure 2b, it can be deduced that the average thickness of a single carbon layer before firing is about $30\ \mu\text{m}$ using a screen of 325 mesh.

Different temperature profiles can be used for firing structures with carbon sacrificial layers, e.g., two-stage with a peak temperature of $720^\circ\text{C}/875^\circ\text{C}$ or three-stage de-signed for LTCC applications (450°C – firing of organic component, 700°C – complete oxidation of carbon material and sintering of ceramics, 875°C – final sintering of

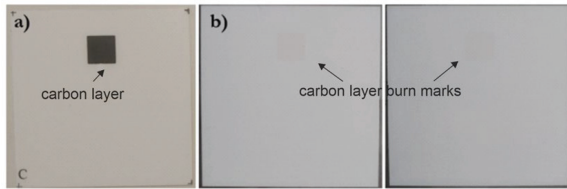


Fig. 3. Samples: a) before firing, b) after firing using different two-stage temperature profile

ceramics) [30]. After screen-printing, drying and observation, SVM structures on Al_2O_3 substrates were subjected to firing in a chamber furnace (Nabertherm HTC 03/16). A comparison of the samples before and after firing is shown in Figures 3a and 3b.

Performing a visual inspection of the samples (Fig. 3b), it can be seen that the use of both temperature profiles leads to the burning of the SVM layer without noticeable differences. The probable reason is the oxidation reaction of carbon materials, which occurs at about 700°C . However, the peak temperature in the profile used is crucial – it affects the sintering process and obtaining reproducible results/parameters of the components, while its value depends primarily on the materials and pastes used. In the case of platinum pastes, the minimum firing temperature is 850°C ; hence, it will be necessary to use a temperature profile with a peak temperature of 875°C .

2.1.1. Platinum bridge-type structures directly on Al_2O_3 substrate

The first approach used to form the platinum bridge was to fabricate the structures directly on the alundum substrate by introducing different thicknesses of the sacrificial carbon layer. The technological process began with designing the structures and producing the lithographic masks necessary for the screens. The Al_2O_3 substrates were then cleaned with acetone and dried. An $8 \times 8 \text{ mm}^2$ of SVM was applied using a screen-printing process (Aurel VS1520A), which was then dried for 10 minutes at 120°C . Repeating this process, a second SVM layer was applied to some of the samples to increase the distance between the platinum bridge and the substrate. All samples were then covered by a strip of platinum metallization

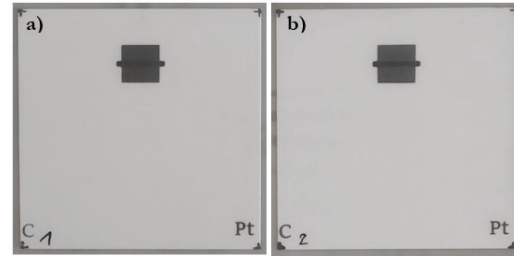


Fig. 4. Structures of platinum bridge directly on Al_2O_3 substrate before firing: a) 1 SVM layer, b) 2 SVM layers

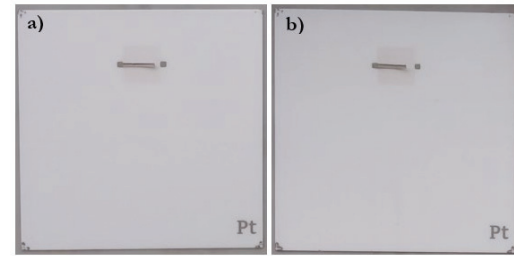


Fig. 5. Structures of platinum bridge directly on alundum substrate after firing in a chamber furnace: a) 1 SVM layer, b) 2 SVM layers

($10 \times 1 \text{ mm}^2$) applied centrally on the carbon layer by screen-printing DuPont's 9141R platinum paste. The structures made in this way were subjected to firing according to a two-stage temperature profile in both a chamber furnace (Nabertherm HTC 03/16) and a belt furnace (BTU QA 41-6-54). Figure 4a and 4b show the structures described above before firing, while Figures 5a, 5b, 6a and 6b after thermal treatment for the chamber furnace and belt furnace, respectively.

In preparing the test structures, no difficulties were noticed in their fabrication or differences in the appearance of the structures before firing (for both single and double SVM paste layers). After firing the structures, a recurring problem of platinum discontinuity at the substrate/SVM interface was noted. However, this phenomenon was independent of the thickness of the carbon layer and the used furnace. The possible reason for such results could be stresses occurring during sintering/cooling or inadequate geometry of the bridge. The only difference between the fabricated structures turned out to be a stronger C-shaped

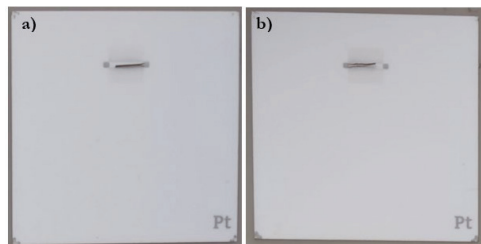


Fig. 6. Structures of platinum bridge directly on aluminum substrate after belt furnace firing: a) 1 SVM layer, b) 2 SVM layers

deformation in the cross-section when using a belt furnace. For a chamber furnace, this phenomenon was insignificant, as can be seen in Figure 5, and the reason for this behavior may have been a faster temperature drop after the sintering stage at peak temperature.

