


Plasma Surface Modification of Flexible Substrates to Improve Grafting for Various Gas Sensing Applications: A Review

Ashish A. Nimbekar and Rajendra R. Deshmukh 

Abstract—Surrounding air pollution is one of the important warnings to human health. It is necessary to detect hazardous pollutant gases in the air. The gas sensor is the candidate for such detection. Progress of smart and flexible sensors has been receiving growing curiosity in recent years because of their enormous sensing applications. Such smart and flexible sensors can be manufactured by depositing conducting materials, such as conducting polymer, carbon nanotube (CNT), graphene oxide, and metallic nanoparticles, onto nonconducting polymeric flexible substrates, such as paper, plastic films, and textiles. Most of the flexible polymeric substrates, such as polyethylene, polyester, nylon, and polypropylene, have low surface energy and poor wettability, which weakens the adhesion between conducting material and flexible substrate. Plasma surface modification is a novel technique to enhance surface properties to produce stable composites. This review focuses on various types of plasmas for surface modification and their effects on the surface after plasma treatment to improve the grafting and adhesion of the sensing layer with the substrate. The aim of this article is to systematically overview the development of plasma-enhanced smart and flexible gas sensors to date and to envisage their future progress.

Index Terms—Flexible substrates, gas sensors, grafting, plasma, surface modification.

I. INTRODUCTION

THE current developments in electronic materials in the form of stretchable and flexible substrates have unfolded new expectations in the field of advanced electronics. Such smart and flexible composites are one of the innovative materials in the 21st century and have achieved virtuous progress in the last two decades [1]–[5]. They are really smart as they can sense and communicate with various physical parameters, such as humidity, stress, temperature, and gas, present in the surroundings. They have many sensing applications in various fields, such as gas sensors [6], humidity

sensors [7], strain sensors [8], breath rate monitoring [9], biosensors [10], EMI shielding [11], emotion sensor [12], and optical fibers [13]. Detection of toxic gases and monitoring of air quality are essential for both environmental safety and energy saving. The World Health Organization in its 68 world health assembly meetings held in the month of May 2015 reported that 3.7 million deaths every year are explicable to ambient air pollution [14]. Gas sensors are the key components to trace the presence and concentration of various gases in the surrounding. Smart, low cost, flexible, lightweight, low power consuming, portable, and reliable gas sensors are in extensive demand in flexible and wearable sensing technology. Such smart and flexible materials or composites can be manufactured by depositing conducting materials, such as carbon nanotube (CNT), graphene oxide (GO), conducting polymer, and metallic nanoparticles, onto the surface of nonconducting flexible substrates, such as paper, plastic films, and textiles materials, such as polyester, nylon, polypropylene, and cotton. The main hurdle in manufacturing stable, smart, and flexible composites is the weak adhesion between conducting material and flexible substrate as most of the flexible substrates, such as polyester, nylon, polypropylene, and PET film, have low surface energy and poor wettability. Hence, it is necessary to uplift their surface energy and wettability for better adhesion with conducting material to produce stable composites. Various studies revealed that the characteristics of polymeric flexible substrates could be altered by surface modification [15]–[19]. There are many surface modification techniques, such as wet chemical processing, mechanical abrasion, flame treatment, enzymatic surface modification, sonication, and plasma processing [20], to improve the surface properties of flexible polymeric films and textile substrates. Out of these mentioned techniques, plasma processing is a novel method of surface modification. Plasma irradiation on polymeric flexible surfaces is the most extensively used method for cleaning from adsorbed impurities, etching, activation by generating new polar functional groups, cross-linking, and effective modification of polymer surface [21]–[23]. It alters the surface properties without affecting the bulk properties of the substrate. It is basically a dry procedure and uses a very low amount of chemicals for surface treatment unlike wet chemical processing, which requires a huge amount of chemicals and disposal of polluted water, which are not safe for the environment. Another attractive

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feature of plasma surface treatment is that its processing time is shorter than other available techniques. Once the surface of the flexible substrate is treated with plasma, conducting material can be deposited on its surface to produce stable and durable smart flexible composites. There are many methods of deposition of conducting material on flexible substrates, such as coating [24]–[26], chemical vapor deposition [27], [28], physical vapor deposition [29]–[31], printing [32]–[34], self-assembly [35], [36], *in situ* polymerization [37]–[39], and *in situ* growth [40], [41]. Using these methods, a novel smart and flexible composite can be fabricated by grafting or depositing sensing material on the plasma-pretreated flexible substrate. Hence, plasma could be an innovative intermediate component between sensing material and flexible substrate to produce inventive smart and flexible material, which could be useful in many sensing applications. In recent years, the progress of sensors using plasma-treated flexible polymeric materials has resulted in a very encouraging technology for applications in gas sensing. In this review, we concentrated on such very recent developments. First, this review analyses various kinds of gaseous plasmas used for flexible polymeric surface treatment and their impact on the surface properties of the flexible substrates. The objective of this review article is to provide an analytical and up-to-date overview of the applied science of plasma surface modification for flexible polymers with reference to wettability, surface chemistry, and adhesion and to recapitulate the development of plasma-enriched smart and flexible materials for various gas sensing applications and to visualize their future growth.

