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POLYMERIC PRESSURE SENSORS: A CONCEPTUAL VIEW

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Abstract. *In the first part of this paper, we present a review of the piezoresistive pressure sensors based on polymeric thick films deposited on rigid and flexible diaphragm. The study of the state of the art has shown the performances of this technology, where maximum sensitivity is obtained on thin flexible diaphragm for a gauge factor of about* 10*, in a pressure range of* 0÷5 kPa*. The present challenges come from the high temperature coefficient of the resistance of about* 500 ppm/°C*, and the long temperature drifts of about* (0.5÷2)%*, which may require improved repeatability of fabrication technology and advanced differential signal processing techniques for the market acceptance. In the second part of the paper, we present our novel concepts for the realization of the piezoresistive pressure sensors. The first concept consists in the surface modification of the organic substrate by ion implantation of nitrogen and phosphorus species for creating piezoresistive behavior and high electrical conductivity of organic piezoresistors. The second concept consists in the novel chemical synthesis route of organic thin film by doping the polyaniline with large molecules of p-sulfonated calix[n]arene* (*n* =4, 6, 8)*, sulfonated crown ethers, in the liquid state. Addition of the metal nanoparticles to the previous homogeneous solution can further increase the piezoresistive factor. Other new features of our second concept come from the direct printing from solution of the above piezoresistive organic thin films, as well as metallic films interconnecting the piezoresistors, and finally the monolithic fabrication of the sensor rim and diaphragm by plastic injection molding, where the pressure diaphragm could be as thin as* 75 micrometers*.*

Keywords: piezoresistive organic films, polymeric thick film, pressure sensors, metal Nanoparticples, *p*-sulfonated calix[n]arene, sulfonated crown ethers

1. Introduction

Pressure monitoring is an important parameter in the control of a large diversity of industrial processes and medical applications. Pressure can be measured by mechanical devices, as well as electro-mechanical and electro-optical instruments. The measurement of the pressure of a fluid by pure mechanical principle is based on the presence of an elastic diaphragm fixed at one end, which is moving its free end as a result of pressure variation, and its position change is indicated by a needle connected to the free end, and which is thus rotating with respect to its zero position (Fig. 1).

This principle is used for the pressure measurement on gas/liquids pipelines, where pressure manometers based on Bourdon tubes are still in place, today.

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In other cases, the elastic diaphragm is fixed at both ends, and this is deflecting in the central part as a result of pressure change (Fig. 2). Such deflection is creating strain in the diaphragm, and its value can be also used for the pressure measurement, in the so called strain gauges. These deflecting diaphragms have opened the way for the electro-mechanical principles of pressure measurement.

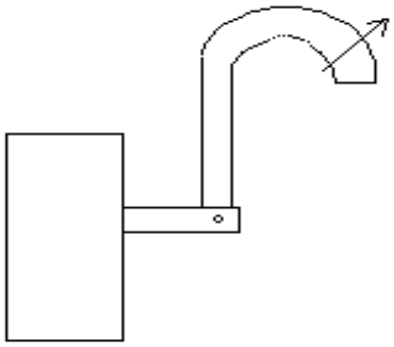

Fig. 1. Schematic picture of a Bourdon tube used for a pressure manometer.

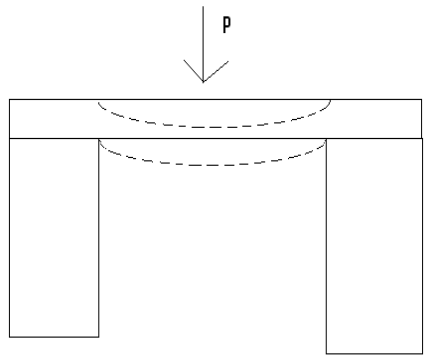

Fig. 2. Pressure diaphragm flexing under applied external pressure.

