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NON-SILICON MEMS PLATFORMS FOR GAS SENSORS

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Abstract

The target of this work is the demonstration and optimization of approaches able to provide non-silicon MEMS platforms for the chemical sensor applied under harsh environmental conditions and, on the other hand, to provide a platform stable at high temperature, which can be used for the deposition of refractory gas sensing materials, for example, oxides of gallium, zirconium, or hafnium. Non-silicon materials, which can be used for this application are aluminium oxide, yttria stabilized zirconia and thin boron-silicate glass. It was demonstrated that thin ceramic films made of these materials can withstand annealing temperature up to 1000^{0}C, MEMS sensor based on these films consumes < 70 mW at continuous heating at 450^{0}C and ~ 1 mW in pulsing heating operation mode. Ceramic MEMS show higher stability at high temperature compared to silicon based MEMS, whereas power consumption of both types of devices is comparable.

Introduction

Analysis of the situation with chemical and physical sensors shows that most efforts of researchers and technologists are focused on the development of devises for normal exploitation conditions. However, very often it is necessary to monitor harsh environment at high temperature, high humidity, in aggressive atmosphere. This situation is typical of many resent industrial processes, which are characterized by the concentration of energy production (traditional and nuclear power stations, heating systems with control of combustion processes, etc.). It is met also in oil and natural gas production, monitoring of pipelines, production of chemical products, oil refinery and petrochemical industry, control of combustion processes and in many other fields of human activity. All these processes require adequate development of systems for the monitoring of technology and environment under harsh conditions.

Another, much more seldom, but very important application of such systems is the monitoring of environment and technological processes not only at normal exploitation, but also during accidents. The consequences of such accidents can be very dangerous, and industrial catastrophes of last years, for example nuclear power station of Fukushima, accident in BP platform in Mexico Gulf, etc. have demonstrated this. These accidents have shown that after the catastrophe, when any information about its development is extremely important, existing sensor systems were destroyed as well.

Recently, sensors and systems based on these sensors able to operate under harsh conditions (desirably at ambient temperature up to 250 ^{0}C and humidity up to 100 %) for long enough time (at least, for several days), are not available. In addition, silicon based MEMS are not stable at high operation temperature because of poor adhesion of Pt to SiO_2 and Si_3N_4, hydrolysis of Si_3N_4, and internal strains in SiO_2/Si_3N_4 multilayer membrane. As a result, actual information about concentration of hydrogen, toxic gases, humidity pressure, etc. is not accessible for immergence service, and, consequently, an accident develops following the most dangerous and even tragic scenario.

Such application of sensors requires very low power consumption of the device, because under such conditions, the power supply is very often interrupted, and the instrument must operate autonomously. Therefore, the use of MEMS technology enabling significant decrease in power consumption of whole device is necessary.

These reasons caused our interest to the development of MEMS sensors able to operate under harsh exploitation conditions and to wireless monitoring systems based on such sensors.

The advantages of ceramic MEMS technology are well pronounced also at the application of sensors at normal operation conditions. It is well known that one of most important problems met in the application of metal oxide semiconductor gas sensors is insufficient selectivity of such devices. The selectivity can be improved significantly by the application of temperature modulation of sensor [1]. However, mass production silicon MEMS technology (for example, [2, 3]) existing now, does not permit, generally, the heating of the sensing layer at temperature higher than approximately 300 – 350^0C in operation mode and does not enable technological heating at high temperature necessary for the stabilization of the properties of metal oxide sensing layer (700 – 800^0C typically). In some recent works, for example, in [4 – 6], there is a communication about the possibility to fabricate high-temperature microheaters operating at 450^0C [4] and even at 650^0C [5, 6]. The main question remaining in these papers is long-term stability of platinum heater formed on SiO_2/Si_3N_4 membrane.

The problems met at the application of silicon based MEMS microhotplates of gas sensors can be solved by the application of non-silicon materials and, in particular, ceramic MEMS (Fig. 1). The membrane of these MEMS can be made by spark electrolyte oxidation of aluminum [7]. The fabrication process and results on gas sensitivity of these MEMS devices were described in [8]. The maximum temperature of technological annealing of this microhotplate is equal to 800^0C, maximum operation temperature reaches 550^0C, the power consumption at working temperature equal to 450^0C (this is the optimum for the detection of methane concentrations) is of about 70 mW. The MEMS structure withstands up to 5 millions of switch on – off cycles.

Heating power consumption of alumina based sensor is similar to the results presented in [3] for MEMS sensors based on silicon technology. For example, MiCS-5524 CO sensor [3] consumes 71 – 81 mW at working temperature, which is equal, probably, to about 300^0C.

However, in spite of important results obtained with alumina membranes made by spark electrolyte oxidation of aluminum, high porosity of such Al_2O_3 prevents wire bonding of Pt elements of the MEMS platform. Another disadvantage of these films is the roughness of the surface relief leading to the necessity of the deposition of relatively thick platinum heater lines (~ 1 micron) by magnetron sputtering process. In addition, very porous membrane fabricated by spark electrolyte oxidation of aluminum is not strong enough under shock conditions.