II. TYPES OF PLASMAS FOR POLYMERIC FLEXIBLE SURFACE MODIFICATION

Plasma is an ionized gas. The gaseous plasma is also described as the fourth state of matter and electrically conducting medium of electrons, radicals, ions, radiations, and neutral species. Hence, gaseous plasma is a composition of reactive and energetic species. Plasma is created when the energy in the form of thermal, electromagnetic radiation, or electric field is applied to the gas. In the laboratory, various types of plasmas are produced using electric energy. The electrons gain energy from the electric field, which, in succession, passes on the fraction of this energy to neutral gas molecules via collisions. Ionization produced by electron collision is the main initiator of charged species in the plasma. The classification of plasma for surface modification of polymeric flexible substrates is presented in Fig. 1.

A. Thermal Plasma and Nonthermal Plasma

Plasmas are categorized as thermal plasma (hot) and nonthermal (cold) plasma on the basis of the difference in temperature between electrons and ions in it. Thermal plasmas are considered to be in thermal equilibrium and are having temperatures around 4000 K or more. In thermal plasma, the temperature of electrons and other species is the same. However, in nonthermal plasma, electrons have remained at a higher temperature than other species; hence, nonthermal plasmas are in thermally nonequilibrium [42], [43]. Because

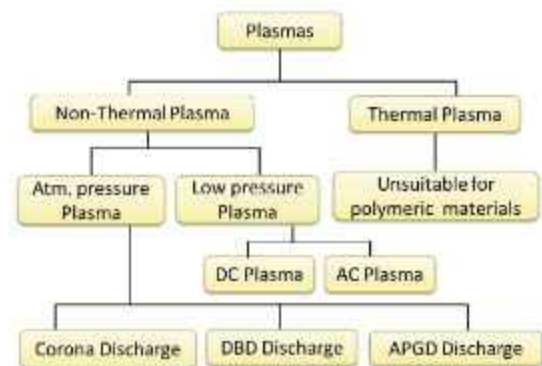


Fig. 1. Categorization of common plasmas used for surface modification.

of high temperature, thermal plasma is not considered to be suitable for polymeric flexible substrates, whereas the temperature of the nonthermal plasma is around ambient temperature, so it can be employed for surface modification of polymeric flexible materials [44].

B. Atmospheric Pressure Plasma

Nonthermal plasmas are further classified as atmospheric pressure plasma and low-pressure plasma. As the name suggests, atmospheric plasma is created at normal pressure (1 atm), while low-pressure plasma is created at low pressure ($\sim 10^{-2}$ – 10^{-3} mbar). Atmospheric pressure plasmas are further categorized as corona discharge, atmospheric pressure glow discharge (APGD), and dielectric barrier discharge (DBD). Corona discharge is produced between two parallel electrodes, out of which one electrode has a knife shape and the other has a cylindrical shape, and these electrodes are separated by a very small distance of the order of 0.1 cm. A very high dc electric field of the order of 15 KV is to be maintained between electrodes to trigger corona discharge. Corona discharge is inhomogeneous and inadequate for textiles as plasma density reduces dramatically with distance from a point of generation. Susan *et al.* [45] have investigated the effects of irradiation on the hydrophilicity of cotton and polyester fabrics by corona discharge created by high dc voltage with multiple points to plane electrodes. Xu and Liu [46] have studied hydrophilic and wicking properties of polyester fabric treated by corona discharge at discrete voltages. DBD discharge is produced between two parallel electrodes separated by a very small distance, and a high electric field of around 20 KV is maintained across them. A dielectric material is used to shield the electrodes to get rid of the short circuit. A small alternating current with low frequency (1–20 kHz) is to be applied to the system to trigger DBD plasma. DBD discharge is more homogeneous than corona discharge. Vu *et al.* [47] have generated DBD plasma between two parallel electrodes coated with ceramic and silicon with a high dc voltage of 10 KV and an alternating current frequency of 40 kHz across the electrodes. They have applied DBD plasma on polyamide fabric to study the consequence of particle size on Ag nanoparticle deposition [47]. Gasi *et al.* [48] employed hybrid DBD plasma treatment on polyamide fabrics to study its chemical, morphological,

and physical properties. They have generated DBD plasma between cylindrical ground electrodes insulated with silicone and a set of high voltage electrodes. The 50-kHz ac power was used to trigger DBD discharge [48]. APGD plasma is generally produced between two parallel electrodes narrowly separated by a few mm distance with low voltage, but the high frequency of the order of MHz is used to trigger discharge. No dielectric insulation is provided to the electrodes in this discharge as the transition from glow-to-arc is maintained by the electron trapping process promoted by the high-frequency applied voltage. Moreover, such high-frequency oscillation of a given voltage prevents a constant energy supply to an unlimited rise of current. Therefore, RF APGD is usually stable, and hence, it is inessential to apply dielectric insulation to the electrodes [49], [50]. A carrier gas, such as helium and nitrogen, is used to produce this plasma. APGD plasma is relatively uniform and stable than DBD plasma. Guimond *et al.* [51] have applied APGD plasma created by using nitrogen with high voltage and supply frequency 6 kHz on the surface of biaxially oriented polypropylene to study its surface properties. Hu *et al.* [52] employed large area APGD for surface treatment on wool to study its wettability property.