Recognizing the problem of discontinuity of platinum bridges, it was decided to investigate whether changing the geometry would improve the final result. Thus, a structure matrix design was made with varying platinum component dimensions – different metallization widths (0.5 mm, 1 mm, 1.5 mm, 2 mm) and element lengths defined by the width of the carbon sacrificial layer (0.5 mm, 1 mm, 1.5 mm, 2 mm, 2.5 mm). The process flow was identical to that for the structures in Figure 4. (same pastes and technological processes, single SVM layer). The results are shown in Figure 7a to 7c.

As can be seen in Figure 7b, the geometry of the structures has quite a large tidal effect on the final result; however, changing only this parameter does not allow for fabrication separated from the substrate and electrically continuous platinum bridges – some of the elements broke again (mainly wide and short bridges), and some collapsed and sintered on the substrate (narrow bridges). Thus, further attempts to make bilaterally supported platinum structures printed directly onto the substrate and carbon layer were abandoned.

2.1.2. Platinum bridge structures on ceramic supports

Observing platinum structures made directly on the SVM layer and Al_2O_3 substrate, a recurring

problem of discontinuous platinum bridges at the substrate/SVM layer interface was noted. The location of this defect indicated that the problem was stresses within the level difference between the layers (SVM printing on the substrate). The fabrication of two parallel-oriented ceramic supports between which a sacrificial carbon layer was applied enabled the printing of a platinum structure on a relatively flat surface within the location of the pre-existing defects. The fabrication of the test structures began with a design – a matrix of elements with different widths of the carbon layer and platinum metallization defining the length of the bridge (0.2 mm, 0.5 mm, 1 mm) and its width (0.2 mm, 0.5 mm, 1 mm, 1.5 mm) successively was designed.

Firstly, the masks needed to reproduce the patterns on the screens were made, the Al_2O_3 substrates were prepared, and the SVM layer was screen-printed. After a standard drying process, ceramic structures were printed using ceramic-glass paste (LTCC 502K08 ceramic powder from Heraeus, 10% by weight of organic carrier in the form of ethyl cellulose from Sigma Aldrich, 9180 solvent from DuPoint, as in [31]). After another drying process, some of the samples were laminated (temperature 70°C , pressure 20 MPa, time 10 minutes). A platinum layer (9141R from DuPoint) was printed on prepared structures and subjected to a firing process in a chamber furnace (Nabertherm HTC 03/16) using a standard two-stage temperature profile with a peak temperature of 875°C . The proposed solution is a modification of the process for making a matrix strip electrode using LTCC technology described in [31]. The schematic design of the structures and the fired structures are presented in Figure 8a and 8b.

The use of ceramic supports and the restriction of faults within the suspension site of the structure, unfortunately, did not allow us to obtain released platinum bridges. From Figure 8b it can be seen that there is still the problem of discontinuity of platinum structures – most often in the middle of the bridge length, and this effect is more pronounced for structures not subjected to the lamination process. In addition, with ceramic supports, there was a concern with the porosity of

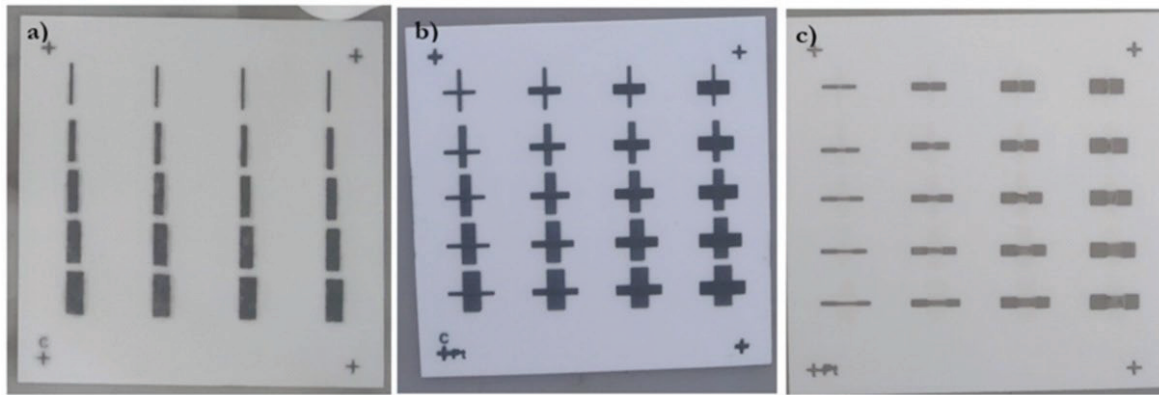


Fig. 7. Matrix of platinum bridges with different geometries: a) after application of carbon layer, b) after application of platinum layer, c) after firing