First attempt of the manufacturing of MEMS device based on anodic alumina was presented in [9, 10]. These sensors are of thermocatalytic (calorimetric) type. The sensor described in [9] has a shape of suspended horseshoe fabricated by laser cutting of alumina. The thickness of this alumina is of about 30 microns and the dimension is of about 200 x 300 microns. Thermal efficiency of the heater is of 50 μW/K; therefore, the heating up to 450^0C requires the power of about 23 mW.

A similar device, a thermocatalytic sensor based on anodic alumina substrate, was described in [11]. The size of the microhotplate of this sensor is equal to about 200 x 200 microns. The application of pulsing heating leads to a decrease in everage power consumption of the thermocatalytic sensor from 35 mW typical of traditional platinum wire sensor operating at constant temperature to about 1 – 2 mW.

Semiconductor gas sensors based on anodic alumina substrates were discussed in [12]. The application of pulsing heating enables a decrease in everage power consumption down to about 2 mW.

We discuss in this paper the alternatives for the ceramic MEMS technology: the application of annealed anodic aluminum oxide, yttria stabilized zirconia, and thin boron-silicate glass.

Experiment and Results

In our experiments, we used three types of thin non-silicon membranes: (1) alumina membranes manufactured by anodic oxidation of aluminum foil, (2) membranes made of nanoparticles of yttria stabilized zirconia, and (3) thin foils made of boron silicate glass.

Anodic alumina films

Thin membranes made of nanoporous alumina were employed in ceramic MEMS devices. The membranes were fabricated by anodic oxidation (anodizing) of aluminum foil in acidic electrolyte, following generally the way described in a previous work [13]. Anodic alumina (AA) membranes must remain flat after annealing at temperature equal to, at least, 800^0C. Usual problem met at the fabrication of AA membranes is their mechanical instability resulting in deformation or even scrolling of initially flat AA membranes during a high-temperature annealing. This may be due to structural and compositional changes in AA membranes at temperatures exceeding ~600^0C. This disadvantage limited the application of AA membranes in thermocatalytic and semiconductor sensors annealed usually at ~ 700 – 900^0C; this annealing is necessary for the adequate formation of sensing layer. In addition, in some applications, high-temperature materials, for example gallium oxide, are used as a sensing layer [14]. This material requires sensor operation temperature exceeding 600^0C.

To solve the problem of membrane instability at high temperature, we developed a high-speed constant-current anodizing process and applied this process to samples prepared from ordinary aluminum foil used for food wrappers, available at supermarkets as low-price aluminum foil rolls. The typical conditions of the anodic process were: the current density of about 60 - 70 mA cm^{-2} and the formation voltage ranging 45-60 V, while electrolyte temperature was maintained at 15^0C as constant as possible, preferably within $\pm 2^0$C.

Before the application as a substrate for metal-oxide gas sensors, AA membrane must be annealed at temperatures higher than 1000^0C. Such annealing is necessary because the working temperature of a sensor may reach 600^0C and more, and the temperature for forming stable sensing layers is usually 700–900^0C. We found out that a critical temperature for annealing the AA membranes ranges from 600 to 650^0C. Within this temperature interval, initially amorphous alumina, containing boehmite (AlOOH) phase, loses structurally bound water, and this process leads to deformation of the initially flat sample. Moreover, crystallization of alumina begins approximately at the same temperature. Through numerous experiments, we have revealed that, if a load is placed over the membrane and the temperature ramp is set to about 100^0C/hour, these conditions allow us to keep 48 x 60 mm membrane as flat as it was before the annealing (Fig. 3). After annealing, AA membranes contain crystalline phases of non-stoichiometric $\gamma\text{–}Al_{2,14}O_{3,3}$ and $\gamma\text{–}Al_{2,43}O_{3,64}$. The annealing at higher temperatures (>1250^0C) leads to full crystallization of alumina with the formation of $\alpha\text{-}Al_2O_3$ phase (corundum), which makes, however, the membrane very fragile.

Cross sectional and surface SEM views of a 16-micron thick AA membrane prepared as described above obtained in a JEOL JSM 6500 electron microscope are shown in Fig. 2. The mean diameter of the pore outlets at the membrane surface is estimated to be 20 nm. Such small pores are not expected to impact the properties of gas sensors fabricated over the membranes because the millimeter-sized sensor elements (heaters, areas of sensing layers, contacts to the sensing layers, etc.) are incomparably bigger. SEM analysis of numerous membranes confirmed high quality of porous alumina within the membranes and at their both surfaces. In all experiments, we used aluminum foil “as bought”, i.e. without any pretreatment such as electropolishing or even chemical etch-cleaning. Despite the impure, low-cost aluminum and the absence of usual pretreatments, the quality of the alumina membranes appeared to be satisfactory for the purpose of their application as gas sensor substrates.

A very important advantage of MEMS devices made of thin AA membrane compared to SiO_2/S_3N_4 MEMS consists in the possibility to apply laser machining of alumina films. This is possible, because, in contrast to SiO_2/Si_3N_4, alumina film is not stressed, unlike the case of SiO_2/Si_3N_4 membranes [15]. An example of this cut, an 1 mm hole laser-drilled, is presented in Fig. 5. This cut does not lead to the formation of cracks in the membrane; this is illustrated by SEM photo given in the same figure. As a result, laser beam can be applied for the formation of holes, cantilevers, and other elements of MEMS able to improve the operation of chemical and physical sensors based on alumina membranes.