C. Low-Pressure Plasma

Low-pressure plasma is the most usual discharge to modify the surface of flexible polymeric substrates. It can be generated and maintained using less power compared to atmospheric pressure plasma. For a constant separation between electrodes and a given gas, there exists an optimum pressure called critical pressure at which a minimum voltage or power is adequate to start discharge. After this critical value of pressure, the voltage required for discharge increases rapidly with pressure because, as the gas pressure is increased, the mean free path of electrons is reduced; therefore, the voltage requires to be higher to speed up the electrons adequately to ionize atoms of the gas. As the pressure is decreased, the mean free path of the ions increases, so the electric power required to give an ion the energy to create new ions by collision is decreased. Hence, the voltage needed to begin discharge first decreases up to a fixed minimum value. For a very high vacuum, the mean free path is very high. Hence, the number of collisions and the production of new ions will be very low, so it becomes very difficult to ignite discharge even with a very large value of voltage [53]. The low-pressure plasma system requires a very small amount of gases/monomers for surface modification compared to the atmospheric pressure plasma system. More uniform modifications can be achieved using low-pressure plasma compared to atmospheric pressure plasma. It requires the vacuum system to create low pressure inside the plasma chamber. A typical low-pressure plasma system is presented in Fig. 2.

It consists of two metallic electrodes separated by an appropriate distance of a few centimeters enclosed in an airtight PYREX glass chamber known as the plasma chamber. There are two inlets and two outlets to the chamber. One inlet is used for air admittance, and the other inlet is used for non-polymerizing gas admittance. One outlet is connected to the Pirani gauge to monitor pressure inside the plasma chamber.

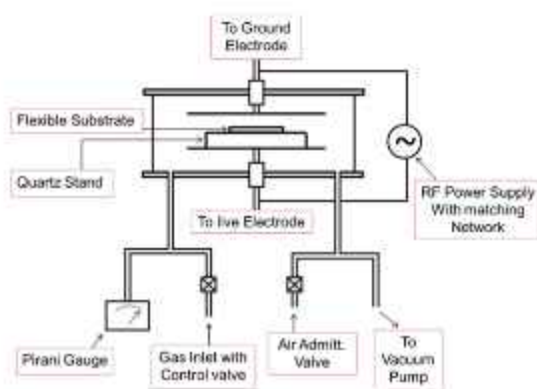


Fig. 2. Low-pressure plasma system for surface modification [54].

Another outlet is connected to a rotary vacuum pump that is used to create a vacuum inside the chamber. A quartz stand is used to be placed on the lower electrode, and a flexible polymeric substrate is to be kept on the quartz stand for surface treatment. Low-pressure plasma can be triggered by using a dc or ac electric field connected across the two electrodes. To create dc plasma, high dc voltage is to be maintained between two electrodes. To create ac plasma, a lower voltage compared to dc is required. The radio frequency of 13.56 MHz or the microwave frequency (2.45 GHz) is generally used to trigger plasma. Gregorski and Pavlath [55] employed low-pressure (1 mm of Hg) RF oxygen and nitrogen plasma treatment on wool fabric for 5–90 min at power ratings of 50 and 100 W to check their effectiveness on various surface properties. Oktem *et al.* [56] have treated polyester and polyamide fabrics with low-pressure (0.5 torr) RF oxygen, air, water, acrylic acid, and argon plasma at a power of 5 to 20 W and the treatment time of 1–90 min to improve wettability, soil resistance, and dyeing properties. Mortazavi *et al.* [57] have applied low-pressure (0.1 mbar) N_2/O_2 dc plasma on the surface of polypropylene films for the investigation of wettability and surface energy of polypropylene films. Inbakumar *et al.* [58] have treated cotton fabrics with low-pressure dc plasma in argon to study the wicking behavior of cotton fabrics with reference to discharge power, treatment time, and pressure. Rashidi *et al.* [59] employed low-pressure (0.5 torr) helium–air dc plasma on the surface of polyester and cotton fabrics to study the effect of treatment time on the wettability and surface resistivity of cotton and polyester fabrics.

III. EFFECT OF PLASMA ON A FLEXIBLE POLYMERIC SURFACE

When plasma of nonpolymerizing gases, such as oxygen, nitrogen, argon, helium, and air, is bombarded on the surface of polymeric material, energetic species (electrons, radicals, neutrals, and ions) and UV photons in plasma distribute their energies on the surface, and some highly energetic species break chemical bonds and chain scission occur on the surface of the polymer [60]. The depth of substrate affected by plasma can be typically up to few nanometers [61]. The resulting changes on the surface depend on the type of polymeric substrate and working gas used to generate plasma. Various

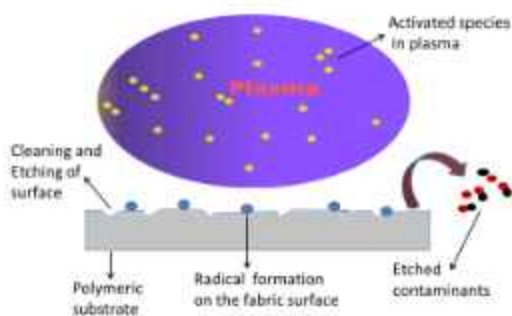


Fig. 3. Cleaning and etching of polymeric surface due to plasma.

gases are used for the plasma treatment of polymeric flexible substrates. Each gas creates a distinctive plasma constitution and results in discrete surface characteristics. For example, plasmas created with argon, oxygen, helium, and nitrogen can energize the surface through etching or ablation. The proportion of surface etching is controlled by the energy of ions, which successively depends on input power and the working pressure. Substantially, three main effects can be achieved depending on the conditions of treatment, namely, surface cleaning, surface etching, and surface activation.