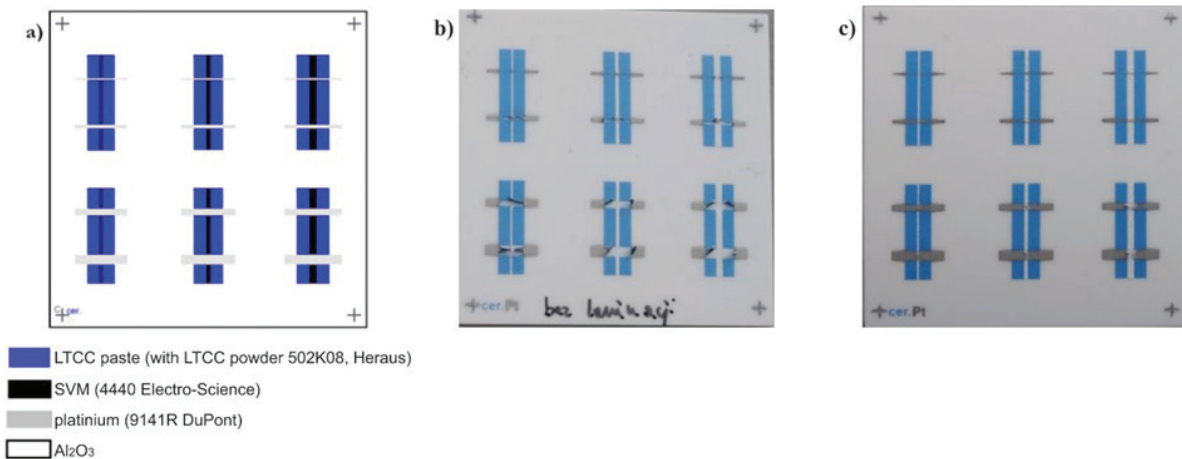


Fig. 8. Platinum bridge structures on ceramic supports: a) design pattern, b) fired structures without the lamination process, c) fired structures with the lamination process before firing

the platinum electrode on the surface of the sintered LTCC ceramic, which is not visible for screen printing on the surface of the alundum substrate. Thus, further attempts have also been abandoned.

2.1.3. Structures supported on one side – the form of a hanging platinum beam

The concept of unilaterally supported structures intended to form hanging platinum beams emerged with the idea of reducing the stresses that cause cracking and rupture of the previously described bridge structures. At the same time, different forms and geometric dimensions of platinum structures (U-shaped, rectangular and rectangular with holes) were proposed to test the feasibility. At

the beginning, gold electrodes (DuPont 5742 gold paste), SVM material and platinum metallization were applied in subsequent screen-printing and drying processes. The entire work was finished with a firing process in a belt furnace (BTU QA 41-6-54), where the maximum temperature reached 850°C at a belt speed of 1 inch/min. The exemplary results are shown in Figure 9a to 9c.

As can be seen in Figure 9c, the resulting structures after firing were incomplete – a partial absence of platinum metallization causing electrical discontinuity. Such a result was mainly influenced by problems in printing platinum paste onto carbon paste due to its high porosity and susceptibility to tearing in the screen-printing process.

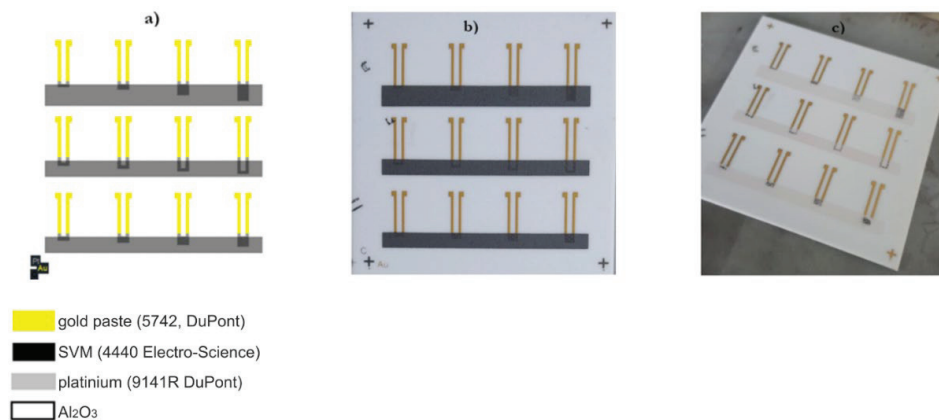


Fig. 9. One-sided structures: a) design pattern, b) unfired structures, c) fired structures