An example of the application of laser cut is given in Fig. 6. The heater of the gas sensor is located in the tip of triangle shaped cantilever. The application of cantilever shaped microheaters gives a possibility to reduce, first, power consumption of the microhotplate and, second, improves long-term stability of the microhotplate at temperature cycling. Appropriate plots are presented in Fig. 7. For the measurement of temperature, we used pre-determined value of the temperature coefficient of resistance (TCR) equal for thin films of platinum deposited by magnetron sputtering to 0.00275 K^{-1} in a temperature range 20 – 500^0C. This value was measured using external oven calibrated with the application of thermocouple. The comparison of the plots 1 (power consumption of whole circular membrane 3 mm in diameter fixed by melted glass on ceramic substrate with laser drilled hole; this type microhotplate is drawn in Fig. 1 [4]) and plot 2 (triangle cantilever shaped microhotplate) shows that the application of the laser cut cantilever enables a decrease in power consumption of the microheater by approximately 20 %. The analysis of the results showed that the optimization of the shape of the cantilever (for example, the application of triangle with sharper vertex) will decrease power consumption down to ~ 60 mW at 450^0C.

Membranes based on yttria stabilized zirconia

The main advantage of ZrO_2 ceramics compared to all other ceramic materials is extremely low thermal conductivity. It is equal to about 2.5 W/m·K. This value is approximately one order of magnitude lower that the thermal conductivity of aluminum oxide. Therefore, MEMS device made of zirconia ceramics will consume conciderably lower power compared to the device made of alumina.

We fabricated experimental samples of MEMS platforms based on ceramic membranes made of ittria stabilized zirconia (YSZ). Ceramic YSZ film was fabricated by slip casting followed by sintering under mechanical load. Ceramic slim consisted of 20 nm particles. Therefore acceptable quality of sintering was obtained at relatively low sintering temperature of about 1150^0C during 12 hours. Resulting membrane had thickness of 10 microns. Fig. 8 presents photo of the membrane and SEM pictures of the membrane surface and its cross section.

After sintering, the membrane consists of roundish particles with size of ~ 0.2 microns. This particle size and surface roughness enables easy deposition of platinum heater layer using magnetron sputtering.

The membrane was glued to ceramic substrate (0.5 mm thick) with 3 mm holes made of the same YSZ material as membrane (see Fig. 1) . For gluing, the surface of the substrate was coated with thick film ink using screen printer PT-100; this ink consisted of melted glass with the expansion coefficient close to the expansion coefficient of YSZ material and organic vehicle (ehylcellulose solution in terpineol. After ink deposition, the membrane was carefully pressed to the substrate, dried at 150^0C, and annealed at 950^0C necessary for the melting of glass binder.

Microheater was fabricated in the same way as in the case of anodic aluminum oxide that is by magnetron sputtering of platinum trough shadow mask. Platinum thickness was of about 0.3 micron. After platinum deposition, the substrate with membrane and heater was annealed at 800^0C and laser cut into 6 x 6 mm chips.

The chips were packaged in TO-8 package. The most important characteristics of the microhotplate – power consumption as a function of temperature – was determined after the measurement of volt-ampere characteristics of the device. The results of the measurement are presented in Fig. 7. The microheater consumes approximately 75 mW at working temperature of 450^0C sufficient for the efficient measurement of methane concentrations in air. Further decrease in power consumption is possible with the application of microcantilevers similar to those presented in Fig. 6.

Another way available for a decrease in power consumption is related with the application of pulsing heating of the sensor. In this way, it is possible to decrease power consumption of the sensor down to approximately 1 mW (duty cycle of 1 %) at methane detection.

<u>Microhotplates based on thin boron-silicon glass.</u>

The main target of the application of thin boron-silicate glass as a substrate for gas sensors is the development of technology, which permits the fabrication of low-cost devices using simple inexpensive equipment.

A very important advantage of thin boron-silicate glass substrates is that their coefficient of thermal expansion is close to this coefficient of LTCC (low-temperature co-fired ceramics) and of silicon. This means that the appropriate MEMS structures could be fabricated by fixation of glass cantilever on thick and robust ceramic of silicon chip.

Recently, the possibility of the fabrication of such cantilevers is given by the application as a substrate 30 micron thick boron-silicate glass with transformation temperature $T_g = 720\ ^0$C. Glass substrate was coated by platinum using magnetron sputtering. Platinum was patterned in two ways. The first was the application of shadow masks the same as used before for Pt deposition on alumina and zirconia films. The second was the application of laser ablation of platinum for the formation of the microheater.

Fig. 9 presents the result of the fabrication of glass microheater using laser patterning of heater and microcantilever. For this, we used compact high precision marker system based on 20 W fiber laser; this laser system has positioning accuracy of 2.5 μm, and spot size of 50 μm. Layout of the cantilever microhotplate was designed using AutoCAD software. After laser beam engraving of platinum microhotplate, the same laser beam was used for cutting the contour of the chip and for drilling the holes used for soldering the chip to TO-8 holder.

Thermal characteristics of the microheater based on 30 micron boron-silicate glass are presented in Fig. 10. The curve (1) shows power consumption of whole membrane. Laser cut of the membrane leading to the formation of cantilever (Fig. 9) decreases heating power by approximately 80 mW at 450^0C due to a decrease of heat transfer by the material of membrane. On the other hand, the application of cantilever heater makes the microheater more robust at temperature cycling because this type microheater does not bend when its temperature changes.