A. Surface Cleaning

In surface cleaning, the elimination of organic contaminants, such as oxides, greases, oils, and other impurities, takes place from the surface of the substrate through the ablation process (see Fig. 3). The bulk properties of the substrate remain unaltered. Argon gas plasma is generally used for surface cleaning because of its low price, chemical inertness, and, most importantly, high ablation efficiency [62].

Inert gases, such as helium and argon, and oxygen or nitrogen plasmas are predominantly used for the surface etching of polymers. Many parameters, such as substrate nature and position, plasma composition, gas flow rate, and plasma power, affect the etching rate. Zeuner *et al.* [63] and Sprang *et al.* [64] have reported that polymer surface etching rate is enhanced with the increase in discharge power. Due to high power, the acceleration of high-energy species in plasma toward the substrate increases, which results in strong etching of the surface. Matthews *et al.* [65] have found that plasma of gaseous mixture of oxygen and helium together acquired superior etching rate of PET film compared to that of only helium plasma.

B. Surface Activation

Surface activation involves the insertion of new polar functional groups onto the surface treated with the plasma of nonpolymerizing gases, so as to achieve improvement in surface energy, which is responsible to enhance the wettability property of the surface. The attack of energetic species in plasma onto the surface breaks covalent bonds and generates free radicals on the surface. Active species in plasma react with these free radicals to create different active polar functional groups, such as carbonyl, hydroxyl, carboxyl, and amine

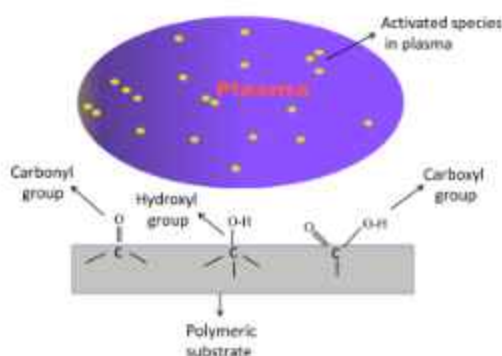


Fig. 4. Surface activation of the polymeric substrate due to plasma.

groups on the surface of the substrate (see Fig. 4). Oxygen plasma is generally used to increase the surface energy of the substrates by introducing oxygen-based polar functional groups and hydrophilic activity on the surface, whereas plasma of carbon tetrafluoride provides antiadherence property to the surface due to fluorination. The main purpose of surface activation is to improve the surface energy of the substrate, so as to enhance wettability and adhesion [66].

C. Plasma Treatment for Improvement in Wettability

Change in wettability or surface energy depends on many parameters, such as type of plasma, plasma pressure, nature of gas, plasma power, plasma treatment time, and nature of the substrate. Estimation of wettability of plasma treatment is normally done with the help of wicking and wetting time [67]. Xu *et al.* [68] have applied atmospheric pressure pure helium and oxygenated helium plasma jet on the surface of wool fabrics and observed a reduction in contact angle, but the rate of reduction was found to be more in oxygenated helium plasma than that of pure helium plasma. A higher rate of increment in wettability was might be because of a higher concentration of carbonyl and carboxyl groups on wool fabric surface due to oxygenated helium plasma than pure helium plasma [68]. Zemljic *et al.* [69] have reported a reduction in water contact angle of viscose fabric from 66° to 15° when it is exposed to low-pressure oxygen plasma and observed the improvement in chitosan absorption ability of the fabric leading to enhanced antimicrobial activity. The contact angle was also not found to be changed much even after 96 h of aging [69]. Bakhshzadmahmoudi *et al.* [70] treated polystyrene surface by cold atm. pressure DBD argon plasma jet with rf power supply with different power and treatment time to obtain hydrophilic surface. They observed that polystyrene surface changed to hydrophilic from hydrophobic after 20 sec. plasma irradiation and surface wettability increased with time. Such improvement in surface wettability was due induction of oxygen-based functional groups in polystyrene after argon plasma treatment [70]. Hossain *et al.* [71] have applied Ar/O_2 , He/O_2 plasma on polyester fabrics and reported that hydrophilicity strongly depends on the type of yarn and weave construction. They found that the wettability increment was much higher in loosely structured fabrics, such as knit fabrics, than tightly woven fabrics. This outcome was clarified by the fact that