2.2. LTCC with SVM

Due to the possibility of forming single layers and the ease of fabricating 3D structures, it was also decided to attempt the fabrication of a platinum bridge using LTCC technology with SVM materials. To achieve this, a design of 3D ceramic layered structure with a matrix of cavities of different dimensions (width, length and depth) was made to explore the possibility of making hanging bridges. Then, LTCC green tape (254 μm thickness, DuPont 951PX) was formed using a laser system (355 nm Nd:YAG, LPKF ProtoLaser U). The ceramic spatial structure was assembled with successive layers (four or six, depending on the assumed thickness of the structure) relative to the positioning marks. The resulting cavities were filled with carbon film (ESL 49000, as SVM) formed with the LPKF laser system. The prepared samples were then laminated using an isostatic press (temperature 70°C, pressure 20 MPa, time 10 min.). Then, a platinum layer (Pt ESL 5570 and DuPont 9141 pastes for comparison) was applied to the filled ceramic cavities by screen printing (Aurel VS1520) and dried at 120°C for 10 minutes. Finally, the structures were subjected to cofiring in a chamber furnace (Nabertherm HTC 03/16) using a standard three-stage profile with a peak temperature of 875°C. The results are shown in Figure 10.

Comparing the results presented in Figure 10b, none of the geometrical and material configurations allowed us to obtain suspended platinum

bridges. For some of the structures (narrow and long bridges), bilateral rupture and collapse of the platinum structure can be observed. Another important remark is that with the shortening and widening of the platinum metallization, the structure better maintained the bridged form but without electrical continuity. The most significant factor causing defects in this case is the burning of the SVM layer before the process of complete sintering of the metallic grains of the platinum paste, as a result of which the structures lose support, sink, break and sinter inside the fabricated cavity.

2.3. Platinum-gold structures on Al_2O_3 substrates released by wet etching process

Dry etching and wet etching are the basic processes for forming spatial structures in microelectronics and MEMS technology [32–37]. In particular, the etching of silicon, silicon oxide or glasses is widely developed. The process of etching aluminum substrates is not a widely used technological process, but it was proposed as early as the 1970s. – At the time, the goal was to increase adhesion between the copper layer and the ceramic by increasing the porosity of the Al_2O_3 substrate [36]. In the wet etching process of Al_2O_3 , aqueous acid solutions (sulfuric or phosphoric), hydrogen fluoride, potassium hydroxide or sodium hydroxide (in molten form or aqueous solution) stand out as reactants. The basic parameters in any etching process are solution concentration, temperature and time.

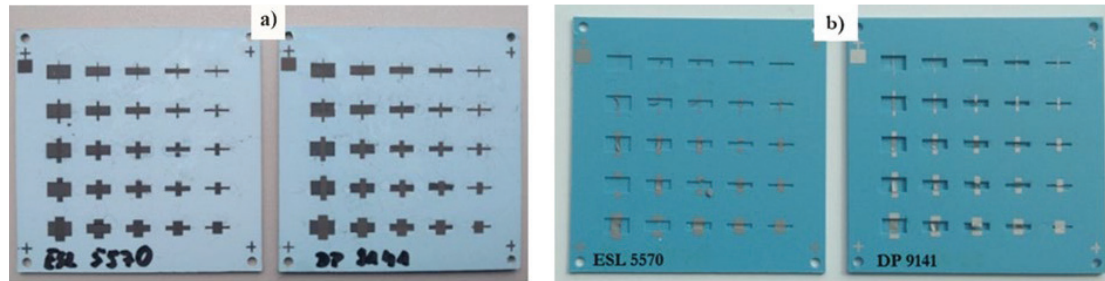


Fig. 10. LTCC platinum bridges: a) after screen printing of the platinum layer, b) after the firing process

After unsuccessful attempts to use carbon SVMs, it was decided to make planar test structures directly on alundum substrates and then subject them to wet etching. To speed up the work, the design of the structures and the technological process are proposed in subsection 2.1.3. (Structures supported on one side – the form of a hanging platinum beam) were used with the omission of the carbon SVM layer. After firing, the obtained sample arrays were divided into individual structures by scratching (using the LPKF laser system) and breaking the substrate. When selecting the etching substance, it was necessary to take into account the dissolution ability of Al_2O_3 and the lack of reactivity with platinum and gold. The use of molten potassium hydroxide KOH met the aforementioned requirements.