Historically speaking, in 1856 Lord Kelvin has discovered that the electrical conductor can change its electrical resistance, when it is strained due to an external applied force. Thus, he can be credited with the discovery of the piezoresistive effect in metals, which was then used in metallic strain gauge devices for multiple applications (strain, torque, force) including pressure sensing. Later, the piezoresistive effect was defined as a change in the electrical resistivity of a material as a function of the externally applied stress on it. Now, it is generally agreed that the discovery of piezoresistive effect is at the origin of the most of electric sensors for mechanical measurands defined as devices able to convert a non-electrical signal (like fluid) pressure into an electrical signal (like voltage). The silicon technology followed by the micro-electro-mechanical system (MEMS) technology have used all these principles for the miniaturization of the existing macroscopic devices and sensors.

The impetuous development of the MEMS technology was founded on two important technical pillars, consisting of well-established integrated circuit (IC) infrastructure and excellent operation of the macroscopic principles at the micrometer scale. A convincing demonstration of this successful approach is coming from microsystems for pressure measurement, where the well-known principle of macroscopic diaphragm movement as a function of pressure has been transferred to microtechnology scale with very good results.

The era of miniaturized pressure sensors has been triggered by the discovery of the piezoresistive effect in silicon and germanium, in 1954 by Charles Smith [1].

The time interval from piezoresistance discovery to the first associated commercial product was short, as in 1959, Kulite was already delivering the first silicon strain-gauges bonded on a metal diaphragm. During sensor operation, the metal diaphragm is elastically deflecting due to the applied pressure, and a tensile stress is developed in the central region of the diaphragm, while a compressive stress is developed at the periphery of the diaphragm (Fig. 2.).

Such a stress is transmitted to the strain gauge, which "is feeling" it by the well-known stress-strain correlation, and a change in the resistance is obtained due to the piezoresistive effect, which is thus an indication of the pressure to be measured. In the presence of an external pressure, a piezoresistor located on the central part of the diaphragm from Fig. 2. is exposed to tensile stress and its resistance value is increasing, while a piezoresistor located at the periphery of the diaphragm, near the edge, is exposed to compressive stress and its resistance value is decreasing with respect to the value specific to zero stress. For an accurate pressure measurement, where the ageing effects in the piezoresistors, as well as external temperature variations to be compensated, four piezoresistors are located in the arms of a Wheatstone bridge, and thus obtaining a differential sensing configuration for the signal conditioning.

Using such an approach, at the beginning of 1970's, IBM has proven the operation of the first piezoresistive pressure sensor with silicon diaphragm, while the first commercial "all-silicon" pressure sensor was delivered in 1974 [2].

The piezoresistive effect was measured in doped silicon resistors, which were located in well-defined regions of the diaphragm, as described above. The excellent elastic properties of silicon diaphragm, combined with the fact that the silicon piezoresistor is obtained intrinsically in the diaphragm, without any need of strain gauge bonding to silicon diaphragm can explain the excellent performance of silicon MEMS pressure sensors. Thus, the MEMS silicon technology has proven its capability to generate commercial products, where thin silicon diaphragms have been used from very beginning in medical application for measuring blood pressure.

In parallel with silicon MEMS technology, which has brought to the market the first miniaturized microsystems, sensors and actuators, like pressure sensors, accelerometers, inkjet nozzles for thermal inkjet technologies and thus predicting its long-term innovation capabilities, other sensing technologies were emerging, which were targeting mechanical sensing applications (pressure torque, force) based on other than silicon materials and substrates. It is the case of thick film technologies which have found their niche sensing applications.

In this paper, we shall briefly present polymeric film technologies for pressure sensing applications. At the beginning of the study, the state of the art for the

piezoresistive polymeric thick films will be shown, where such materials are deposited on diaphragms made of glass, alumina, or even flexible substrates, and their pressure sensing properties are described [3-6].

Finally, our concepts for new polymeric pressure sensors, including "all-organic" technologies will be shown. Here, firstly, we present a novel pressure sensing concept, where the plastic substrate will receive electrical conductivity and piezoresistivity properties on well-defined regions of the organic diaphragm by using IC specific technology like ion implantation [7]. Then, another novel concept for "all-plastic" pressure sensor is described where chemical synthesis for the preparation of new organic thin films with enhanced electrical conductivity is shown. This piezoresistive pressure sensor is made by additive, maskless direct printing of the organic films in well-defined positions of plastic diaphragm, which is obtained by injection molding [8].