plasma particles caused deeper penetration in the case of loosely structured fabrics than tightly woven fabrics, resulting in higher hydrophilicity [71]. Armagan *et al.* [72] treated polypropylene nonwoven fabrics with low-pressure RF oxygen plasma and found that increasing discharge power and the time of treatment resulted in higher wettability of treated PP fabrics. Improvement in wettability occurred due to the creation of oxygen-based polar functional groups, such as carbonyl and carboxyl on PP fabric surface, as confirmed by ESCA results [72]. Oliveira *et al.* [73] applied double-barrier dielectric (DBD) plasma dosage on polyamide fabrics and found that higher dosage drops the contact angle (increases wettability) and adsorption time of dye solution and water. Results obtained were due to the formation of more polar functional groups on polyamide surfaces treated with a higher dosage of DBD air plasma. They also observed that there were no appreciable variations in mechanical properties of polyamide fabrics after plasma treatment, validating that plasma treatment can alter surface properties by keeping its bulk properties unchanged [73]. Hrycak *et al.* [74] treated polyethylene surfaces using atmospheric pressure microwave ($f = 2.45$ GHz) argon plasma for the evaluation of change in wettability or surface energy of PE, and they found 75% excess reduction in water contact angle on polyethylene surface and confirmed the improvement in wettability after plasma treatment [74]. Peng *et al.* [75] investigated wettability distribution on polydimethylsiloxane (PDMS) surface treated with atmospheric pressure argon plasma jet at distinct flow rates, and they found that a higher flow rate leads to a better and uniform wettability distribution. This result was justified by the fact that a greater flow rate promotes turbulent flow and brings more H₂O molecules from air into plasma, which leads to a large amount of OH radicals resulting in more silanol (Si-O-H) groups on the PDMS surface, which makes the treated surface more hydrophilic [75]. Pragma and Deogaonkar-Baride [76] applied atmospheric pressure He/O₂ DBD plasma treatment on the surface of different interlacement structures of the polyester fabric prior to polypyrrole (PPy) deposition via *in situ* chemical polymerization to study the effect of yarn interlacement pattern on surface conductivity of PPy-polyester fabrics, and they found higher uptake and better retention of PPy on plasma-pretreated fabrics than that of untreated one using abrasion fastness test for different rubbing cycles. Better deposition and retention of PPy on plasma-pretreated fabrics can be explained by the fact that the He/O₂ plasma treatment makes the fabric surface rougher and introduces oxygen-based polar functional groups, which, in turn, is responsible for higher and better adhesion of PPy with plasma-pretreated polyester fabrics [76]. Nimbekar and Deshmukh [77] have treated polyester fabrics with low-pressure RF oxygen plasma and then grafted PPy on plasma-pretreated polyester fabrics using chemical polymerization. They found better and uniform grafting of PPy on plasma-pretreated polyester than that of untreated one. They could not measure water contact angle on plasma-treated polyester fabrics because of very high wettability after oxygen plasma treatment. Better and uniform grafting of PPy on plasma-pretreated fabrics was attributed to the formation of various

oxygen-based polar functional groups on the surface of polyester responsible for high wettability due to oxygen plasma treatment, which was confirmed by XPS analyses [77]. Some important reports on improving surface energy using plasma are presented in Table 1.

IV. GAS SENSING MECHANISM AND APPLICATIONS OF CONDUCTING MATERIAL GRAFTED ON PLASMA-PRETREATED FLEXIBLE SUBSTRATES

Many volatile organic chemicals (VOCs) and gases can be detected and identified with the help of polymeric flexible sensors. Their gas sensing mechanism normally based on electrically sensitive materials, such that the resistance or electrical conductivity of the polymeric substrate, changes after interaction of analyte vapors with them. High sensitivity, stability, selectivity, low cost, low response and recovery times, low operating temperature, and low detection limit are the salient features of the gas sensor. A gas sensor might not have these features together at a time. Moreover, a sensor does not require all these features at once for real applications. There are many sensing materials, such as GOs, CNTs, and conducting polymers, which can sense various types of vapors and gases. GO comprises a layer of graphene, enhanced with functional groups, such as carbonyl, hydroxyl, carboxyl, and epoxy. Primarily GO behaves like a p-type semiconductor, and after exposure to target gas, molecules of gas interact with functional groups of GO and insertion or removal of electrons from the semiconductor take place depending on the nature of the gas. Accordingly, the change in the resistance of the sensing layer occurs. CNTs are basically a single-layer graphene sheet rolled like a tube having a diameter of the order of several nanometers and length in the range of 0–100 nm. These are also known as single wall CNTs (SWCNTs). Multi-walled CNTs (MWCNTs) comprise many layers of graphene sheets. CNTs interact with target gas through donor–acceptor or Van der Waals interaction. Moreover, the surface-to-mass ratio of CNTs is high, which expands the area of contact between target gas molecules and the sensing layer, and results in high sensitivity of the sensor. Most of the doped conducting polymers, such as polyaniline (PANI) and PPy, are p-type. When target gas molecules interact with such p-type conducting polymer, its resistance increases or decreases depending on the nature of the gas whether it is a reducing or oxidizing agent. For example, ammonia gas that has a reducing character donates electrons to the conducting polymers and, thereby, reduces the number of positive charges on the polymer chain, which increases the resistance. For oxidizing gas (CO and NO₂) interaction, the resistance of the conducting polymer decreases. Metal oxide-based semiconductor materials cannot be deposited on polymeric flexible substrates for gas sensing as most of the metal oxide-based semiconductors are operated at high temperatures, and polymeric substrates have low operating temperatures [89].