In the first step, a reference test of Al_2O_3 substrate etching without test structures was performed to determine the efficiency and speed of the process. For this purpose, clear KOH (potassium hydroxide from Chempur) was poured into a heated vessel, brought to the melting point (about 380°C [38]), and then the sample was left for 30 minutes. After this time, the structure was removed, cooled and rinsed in water. The effect of the etching was a loss of sample thickness of $15\ \mu\text{m}$ per side of the substrate (measured with a Mitutoyo IP65 COOLANT PROOF digital micrometer with a measurement accuracy of $1\ \mu\text{m}$), resulting in an etching rate of $30\ \mu\text{m/h}$. In the next step, several similar substrates were etched for different times (5, 10, 15 and 30 minutes) to determine the effect of the etching process on surface morphology. Imaging was performed with a scanning electron microscope (SEM) using an Everhard-Thornley Detector

(ETD). Measurement graphics for samples with different etching times obtained on the FEI Helios Nanolab 600i SEM/Ga-FIB microscope are shown in Figure 11.

According to Figure 11a we can see that alundum substrates, after the fabrication process, have a compact structure of interconnected unformed grains. However, as the etching process is prolonged (Fig. 11b–11e), individual grains with polyhedral structures are isolated, forming a near-surface porous layer. Thus, in a general way, it can be concluded that the etching of Al_2O_3 substrates in molten KOH causes a loss of material volume, isolation of grain structure, and an overall increase in surface expansion. The thesis of increased surface roughness of alundum substrates due to the etching process was further confirmed by measuring the roughness profile (Fig. 12) using a confocal microscope (LEXT 3D Measuring Laser Microscope OLS5000-SAF).

After the reference test, the platinum-gold structures were subjected to selective etching (partial immersion of the sample) for different durations (5, 10, 15, 20 and 30 minutes) in order to find the optimal process duration necessary to release (delaminate) the platinum component. After observing the etched structures, it was found that too short a process has little effect on the final result, while too long can cause complete destruction of the structures. The most satisfactory results were obtained after etching for about 20–25 minutes, but among several samples etched during this time, only a few platinum structures were released correctly and retained electrical continuity – such an effect indicates the difficulty of the process and its low reproducibility. Both the

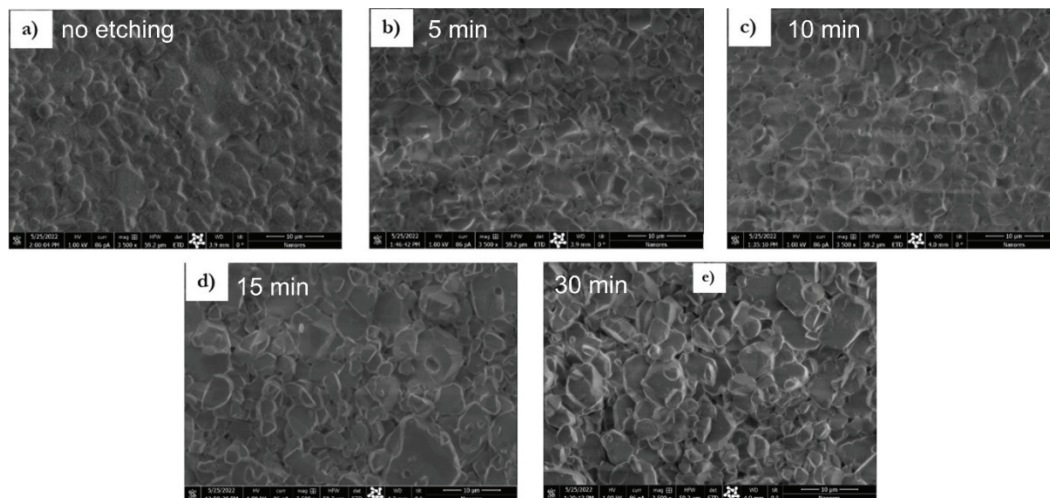


Fig. 11. Surface characterization using SEM/Ga-FIB of FEI Helios Nanolab 600i (operating voltage and current of 1 kV and 86 μ A, respectively) for Al_2O_3 samples: a) without etching and after etching for: b) 5 min, c) 10 min, d) 15 min, e) 30 min

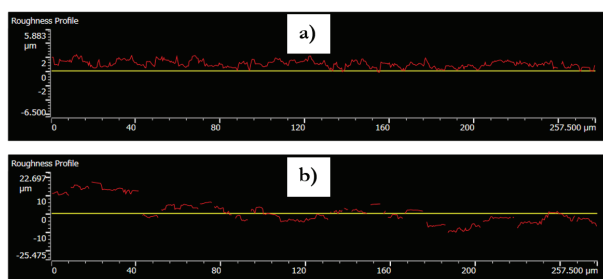


Fig. 12. Surface roughness profile of alundum substrates: a) before etching, b) after etching in KOH for 30 min and 380 °C

satisfactory structure and the failed one (the result of too long an etching process in superheated KOH) were photographed and shown in Figure 13.