2. Piezoresistive pressure sensors based on thick film resistors

The operation principle of the piezoresistive mechanical sensors based on thick films is similar to that described for silicon sensors, but the diaphragm and the piezoresistor are made of different materials. For the evaluation of different piezoresistive materials, the gauge factor is used, and this is equal to the ratio between the relative variation of the resistance ($\Delta R/R$) and the relative variation of the resistor length (or the strain ($\Delta \ell/\ell$)). The piezoresistive behavior in thick resistive layers was systematically investigated by using commercial ruthenate thick films from Dupont [9]. The thick films were deposited on alumina substrate as a gel of high viscosity by screen printing technology, and then fired at high temperatures, around 850-950 ^{o}C, for obtaining thick solid layers. The gauge factor for these ruthenate thick films was in the range of 11-14, being weakly dependent on the strain direction.

Such piezoresistive thick films have a gauge factor which is higher than that of the metal strain gauge (1.8-4.5), and much smaller than that of semiconductors (40-200) [10]. The temperature coefficient of these thick ruthenate film piezoresistor (TCR) was about 100 ppm/^{o}C, while the temperature coefficient of the gauge factor (TCGF) was smaller than 500 ppm/^{o}C.

These values can be compared with the similar ones of the metal wire strain gauges, where the TCR has a large range of variation (20-4000) ppm/^{o}C, and TCGF is in the range 20-100 ppm/^{o}C [9]. Unfortunately, such high temperature technology is restricting the type of substrate to be used to only ceramics, and is increasing the cost of processing.

As a low temperature alternative to the well-established ruthenate thick film piezoresistive technology presented above, the pressure sensor based on

polymeric thick film resistors was proposed at the beginning of the ’90 exploiting the piezoresistive effect in such organic layers, which can tolerate processing temperatures not higher than 150-300 ^{o}C [3].

The thick film composition consists of an organic polymer matrix, like a polyimide, which is loaded with carbon for obtaining the resistive and piezoresistive behavior. The sensing diaphragm is made of different rigid materials like, alumina, glass-reinforced epoxy laminate (FR 4), or even a flexible substrate [4].

For the realization of the planar piezoresistor, initially, the metallic electrodes were deposited and patterned on the substrate. Then, the deposition of the polymeric thick films on the rigid substrate, like alumina, glass, or FR 4 is made by screen printing technology, followed by thermal consolidation process at low temperatures, below 300 ^{o}C. These planar polymeric thick films based polymeric piezoresistors have a gauge factor of about 10, which is rather similar to the value obtained for ruthenate thick films, but the TCR is equal to +/– 500 ppm/^{o}C, which is much higher with respect to the value obtained for ruthenate material (100 ppm/^{o}C).

In the case of a “sandwich” piezoresistor configuration, when the polymeric thick film is sandwiched (on the z direction) between two electrodes, with the first electrode deposited on the substrate and the second electrode deposited on the polymeric thick film, the gauge factor has reached a much higher value of about 80 [5]. In addition, these sandwich polymeric piezoresistive pressure sensors have shown a very high value of the TCR, of about – 2200 ppm/^{o}C, which may be more difficult to compensate, even in Wheatstone configuration, due to existing mismatches between the TCR of different piezoresistors.

Also, poorer reproducibility of sandwich sensor with respect to planar ones was obtained.

When such polymeric thick films were deposited on flexible substrates, having much lower thickness with respect to the rigid one, the sensitivity of the planar polymeric thick film pressure sensors has increased of about 6 times, but, in our opinion, this result should be correlated only with the pressure diaphragm properties (thickness and Young module) and not to sensing layer, itself. Such thick films polymeric planar pressure sensors on rigid substrate are working in the temperature range from 0 to 75 ^{o}C, and their linear response was obtained for a pressure range from 0 to 10^{6} Pa.

Typical linearity plots of the planar polymeric thick film deposited on alumina substrates have shown a non-linearity of about 3% in the range [0; +500] microstrains (1 microstrain means a dilation/shrinkage of 1 micrometer of

material having a length of 1 meter), while the sandwiched piezoresistor have had a non-linearity of 14% in the same strain range [5]. One of the biggest drawbacks of such polymeric pressure sensors is the long-term drift, equal to about 0.5-2 %, and which was estimated by the sensor output drift after an accelerated ageing test at 1000 ^{o}C and 85 ^{o}C.