The response or sensitivity of the sensor for a particular gas is determined using the following:

$$S = \frac{R_g - R_a}{R_a} \times 100\% \quad (1)$$

TABLE I
IMPORTANT REPORTS ON PLASMA SURFACE MODIFICATION TO IMPROVE WETTABILITY/SURFACE ENERGY

Plasma	Gases	Substrate	Result	Ref.
Low pressure (100 Pa.) RF	N ₂ , Ar, O ₂ , He, CH ₄ , and H ₂	PET film	Wettability has been improved by Ar, N ₂ , He, O ₂ , H ₂ plasma treatment while no effect has been observed on wettability after CH ₄ plasma treatment.	[78]
Low pressure (0.4 mbar) RF	Ar/O ₂	PET film	Wettability is directly proportional to treatment time up to certain threshold value. Higher discharge power reduces the treatment time.	[79]
Low pressure	O ₂	Polyamide-6 nanofibers	Plasma treatment notably improved the surface wettability of polyamide-6 fibers due to increase in O ₂ content from 13.5 % to 18.4 % & 23.8 for 60 s and 300 s respectively.	[80]
Atm. pressure	N ₂ /He/C ₂ H ₂	Nylon-6 films	Plasma treatment makes the surface rougher and increases the surface energy due to insertion of oxygen based functional groups.	[81]
Atm. pressure & low pressure (20 pa.)	Air, O ₂	Polystyrene (PS) Polypropylene (PP)	carboxyl, carbonyl, and Hydroxyl groups were introduced onto the surface of the PS and PP films by plasma treatment. Atm. pressure plasma produced significant hydrophilic effects on PP film.	[82]
Atm. pressure DBD & low pressure (0.01mbar)	Air, Ar / O ₂	Cotton and Wool fabrics	Gravimetric analyses showed decrease in mass for small treatment time which was associated with cleaning effect after plasma treatment. Weight loss in cotton (vegetal) was lower than that of wool (animal) due to greasy cleaning.	[83]
Atm. pressure plasma Jet (APPJ)	He, O ₂	Nylon-6 films	Atm. pressure He and He/O ₂ plasma enhanced the surface energy of Nylon-6 films by increasing oxygen contents, surface roughness and hydrophilic polar groups.	[84]
Low pressure (0.45 Torr) RF	CO ₂ , O ₂ , (CO ₂ +O ₂)	PET fabrics	Out of CO ₂ , O ₂ & CO ₂ + O ₂ plasma, CO ₂ and CO ₂ + O ₂ provided lower degradation and higher wettability to the PET fabrics as compared to O ₂ plasma.	[85]
Low pressure (50 pa.) RF	Ar / O ₂	Nylon 66 fabrics	Sheet resistance of CNT coated nylon fabrics was reduced from 4.9 kΩ/sq. to 2.0 kΩ/sq. for plasma pre-treated Nylon 6 fabrics.	[86]
Low pressure (0.08 mbar) RF	Ar, O ₂ and (Ar + O ₂)	PVDF films	Results showed roughness increment and surface activation due to plasma treatment on PVDF film to improve adhesion of PPY with PVDF. Ar + O ₂ plasma gave better bonding than alone Ar or O ₂ plasma.	[87]
DC glow discharge (0.2 mbar)	Ar, N ₂	Cotton fabrics	Polypyrrole coated cotton fabrics pre-treated with argon plasma showed higher conductance than that of nitrogen plasma pre-treated fabrics.	[88]

where R_a and R_g are the sensor resistances when subjected to air and ammonia gas, respectively.

Selectivity is another important characteristic of a gas sensor. Selectivity refers to a property that decides whether a sensor can respond specifically to a single analyte or a group of analytes.

A. Gas Sensing Mechanism of Carbon Nanotubes

CNTs basically act as a p-type semiconductor. Resistance of CNT changes when electron donor (ammonia) or acceptor (nitrogen dioxide) gas molecules interact with it. Transfer of electrical charge is the crucial sensing mechanism at room temperature. When target gas molecules interact with CNT, they get adsorb onto the surface of CNT causing electron transfer and altering the electrical conductance of the CNT structure. Fig. 5 schematically represents the modification of conductance of CNT when NH₃ or NO₂ gas molecules come in contact with the CNT structure.

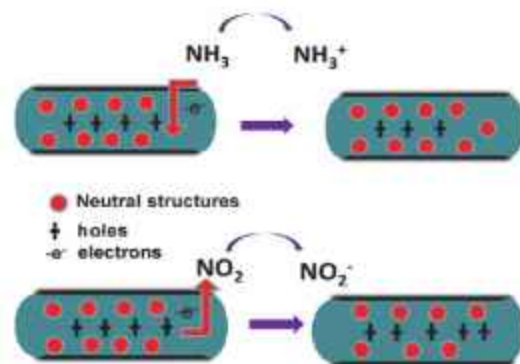


Fig. 5. Gas sensing mechanism of p-type CNT with oxidizing or reducing type gases [90].

Ammonia donates electrons to the carbon nanostructured film. These electrons recombine with hole carriers, thereby reducing the concentration of charge carriers and,

consequently, increasing the electrical resistance of CNT. When NO_2 gas molecules interact with CNT, the opposite mechanism happens, and consequently, the electrical resistance of CNT reduces [90]. To increase the sensitivity or response of the resistive gas sensor, coating of metal oxides, such as TiO_2 , SnO_2 , or conducting polymers, such as PANI and PPy, on CNTs has been extensively used [91].