3. Results and discussion

Numerous trials and preliminary studies conducted by the authors have shown the difficulty of fabricating platinum spatial structures released/separated from the alundum (or LTCC) substrate. The biggest issue in this case is the limited resistance of the alundum substrate to the thermal stresses introduced during the heating of the platinum electrode. Therefore, it was decided

to perform thermal tests to determine the limiting temperature that causes the destruction of the metal-ceramic structure. Temperature measurements were carried out on a purpose-built test stand consisting of a holder holding the test sample, a stand with a mounted thermal imaging camera (ThermoVision™ A40M from Flir Systems), a silicon safety shutter, a closed chamber isolating the measurement system (constant temperature and humidity), a computer with camera software, a laboratory power supply, and an ammeter and voltmeter. An overview schematic of the measuring station is shown in Figure 14.

Due to the desire to measure the structures from the lower limit of the measuring range of the camera (100°C) to the moment of catastrophic damage to the platinum planar structures (substrate rupture), it is necessary to use a special silicon shutter to protect the lens of the expensive measuring apparatus. However, the use of such an aperture introduces major changes in the measurement conditions – an object with a different emissivity coefficient and a certain level of absorption and scattering of infrared radiation will be introduced between the test sample and the lens. These conditions, therefore, require the determination of compensation parameters and the so-called calibration curve that tells how the aperture affects changes

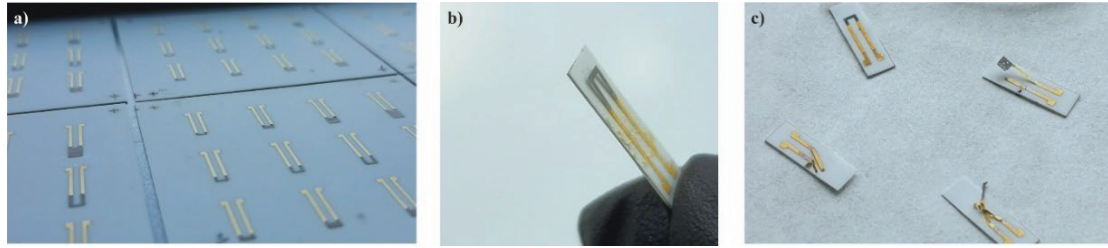


Fig. 13. Al_2O_3 wet etching process in KOH: a) structures before etching, b) acceptable result, c) unacceptable result

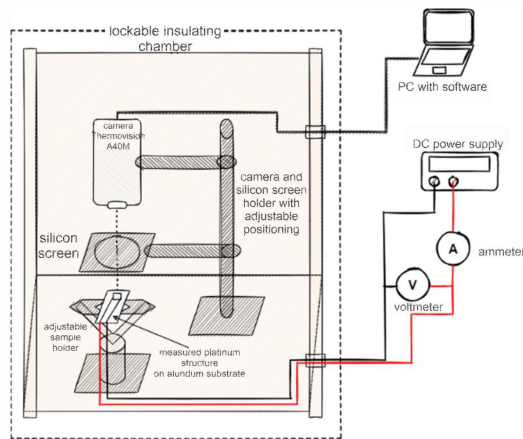


Fig. 14. Schematic diagram of the bench for thermal measurements of a platinum thermionic cathode

in measurement results. The determination of the calibration parameters began with measurements of the T temperature of the unshielded structure in the safe temperature range (up to about 400°C) for several operating points (delivered electrical power). The same operating points were then reproduced, and the T_{Si} temperature of the structure was measured using the silicon shutter. To determine the level of loss, the T_{Si}/T ratio was calculated, and all the obtained data was statistically processed using OriginPro 2018 software, determining the arithmetic mean of the sample and its average standard deviation. Such step goal was to determine the silicon shutter impact on the thermal signal between the object and camera sensors because of silicon absorption for infrared radiation. As a result, measurements through the silicon shutter were taken into account. According to the catalog note of the measuring device, the measurement sensitivity is

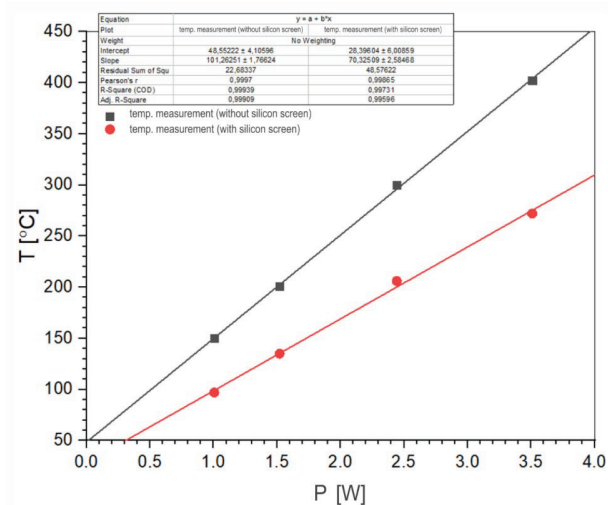


Fig. 15. Calibration curves of the measurement station using a thermal imaging camera