Further decrease in power consumption of the cantilever type microhotplates will be done with the optimization of the sensor layout and with the application of materials with lower heat conductivity (for example, zirconia based ceramics) or thinner films used for the fabrication of microhotplates.

The main advantage of the technological approach suggested in this paragraph, that is of the application of laser engraving for the patterning of both platinum heater layer and thin glass or alumina ceramic film consists in the possibility to modify easily the layout of the gas sensor chip and in the relatively low cost of the equipment used for the fabrication of these chips. This low cost leads to very significant decrease in total cost of middle scale sensor production.

Application

The application of this type of microhotplates permit the improvement of the selectivity of the sensors by the use of temperature modulation scheme of gas concentration measurements [1]. This possibility is given by relatively short thermal response time of the microhotplate equal to approximately 80 ms.

The unique possibility given by the application of non-silicon MEMS compared to the MEMS sensors based on silicon technology and produced by companied Figaro Inc. (Japan), Applied Sensors (Germany), and SGX (Switzerland) consists in the possibility to heat the sensing layer up to 450^0C and even higher (measurement cycle) and up to 800^0C (technological annealing of the sensing layer). This possibility enables, first, the measurements of all gases including methane and, second, makes possible the application of thermal excitation of sensing material described in detail in [1].

An appropriate example is presented in Fig. 11. Sensing layer consisting of tin dioxide particles (~ 10 nm in diameter) doped with 3 wt. % of palladium was deposited on the microhotplate. Sensing layer was annealed at 750^0C for 15 min. For the measurement, the sensing layer was heated up to 450^0C for 0.5 s and then the measurement of sensing layer resistance was carried out at 110^0C. At this temperature the sensing layer is selective to CO concentration, because CO at this temperature can reduce PdO_x formed at 450^0C, whereas H_2 does not react with palladium oxide at this low temperature. Details of this reaction mechanism were described in [1]. As a result, this measurement cycle leads to selective response of gas sensor to CO compared to H_2, the selectivity coefficient of the sensor reaches ~ 10.

Conclusion

We demonstrated that the application of non-silicon technology for the fabrication of sensing MEMS devices is very prospective and assures important advantages in comparison with traditional silicon technology. These advantages are related first of all with a very considerable enlargement of the range of working temperatures (from maximum 250-300^0C for silicon technology to 550^0C for ceramic technology) and of the range of technological treatment temperature of the sensor (up to almost 1000^0C). This enlargement of temperature range results in the possibility to detect gases, not detectable with silicon based devices, first of all methane, and to improve significantly selectivity of gas sensors using temperature modulation regime of gas sensor. Another important result is the possibility to fabricate using ceramic MEMS technology sensors operating under harsh environmental conditions including industrial and natural catastrophes.

On the other hand, application parameters of ceramic MEMS devices, such as power consumption, the possibility to operate in wireless devices is very compatible compared to devices based on silicon technology: power consumption at 450^0C can be reduced down to 50 mW at permanent heating and down to 1 mW in pulsing mode; long term drift of heater resistance is less than 3 % per year, membrane resists to thermal shocks and remains stable after more than 5 millions of switch on-off cycles.

All these parameters make ceramic MEMS technology important candidate for the development of next generation of gas sensors.

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Figure Captions

Figure. 1. Structure of the ceramic MEMS gas sensor. 1 – ceramic substrate; 2 – glue layer; 3 – thin ceramic membrane; 4 – sensing layer; 5 – laser drilled hole; 6 – platinum heater of the gas sensor; 7 – contact pads to the heater and sensing layer; 8 – measuring electrode.

Figure. 2. (A) XRD pattern of an “as prepared” amorphous porous AA membrane fabricated by aluminum anodizing. (B) XDR pattern recorded for the AA membrane after annealing at 1000^0C for 1 hour. Vertical lines designate the calculated peak positions for crystalline phases $Al_{2,14}O_{3,3}$ (red, marked by “1”) and $Al_{2,43}O_{3,64}$.

Figure 3. Thin alumina film made by anodic oxidation of aluminum foil after annealing at 1000^0C for 15 hours. The size of the film is of 48x60 mm, thickness is of 12 microns.

Figure 4. SEM views of cross section of a nanoporous alumina membrane fabricated via anodizing an aluminum foil followed by annealing in air at 1150^0C for 15 hours. The inset in the left image shows the membrane surface morphology.

Figure 5. The hole 1 mm in diameter drilled by laser in 20 micron thick alumina film and the SEM photo of the edge of laser cut.

Figure 6. Cantilever shaped element fabricated by laser cutting.

Figure 7. Power consumption of microheater based on anodic aluminum oxide (AAO) film with thickness of 12 microns as a function of heater temperature: (1) whole membrane (Fig. 1) with the heater located in the center; (2) triangle shaped cantilever with heater located in the tip of the cantilever; (3) whole membrane (Fig. 1) made of 10 micron thick yttria stabilized zirconia material.