B. Gas Sensing Mechanism of Graphene Oxides

The sensing process of GO-based gas sensors depends on the binding between gas molecules and functional groups of GO. In fact, functional groups, such as hydroxyl (-OH), epoxy (=O), and carboxyl (-COOH), improve the adsorption of gas molecules into the GO sheet. Better gas detection occurs when binding energy between target gases and GO is higher. The adsorption of gas molecules on the GO sheet also depends on the nature of functional groups of GO. In the case of NH_3 gas detection, the adsorption of NH_3 gas molecules on the GO sheet is independent of molecules orientation but depends on epoxy and hydroxyl functional sites. Improvement of adsorption energy due to -OH group is higher than that of =O group. Hence, for adsorption of NH_3 gas on GO surface, the contribution of -OH group is more than =O group [92], [93]. Basically, GO behaves as a p-type semiconductor. When target gas interacts with the GO sheet, gas molecules bind with functional groups of GO, thereby inserting or removing electrons from the GO sheet, which, in turn, alters the conductivity of the GO layer. Sensitivity or response of GO depends on binding energy between functional sites of GO and molecules of the target gas, and the manufacturing process of GO that controls the number of functional groups on GO and concentration of target gas [89]. The gas sensitivity of GO can be enhanced by chemical alteration using metals, metal oxides, polymers, and CNTs. For example, graphene may be enriched with nanoparticles of metals such as palladium to improve its performance. Such sensors showed 32.9% sensitivity for 1000 ppm of hydrogen [31].

C. Gas Sensing Mechanism of Conducting Polymers

Gas sensors based on conducting polymers transform the concentration of target gas into a change in resistance or conductivity of conducting polymer. The doping level of conducting polymer decides its conductivity, and it can be easily altered by incorporation or removal of electrons because of oxidation–reduction reaction between target gas and sensing polymer. When gas molecules interact with the sensing layer of conducting polymer, they get adsorbed at its surface, which results in a change in conductivity. When the target gas is removed, gas molecules get desorbed, and the sensor comes again to its original state. Most of the conducting polymers, such as PANI and PPy, exhibit a p-type nature. When electron acceptor gases, such as NO_2 and CO , interact with such p-type conducting polymer sensing layer, they remove electrons from the polymer backbone, and as a result, the doping level and the conductivity of the polymer are increased. An opposite mechanism will take place when the electron donor gases interact with such a p-type polymer. Ammonia (NH_3) acts

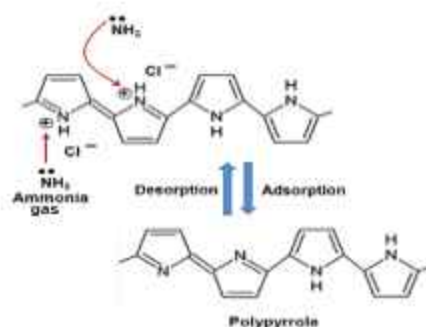


Fig. 6. Gas sensing mechanism of p-type PPY with reducing type gases [95].

as an electron donor; when ammonia gas interacts with PPY, it donates its lone pair electrons to the PPY chain (see Fig. 6), thereby reducing positive charge carriers, and as a result, a decrease in the conductivity or increase in the resistance of PPY layer occurs. When ammonia gas is removed, the desorption of ammonia gas molecules takes place, and PPY recovers its initial state [94], [95].

Barisci *et al.* [96] fabricated an array of sensors based on PPY (PP) polymers prepared using various dopants to recognize and calibrate BTEX gases. A group of eight polymers was found to have the ability to identify the analyte gases with adequate sensitivity and consistency. The PP sensor doped with T1r had a maximum response, whereas, for SB doped PPY sensor, the response was low for BTEX gases. Reproducibility of PP prepared using dopants MS, SB, and NDS was comparatively low. The change in electrical resistance of the PP sensor was attributed to dipole interactions or partial electron transfer between PP and BTEX gases modifying the work function of the polymer [96]. Crowley *et al.* have synthesized thin-film sensors made up of PANI nanoparticles using inkjet printing, and sensors were found to be very sensitive to ammonia gas at room temperature within a 1–100-ppm concentration region. The response time of the sensor was also observed to be decreased after heating. PANI experiences an increase in the resistance or a decrease in the conductivity when interacting with ammonia gas. Such an increase in the resistance results from deprotonation of amine groups in PANI emeraldine salt and transforms it to emeraldine base structure, and the reverse reaction takes place when ammonia is removed from emeraldine PANI base converting it to its initial salt form again [97]. The reaction mechanism between PANI and ammonia is presented in the following [97], [98]:



The increase in conductivity of the PANI film was observed when it was exposed to carbon monoxide (CO) gas. The positive charge at the carbon atom in the resonance structure of $^+C \equiv O^-$ will remove lone pair electrons at amine nitrogen, and hence, amine nitrogen in PANI acquires a positive charge and ultimately increases the positive charge concentration on PANI backbone (see Fig. 7); as a result, the rise in conductivity occurs. Another interpretation of the behavior of PANI to CO gas depends on oxidation–reduction reaction. Reduction of barrier height at the grain boundary is responsible for lowering of the resistance of PANI [99]–[103].

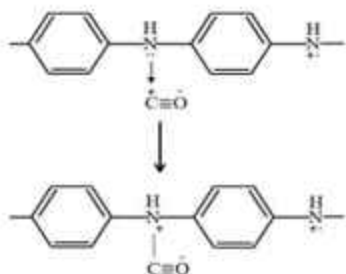


Fig. 7. Sensing mechanism of PANI for carbon monoxide gas [101].