This behavior should be correlated to the specific ageing mechanisms of the organic materials. The 'challenging' temperature behavior of these polymeric pressure sensors followed by their relative high drifts due to material ageing can be partially solved by design of the diaphragm (thickness and Young modulus of organic substrate) as shown in [3,4] and by means of Wheatstone bridge-based differential signal conditioning.

Therefore, much work should be devoted here, but such polymeric sensors are the best candidate for low cost disposable pressure sensors for applications where the accuracy is not critical.

3. Piezoresistive pressure sensors based on surface modified polymeric diaphragm

Flexible electronics (flex circuits), where the "traditional" silicon integrated circuits are placed by surface mounting technologies on a flexible plastic substrate is rapidly advancing in many market applications, from portable video camera to solar cell [11].

For these flex circuits, the metallic interconnections between different integrated circuits are made by standard photolithographic techniques. In parallel, an "all-organic" electronics is emerging where the semiconductor devices like organic light emitting diodes (OLED) and organic field emission transistor (OFET) are manufactured in the body of the organic semiconductor materials [12]. In this context, the sensing domain is going to add new capabilities to the rigid and flexible organic electronics. On this idea, recently, we have proposed a concept for pressure sensors where a polymeric pressure diaphragm is surface modified in order to obtain selectively piezoresistive effect [7].

The novelty of our approach comes from the technology of the piezoresistor realization, where we have applied ion implantation technique for the local realization of the piezoresistivity and electrical conduction enhancement, as it will be described below. In Fig. 3, we show a cross section view of "an-all plastic" piezoresistive pressure sensor, before packaging, where the substrate and the diaphragm come from the same starting rigid plastic material, which could be a polyimide, like Kapton from Dupont or liquid crystal polymer. Alternatively, polystyrene-co-acrylonitrile (SAN) 80/20 could be used. Such organic polymers are dielectric materials, which have a very high electrical resistivity, and therefore they cannot be used "as they are" for reaching the intended function.

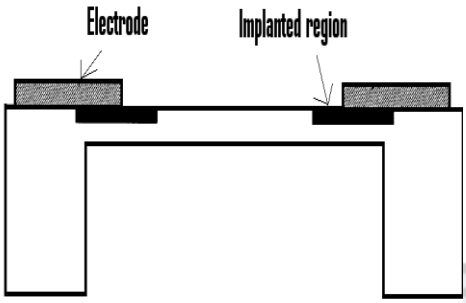

Fig. 3. Schematic of a pressure sensor with diaphragm obtained by etching back side of the substrate and piezoresistor fabricated by ion implantation.

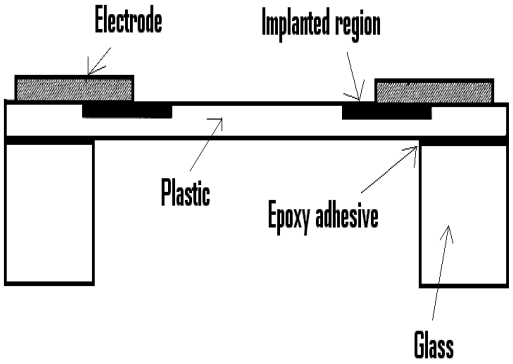

Fig. 4. Pressure with plastic diaphragm bonded by adhesive to glass.

However, it was already shown in the literature that the ion implantation can dramatically increase the electrical conduction of these plastic materials [13]. Therefore, we have proposed to perform a high dose implantation of the nitrogen species, in well-defined regions of the diaphragm for the generation of the piezoresistive regions. This process was followed by a phosphorus ion implantation in the same region for further enhancement of the electrical conductivity of the piezoresistive regions.

For generating such electrical and piezoelectrical properties selectively, in the organic diaphragm, standard IC photo-lithographical processes can be used. This is possible due to the chemical resistance of these rigid plastic materials to the solvents and the other solutions used to remove the photoresist, at the end of the ion implantation processes. Subsequently, the electrical contacts to the piezoresistors are done by IC technology processes like electron-gun physical vapor deposition, where, for example, a thin film combination like chromium/gold can be used. Chromium is assuring the adherence of the gold layer to the plastic substrate and ion-implanted regions of the piezoresistors.