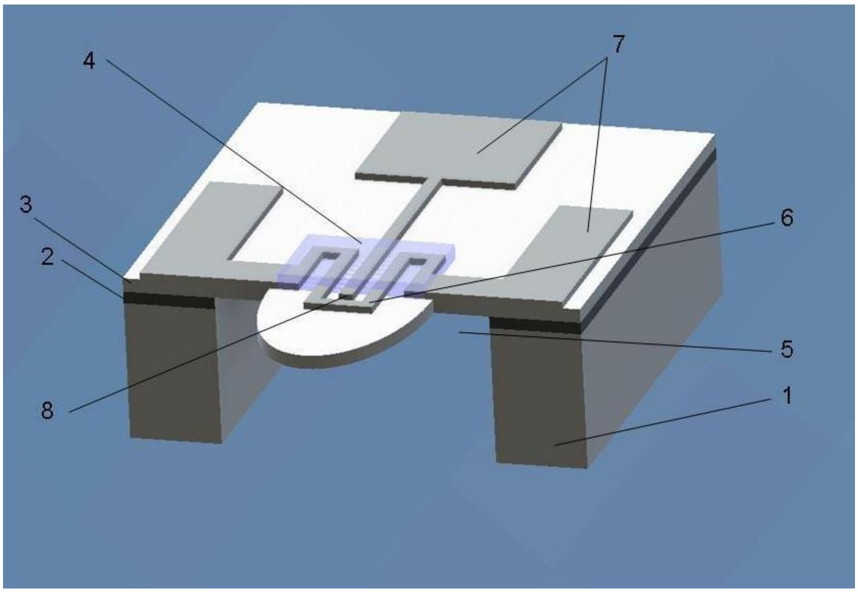
Figure 8. 10 micron thick film made of yttria stabilized zirconia (YSZ) after annealing at 1150^0C for 12 hours and SEM photo of the surface and cross section of the membrane.

Figure 9. Cantilever type microheater on 30 micron thick boron-silicon glass substrate. The platinum heater is patterned by laser ablation.

Figure 10. Power consumption of microheater based on 30 micron boron-silicate glass: (1) whole membrane (Fig. 1) with the heater located in the center; (2) triangle shape cantilever with heater located in the tip of the cantilever.

Figure 11. The application of MEMS sensor for the selective measurement of CO and H_2 concentrations. Sensing material deposited on MEMS microheater is Pd (3 wt. %) doped tin dioxide with particle size of about 10 nm. Measurement cycle consists of the sensing layer heating up to 450^0C for 0.5 s followed by low temperature measurement phase at 110^0C (2.5 s): (A) CO concentration 3 ppm; (B) H_2 concentration 3 ppm. Selectivity coefficient reached for these gases is of about 10.

Figures



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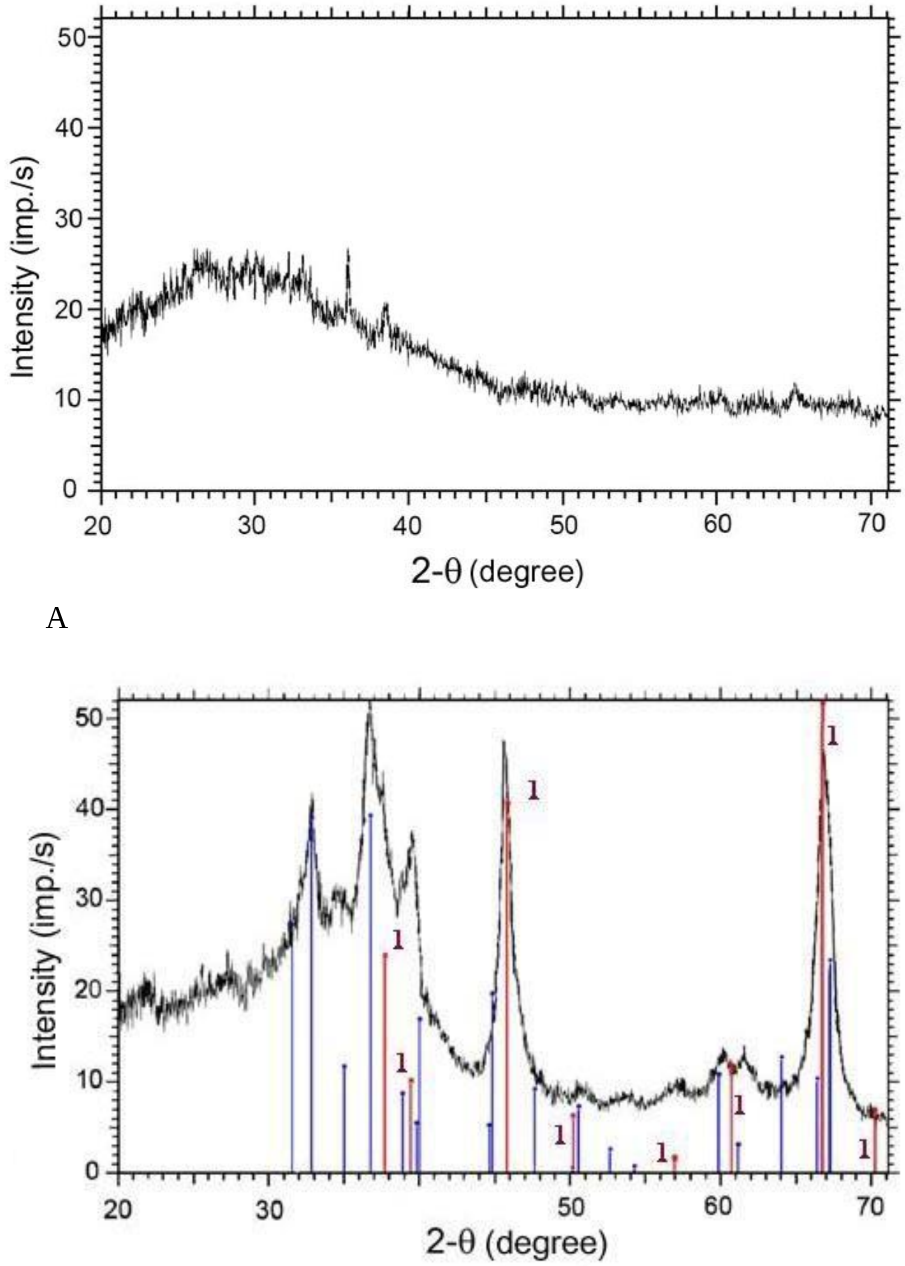
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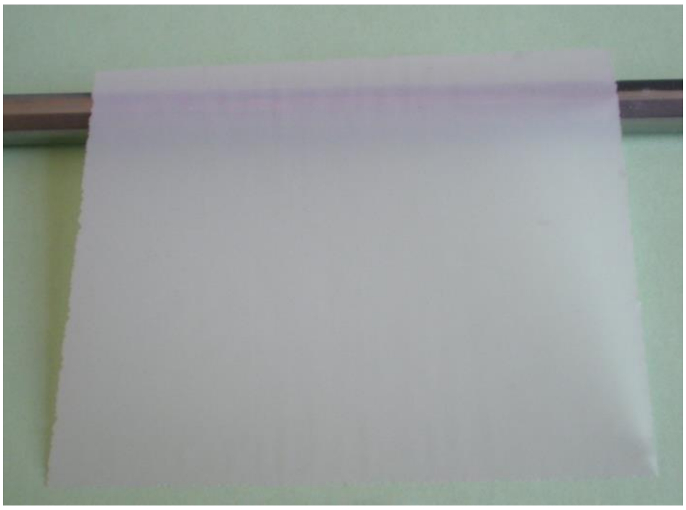
Figure 2