0.08°C (for an ambient temperature of 25°C), while the accuracy of the readings is between 2% and 3% of the value obtained during the measurement (for an ambient temperature in the range of 5°C – 45°C). It is also worth mentioning that for all thermal measurements, constant measurement conditions were maintained, i.e., temperature of 21.5°C and humidity of 35% for reflected temperature determination, which, in the end, leads to proper thermal imaging calibration. Analyzed heater surface emissivity was determined experimentally also using an IR camera (in relation to antiglare layer with well-known emissivity). The obtained temperature and parameter values are shown in Table 1, where the calibration curves are presented in Figure 15.

Based on Table 1, it can be seen that the use of a silicon shutter reduces the value of the determined temperature by about 33% on average.

Table 1. Calibration measurements for silicon shutter bench

Supply current I [A]	Supply voltage U [V]	Power P [W]	Structure temp. without shutter T [°C]	Structure temp. with shutter T _{Si} [°C]	Loss rate T _{Si} /T [%]
0.33	3.05	1.0065	150	97	64.7
0.386	3.94	1.52084	201	135	67.2
0.45	5.43	2.4435	300	206	68.7
0.5	7.02	3.51	402	272	67.1
				Mean	66.925
				Standard deviation	1.654

However, referring to the characteristics determining the effect of the use of the silicon shutter on the measurement (Fig. 15), it is possible to note the linear dependence of the temperature of the tested structure as a function of the supplied electrical power over the entire considered range (the accuracy of linearization at the level of 99.97% – Pearson’s coefficient r). Second, the different levels of the slope of the characteristics determine the change in the calibration factor depending on the operating point of the system. These conclusions are significant for subsequent thermal measurements because the assumption of a constant loss factor of 66.925% will have an error at an average level adequate to the standard deviation (1.654%).

Having determined the calibration factor for silicon shutter measurements, we proceeded with thermal measurements of a planar platinum thermo-emitter. Two heating modes were tested – with slow and fast temperature rise (approximately 2°C/s and 10°C/s, respectively). Such an attitude was used to determine the impact of the heating speed on damage for samples made by multi-material layer structures – substrate materials (ceramics) and heater (metal). The results of the measurements were the temperature of the structure estimated from the readings when measured through the silicon shutter for several operating points. All the results obtained are shown in Table 2. The temperature dependencies as a function of the applied electrical power were also plotted on their basis (Fig. 16).

From Table 2, it can be easily seen that the slow heating temperature rise of the platinum structure is more efficient and improves the achieved temperature range by up to 200°C compared to the

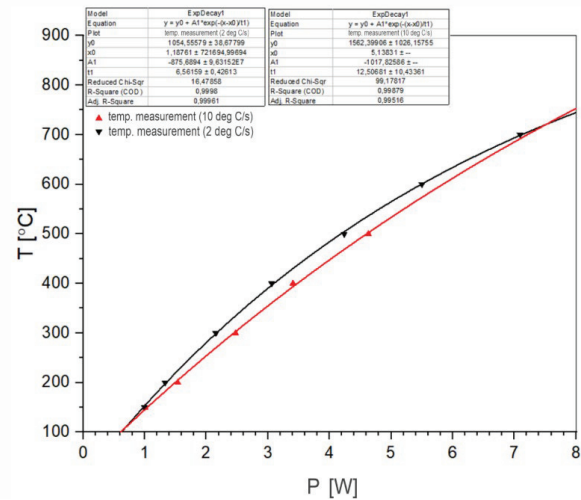


Fig. 16. Temperature dependence of platinum planar structures as a function of applied electrical power

fast temperature rise. For the same temperatures, changes in operating points (electrical power) were also noted, which is due to slight structural differences in the samples studied. Based on the curves shown in Figure 16, it can be determined that the nature of temperature changes as a function of electrical power is exponential, with a tendency to converge to a certain temperature value. The minimum measurable temperature in the presented graph is only due to the temperature range of the thermal imaging camera. In addition, heating the proposed structures to a value of several hundred degrees Celsius requires a large power supply of several watts. Summarizing the measurement part, it can be concluded that the planar platinum structures could not act as an electron emitter in the sensor design, the reason being the failure of the elements (cracking of the alundum substrate) well