For the realization of the diaphragm in the starting plastic material, the back-side etching of the substrate is performed by plasma techniques, like reactive ion etching (RIE). The realization of such pressure diaphragms from Fig. 3. is possible by metal masking of the entire front side, and of selective regions of back side regions of the substrate, which should survive after deep etching and subsequently form what is called the rim of the pressure sensor. The thickness of the pressure diaphragm is determined by the pressure range needed to be measured and the important requirement that the deflection of the pressure diaphragm under external pressure to be in the elasticity domain of that material. Such design conditions are preserving the linearity of the sensor response and also minimizing the hysteresis and long-term drifts of the sensors.

In other applications, it may be useful to use a glass substrate and a plastic diaphragm which can be bonded together for defining the pressure sensor. Such an approach is also possible within the above concept, where initially a plastic foil (similar to the silicon wafer from the point of view of batch processing) of the thickness required by the pressure sensing application is processed, as presented above, but, in this case there is no need for the back side etching, as the entire thickness will play the role of the diaphragm. In this case, which is shown in Fig. 4., micromachining of the glass substrate is needed for the realization of the rim of the sensor, which is also allowing the access of the fluid pressure (air, liquid) to the pressure diaphragm. Glass MEMS is a well-established batch technology for sensor applications, and also for sensor packaging, in general.

In this case, the key process is glass drilling at the "wafer" level for the sensor rim realization, and this can be done by either wet etching in HF based solutions, or by RIE or even laser drilling. The signal conditioning techniques are "standard" consisting in Wheatstone bridge, which will be described in more details, in the next section.

4. Low cost "all-plastic" piezoresistive pressure sensors

In different industrial domains, there is a strong demand for disposable, low cost pressure sensors. In such cases, the silicon technology may still be expensive, considering the cost of clean room processes, and of the monocrystalline silicon substrate itself.

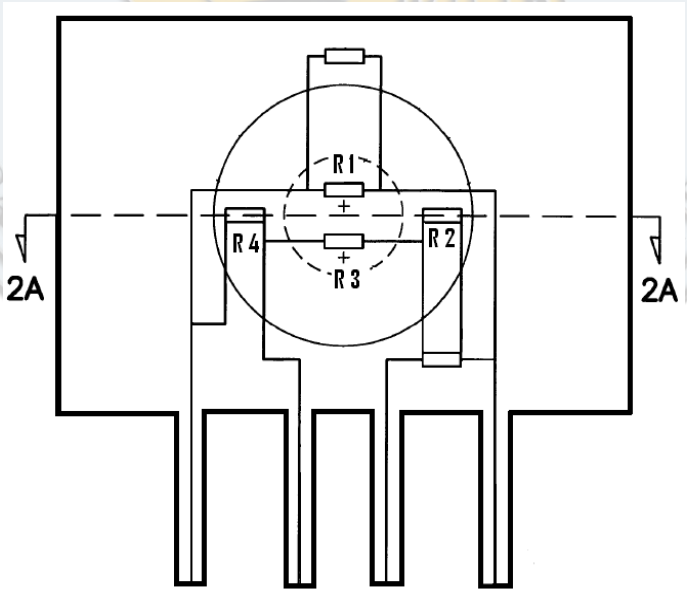

Fig. 5a. Top view of the low cost pressure sensor
made by plasting molding and direct printing of organic piezoresistive layer.

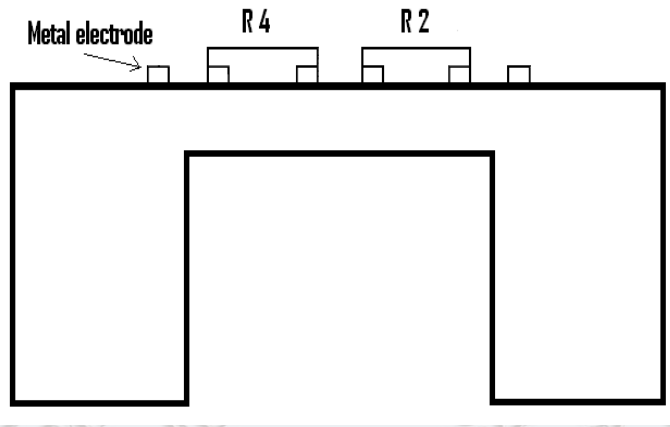

Fig. 5b. Cross section view through 2A-2A axis.