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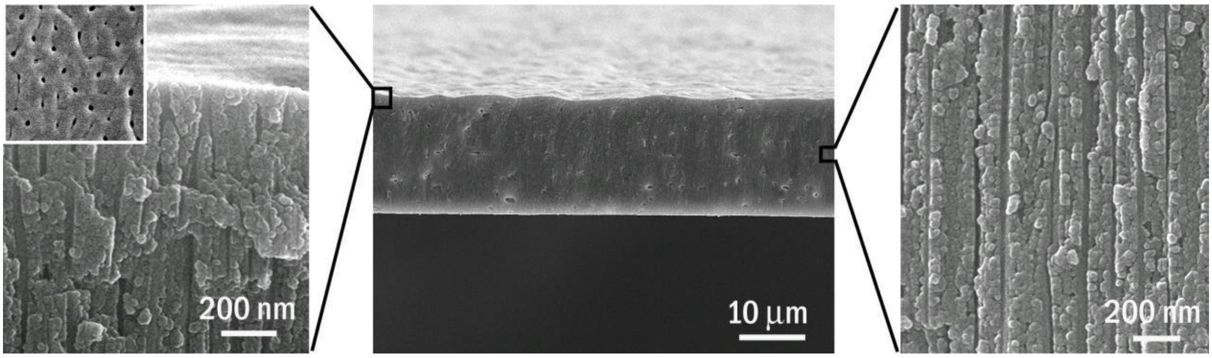

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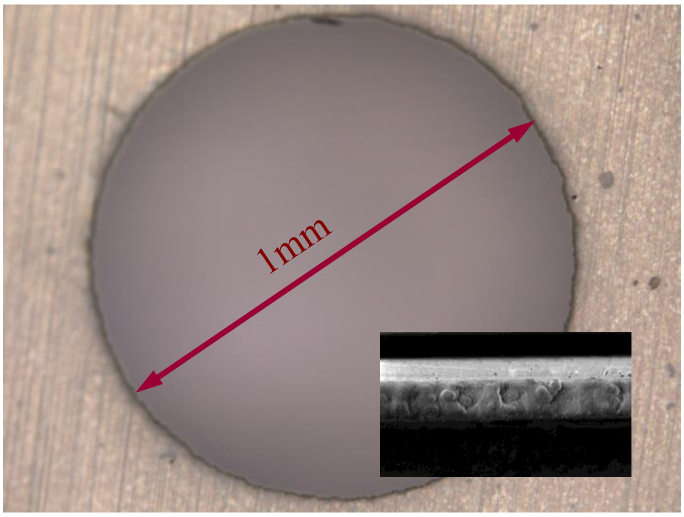