Table 2. Results of thermal measurements for planar platinum structures

Supply current I [A]	Supply voltage U [V]	Power P [W]	Structure temp. Without shutter T [°C]	Structure temp. with shutter T_{Si} [°C] = $T \cdot 0.66925$
<i>Measurement with rapid temperature rise</i>				
0.33	3.05	1.01	150	100.39
0.39	3.94	1.54	200	133.85
0.46	5.43	2.48	300	200.78
0.50	6.83	3.41	400	267.70
0.55	8.42	4.63	500	334.63
<i>Measurement with slow temperature rise</i>				
0.35	2.847	0.99645	150	100.39
0.39	3.44	1.33	200	133.85
0.46	4.73	2.15	300	200.78
0.51	6.02	3.06	400	267.70
0.56	7.52	4.24	500	334.63
0.611	9.00	5.50	600	401.55
0.66	10.70	7.09	700	468.48

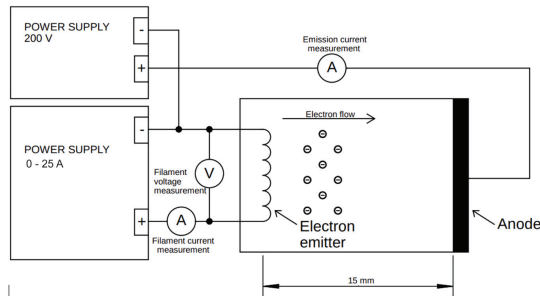


Fig. 17. Experimental setup (emission performance)

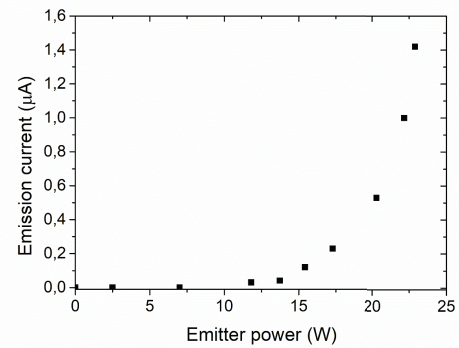


Fig. 18. Emission current vs thermionic cathode power

below the value of 1000°C, which is the minimum temperature necessary to initiate thermo-emission. Thus, the release of the platinum component is crucial for conducting further research.

In the final phase, initial tests were conducted to assess the emission performance of the platinum structure on Al_2O_3 , which was created through wet etching in KOH. The setup used for experimentation is depicted in Figure 17. The structure under examination was placed within a vacuum chamber, secured by a standard CF50 flange with a D-Sub socket, and subjected to current ranging from 0 to 25 A. Another power supply was utilized to apply a voltage of +200V to the anode concerning the emitter potential. A Keithley 2450 Source Measure Unit (SMU) served both to supply bias voltage and to measure current. Throughout the experiment, the vacuum level was

maintained at 10^{-5} Pa. The alteration in emission current concerning the power applied to the emitter was recorded, as illustrated in Figure 18. Approximately 30 nA of emission current was attained at 11.8 W, increasing to approximately 1.42 μA at around 22.9 W. However, applying higher power led to damage to the platinum structure.

4. Conclusions

The development of innovative sensing solutions and the fabrication of device prototypes often involve numerous adversities or multiple trials. In the research presented by the authors of this paper, a number of methods were tested, suggesting the feasibility of making a complete structure

and components of a vacuum gauge in a Bayard-Alpert configuration adapted to thick-film technology. Preliminary studies were performed to test the feasibility of a released platinum structure on an alundum/LTCC substrate acting as a thermoemitter were half-successful. Numerous negative results were achieved, eliminating a number of methods using carbon SVMs. Promising results were obtained only for the method of wet etching of Al_2O_3 in molten KOH, where it would be possible to make free-standing platinum structures after refining the process. On the basis of thermal studies, the possibility of using a planar platinum structure directly on an alundum substrate was also eliminated. This thesis was confirmed by the insufficient mechanical strength of the substrate to withstand the temperature gradients generated when heating the platinum layer in a temperature range that does not allow electron thermo-emission. The emission capabilities of a free-standing platinum structure on Al_2O_3 produced through wet etching in KOH have demonstrated promise as a thermionic cathode for BAG. Initial tests conducted to assess the emission performance of the platinum structure on Al_2O_3 , which was created through wet etching in KOH, have shown a maximum emission current of $1.42 \mu\text{A}$ at around 22.9 W. Nevertheless, further experiments are required, encompassing factors such as emission current density, power usage, stability, and more. These experiments are slated for future research endeavors.

Based on the obtained results, several ideas can be proposed to continue the research and develop the presented vacuum gauge concept. The first idea would be to refine the technology for releasing metallic structures on alundum substrates by wet etching in molten KOH. Another option for releasing structures could be to use a high-temperature glaze as a sacrificial material removed by wet etching in a suitable reactant. It would also be important to determine the optimal yet smallest possible dimensions of the vacuum gauge, taking into account the results obtained and technological limitations. The deficit of integrated vacuum gauges for a wide range of pressures also inspires the integration of different sensing concepts within a single design (e.g., Pirani + Bayard-Alpert design).

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