For such applications, where even the photolithographical processing should be avoided, we have developed a concept for a simple technology based on plastic injection molding of the sensor rim and pressure diaphragm in conjunction with mask-less, direct printing methods for the deposition of the metal, and novel organic piezoresistive layers [8].

In Fig. 5. a), we show a top view of the pressure sensor based on four piezoresistors, R_1-R_4, all of them being located in well-defined positions of the molded plastic diaphragm, while in Fig. 5. b) we show a cross section view through the central region of the pressure diaphragm.

These piezoresistors are electrically interconnected in a Wheatstone bridge, as shown in Fig. 6. In the absence of an external pressure, all four piezoresistors have the same value of the resistance, and the output voltage of the Wheatstone bridge is equal to zero.

In order to obtain this zero voltage, two potentiometers are also connected to the Wheatstone bridge, as in Fig. 5. a) and Fig. 6.

In the presence of an external pressure, the piezoresistors R_1 and R_3, which are positioned in the central region of the diaphragm, are exposed to tensile stress, while R_2 and R_4, which are located at the periphery of the diaphragm are exposed to the compressive stress.

The variation of the piezoresistance as a function of pressure can be written as follows:

$$R_1 = R_o(1 + x)$$
$$R_2 = R_o(1 - x)$$
$$R_3 = R_o(1 + x)$$
$$R_4 = R_o(1 - x)$$

where, R_o is the resistance value of the piezoresistor at the reference pressure, while $x = G^*\varepsilon$, where G is the gauge factor of piezoresistance, and ε is relative deformation of the length of the piezoresistor, $\varepsilon = \Delta\ell/\ell$ [10].

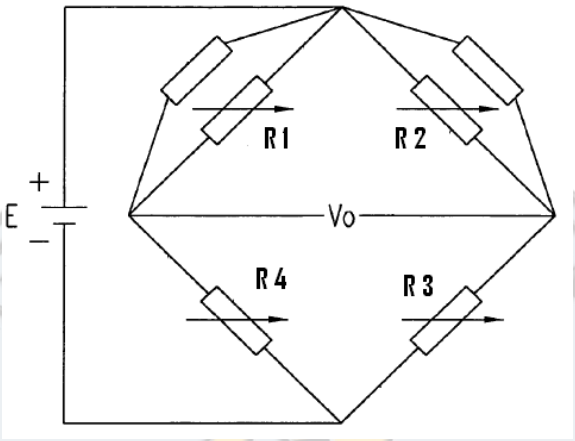

Fig. 6. Wheatstone bridge with four piezoresistors for maximum pressure sensitivity.

As mentioned above, the novel aspects of the pressure sensor realization come from the new organic piezoresistive materials, the maskless method for film resistor deposition, and as well as the cheap technology proposed for the plastic diaphragm realization.

For the realization of the piezoresistive films, we are considering more chemical synthesis routes, which are aiming an increased electrical conductivity and piezoresistivity of the organic layer. In the case of an all-organic piezoresistive layer, for example, we suggest starting with polyaniline and doping it with large molecules like *p*-sulfonato-calix[n]arenes (n = 4, 6, 8), p-sulfonated-calix[n]arenes (n = 4, 6, 8), tosylates, carboxylic acids of calix[n]arenes (n = 4, 6, 8), sulfonated crown ethers, sulfonated cyclodextrines, carboxylic acid nanotubes, or carboxylic acid of fullerenes.

All these compounds can be dissolved in water and other solvents. They can generate π-stacking interactions with polyanilines and thus contributing to the increase of electrical conductivity of the polymeric film.