Figure 5

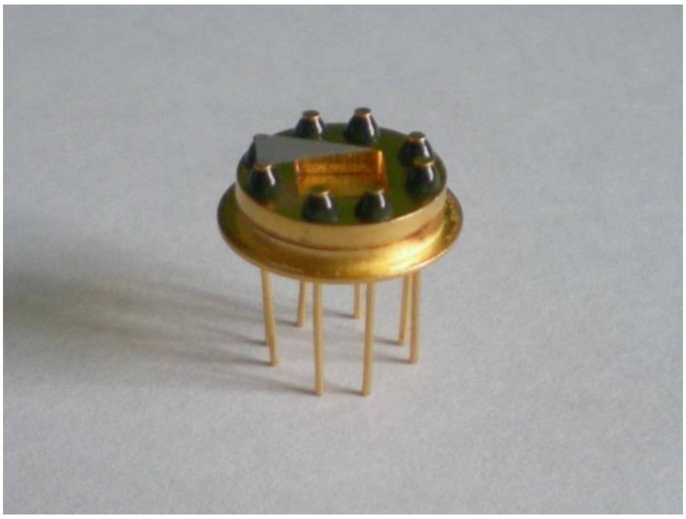

Figure 6

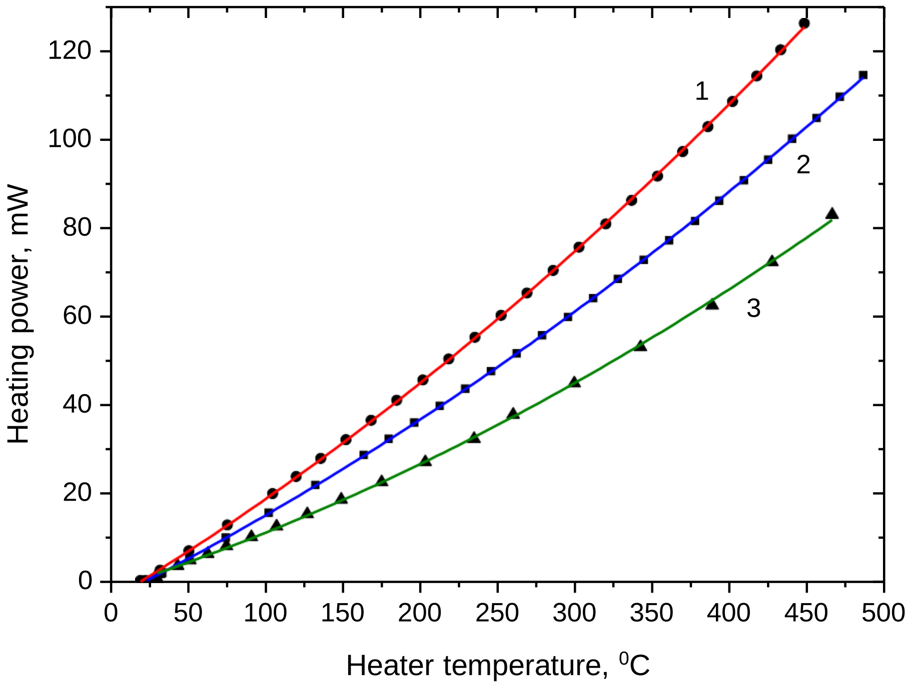
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Figure 7