In Fig. 7. we show the chemical formulae of the *p*-sulfonated-calix[n]arene for n = 4, 6, 8, while in Fig. 8., we show the reaction for doping the aniline by *p*-sulfonated calix[4]arene. The synthesis of a soluble conducting polymer can be as follows. One can start with aniline substituted with an o-methoxy group and an o-ethoxy group in equimolar amounts which can be polymerized by combining with hydrogen peroxide, in aqueous solution. The dopant can be *p*-sulfonated calix[n]arenes (n = 4, 6, 8), tosylates, carboxylic acids of calix[n]arenes (n = 4, 6, 8), or sulfonated crown ethers [14].

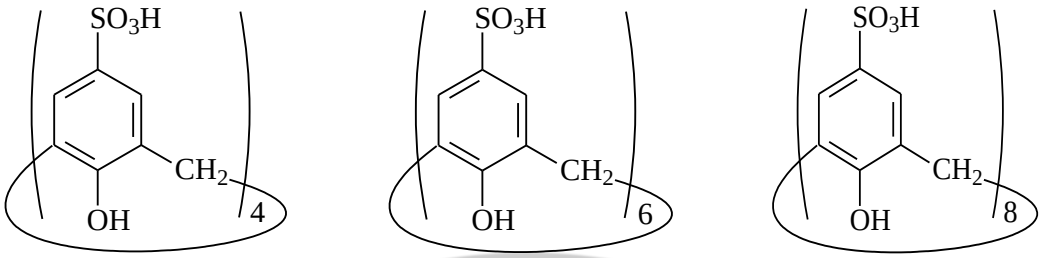

Fig. 7. Chemical formula of *p*-sulfonated calix[n]arene, where n = 4, 6, 8.

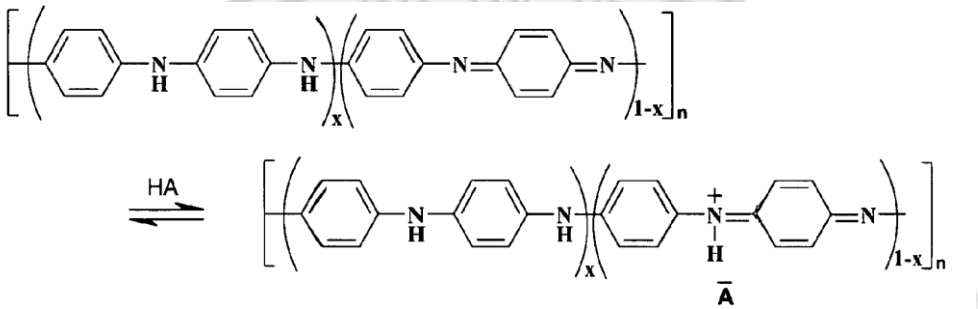

Fig. 8. Synthesis of doped polyaniline; HA stands for *p*-sulfonated calix [4]arene.

as described above. If the electrical and piezoresistive behavior of the all-organic polymer film should be further increased, then, metal nanoparticles may be added to the above polymeric sol, creating thus a heterogeneous inorganic-organic mixture in the liquid state, which will be further used for the direct printing method to be described below. Thus, an efficient increase of the gauge factor of the piezoresistors is expected by the addition of metal nanoparticles to the liquid phase of the initial pure organic solution.

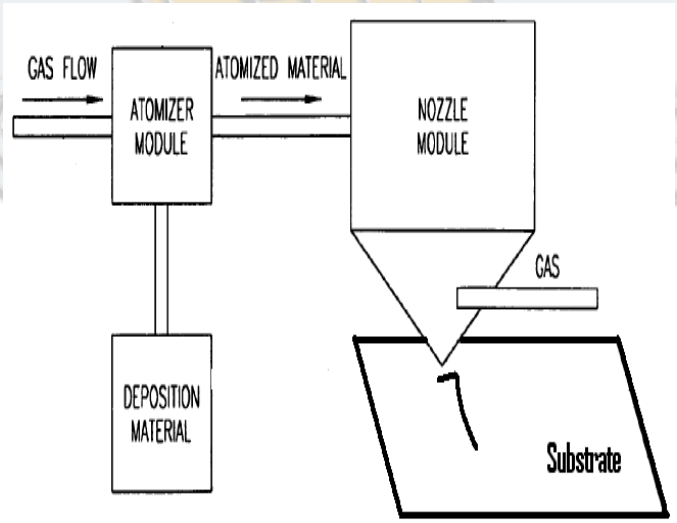

Fig. 9. Schematics of the direct printing method for the maskless preparation of solid films.