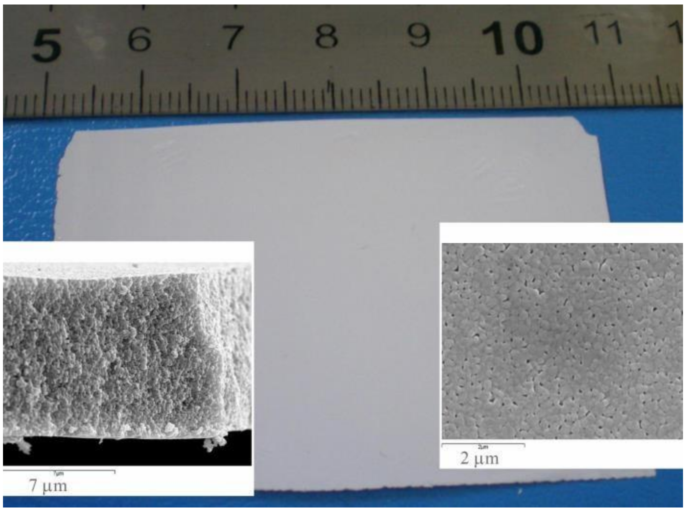


Figure 8

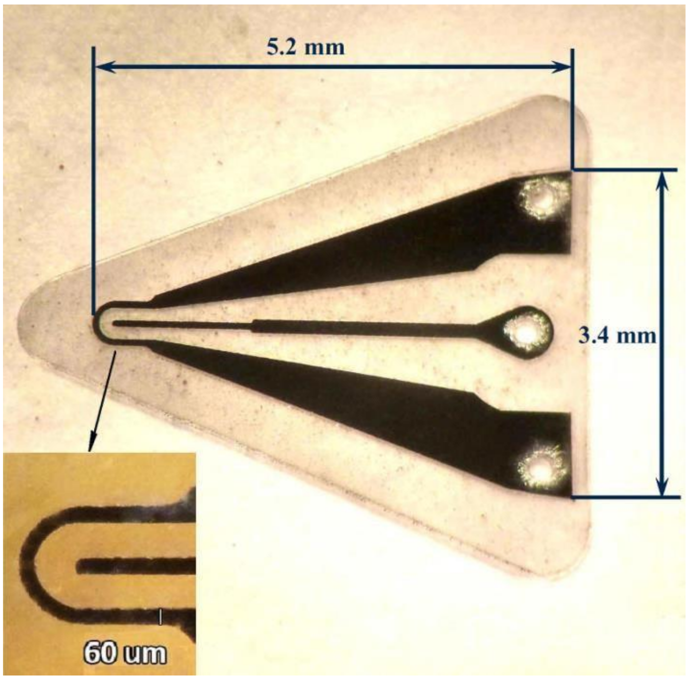


Figure 9

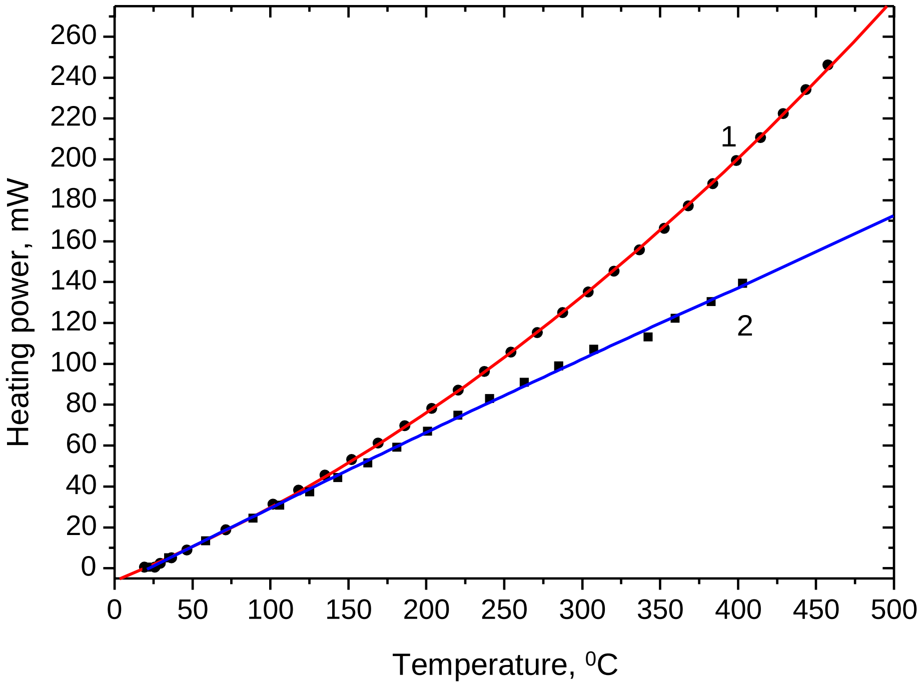
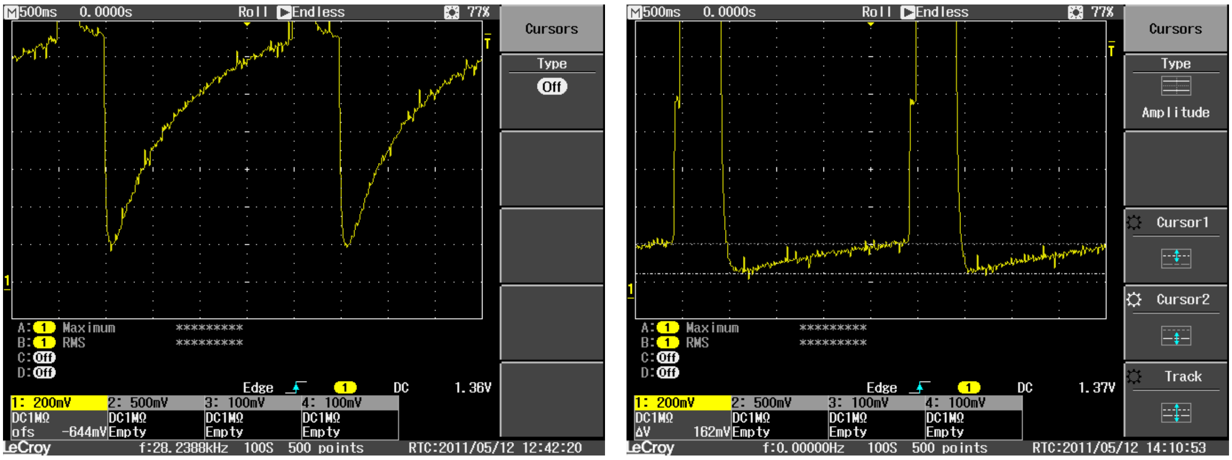

Heating power, mW
Temperature, ^{0}C
0
20
40
60
80
100
120
140
160
180
200
220
240
260
0
50
100
150
200
250
300
350
400
450
500
1
2

Figure 10


M 500ms 0.0000s
Roll Endless
77%
Cursors
Type
Off
A: 1 Maximum *********
B: 1 RMS *********
C: Off
D: Off
Edge 1 DC 1.36V
1: 200mV
2: 500mV
3: 100mV
4: 100mV
DC1MΩ
ofs -644mV
Empty
LeCroy
f:28.2388kHz 100S 500 points
RTC:2011/05/12 12:42:20
M 500ms 0.0000s
Roll Endless
77%
Cursors
Type
Amplitude
Cursor1
Cursor2
Track
A: 1 Maximum *********
B: 1 RMS *********
C: Off
D: Off
Edge 1 DC 1.37V
1: 200mV
2: 500mV
3: 100mV
4: 100mV
DC1MΩ
ΔV 162mV
Empty
LeCroy
f:0.00000Hz 100S 500 points
RTC:2011/05/12 14:10:53

A

B

Figure 11