In Fig. 9., we show a schematic of direct printing apparatus. More data about this additive deposition method can be found elsewhere [15]. Specific to this direct printing method is the formation of the liquid precursor of the future solid film, by any chemical syntheses routes, which is then deposited in the right location of the substrate by a moving nozzle, while its "travel" above the substrate is computer-controlled. If we go into more details, as can be seen in Fig. 8., a gas flow is used to carry the liquid phase of the "atomized" deposition material to a nozzle, which is printing the liquid state on the substrate for the realization the pattern of the future solid film. After printing, the "gelly" layers are dried and thermally consolidated at the temperatures allowed by the organic materials, so that their chemical properties to be preserved. The advantage of this deposition method is that the solid film is printed from the very beginning in the right position and pattern, and there is no need for additional photolithographic and etching process for the layer delineation. Under such conditions, there is no loss of material and many technological steps are eliminated.

By this additive technology we can deposit not only the polymeric films as described above, but also the metallic films used to interconnect electrically the piezoresistors from Figs. 5 and 6. For these metallic conductors, one can use organic conductors, or silver based pastes, which have a low resistivity and do not introduce parasitic resistances to the Wheatstone bridge.

Finally, a novel aspect of our concept is the monolithic realization of the sensor rim and pressure diaphragm in a single process, from the same material by means of plastic injection molding. This is possible by the progress in the field of this molding process, where pressure diaphragms as thin as 75 μm are possible to be obtained. As plastic materials one can mention here, polycarbonates, polyesters such as PET or nylon, or PVC.

As mentioned from very beginning, one of the most important drivers for such all-plastic piezoresistive pressure sensors, based on injection molding and direct printing of the organic conductive and piezoresistive solid films is the high potential for a low cost fabrication process and associated materials. Such concepts show the potential of the organic semiconductors to open the way towards a new family of applications like flexible, intelligent microsystems, where both the electronic circuit signal processing and the sensing devices to be performed on the organic substrate.

5. Conclusions

In this paper we have reviewed the key materials and processes for the realization of the polymeric thick films piezoresistive, and we introduced our concepts for the preparation of novel organic piezoresistive thin films to be used in the next generation of piezoresistive pressure sensor on rigid and flexible substrates.

The polymeric pressure sensor domain was developed in the last two decades starting from the useful results obtained in the years 1980's on piezoresistive ruthenate thick films. Such polymeric thick films consisted of carbon loaded polyimide films have shown a gauge factor of about 10, and they have been used for pressure sensor operating in the range of about $0\text{-}10^6$ Pa on either rigid or flexible substrate.

The relatively high temperature coefficient of resistance (+/- 500 ppm/^{o}C) and long-term drift of these organic polymeric piezoresistive films (0.5-2)% used for either planar and sandwich device configuration are partially solved by Wheatstone bridge-based signal conditioning.

More work should be further devoted to the repeatability of fabrication technology so that the organic piezoresistors connected in the Wheatstone bridge to have similar temperature coefficient of resistance and gauge factor, and thus minimize the long- term drift of sensor output.

We have introduced two novel approaches for the preparation of the thin film polymeric piezoresistive pressure sensors. First concept consisted in the selective surface modification of the organic substrate by ion implantation of nitrogen species for inducing local piezorestivity, followed by phosphorus or boron implantion for enhancing the electrical conductivity or the organic piezoresistors.

The novelty of the second concept consisted in the original chemical synthesis of piezoresistive organic material, deposition method by direct printing, and monolithic fabrication of plastic diaphragm by injection molding.

The new organic synthesis consist in doping of polyaniline as free base(emeraldine) with large organic molecules (*p*-sulfonated calix[n]arenes *p*-sulfonated calix[n]arenes, tosylates, carboxylic acids of calix[n]arenes, sulfonated crown ethers, sulfonated cyclodextrines, carboxylic acid nanotubes, or carboxylic acid of fullerenes. The bulk counterions of dopants improve the conductivity of polyanilines

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