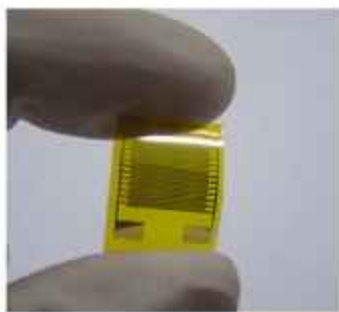


Fig. 8. Optical micrograph of the plasma-enhanced SWCNT sensor deposited on the flexible PI substrate [101].

After plasma treatment on the surface of the flexible polymeric substrate, sensing materials, as mentioned above, can be grafted onto it using various techniques to produce smart and stable flexible composites, which can be used in many gas sensing applications. Wang *et al.* [102] have manufactured single-walled CNT (SWCNT) integrated with the oxygen plasma-pretreated flexible polyimide (PI) substrate (see Fig. 8) using lithography and liftoff techniques to detect nerve agent stimulant DMMP vapors at the room temperature. The sensor showed a high magnitude of response linearity, stability, selectivity, reproducibility, and a low detection limit of 1 ppm with a sensitivity of 3.6% toward DMMP vapors at room temperature [104].

Crowley *et al.* [103] have fabricated a PANI-CuCl₂ sensor on a flexible PET substrate using a piezoelectric inkjet and screen printing for the detection of H₂S gas. They observed a linear response between current and concentration of H₂S gas over the region of 10–100 ppmv and the detection limit of 2.5 ppmv [103]. Kinkeldei *et al.* [104] developed an electronic nose comprising of four carbon/polymer (PIB, PS, PVP, and PVBU) gas transducers fabricated on oxygen plasma-enriched PI and polyethylene naphthalate (PEN) flexible substrates using spin coating and liftoff techniques. These fabricated substrates were cut into strips and then woven into the textile to produce a smart textile to identify different vapors. Initially, they used a PVBU sensor to detect acetone vapors, and they found a detection limit of 50 ppm of acetone for the PVBU sensor. The four-sensor-based smart textile was exposed to four different vapors (toluene, acetone, IPA, and methanol), and they observed the repeated rise of resistance of all sensors. Distinct response pattern was observed for each vapor as methanol > IPA > acetone > toluene for PVP > PVBU > PS > PIB.



Fig. 9. Photograph of PANI grafted on the plasma-pretreated flexible polyester fabric.

PVP showed the highest response from methanol, whereas the effect of toluene was small. PIB showed some response for toluene but very little for methanol [104]. Wan *et al.* [105] have fabricated a flexible gas sensor by synthesizing a nanocomposite film via *in situ* chemical oxidative polymerization of aniline in a fluorinated MWCNT suspension simultaneously grafted onto oxygen plasma-pretreated flexible polyethylene terephthalate substrate. They have used this flexible sensor for ammonia gas sensing for different concentrations. They found higher resistance sensitivity for PANI/FMWCNT composite sensors than PANI aggregates and FMWCNT film. They also studied the sensitivity of the PANI/FMWCNT sensor with reference to reaction time, and they found the highest sensitivity for 8-h reaction time compared to the films polymerized for 6, 10, and 12 h. They also reported much higher selectivity for PANI/FMWCNT flexible sensor toward ammonia gas in comparison with other VOCs (ethanol, methanol, acetone, and isopropanol) [105]. Nimbekar *et al.* [106] synthesized a flexible sensor by grafting PANI on the surface of oxygen plasma-pretreated polyester fabric using chemical oxidative polymerization (see Fig. 9). They have used this flexible sensor for ammonia gas sensing. The PANI-polyester flexible sensor showed a good sensing response toward three different concentrations (10, 20, and 40 ppm) of ammonia gas. They have reported gas sensitivity 93%, 169%, and 249% for 10-, 20-, and 40-ppm concentrations of ammonia with response and recovery times of 132 & 234, 138 & 174, and 126 & 162 s, respectively [106].

Guo *et al.* [107] developed a smart and flexible sensor by simultaneously depositing graphene-PANI composite on oxygen plasma-enhanced PET substrate via polymerization of aniline in reduced GO solution. Sensor synthesized with the reaction time for 6 h showed a high sensing response toward different concentrations (10 ppb–100 ppm) of ammonia gas. It has shown fast response time (36 s) and recovery time (18 s) with sensitivity around 3 and 50 for 5- and 100-ppm ammonia gas concentrations, respectively [107]. Hiyato and Fisher [108] fabricated a SnO₂ nanoparticle grafted paper-based flexible gas sensor (PGS) to detect CO, benzene, and ethanol. They have treated PGS with low-pressure RF Ar/O₂ plasma and the sensor showed remarkably improved response to all target gases even at room temperature [108]. Zhang *et al.* [109] have manufactured DBD CF₄ plasma-modified MWCNTs deposited on the epoxy resin substrate. They evaluated MWCNTs film response toward SO₂ and H₂S. The sensitivity of the MWCNT film was found to be directly proportional to plasma treatment time and best sensitivity, and

TABLE II
COMPARISON OF IMPORTANT CHARACTERISTICS OF DIFFERENT PLASMA-ENHANCED FLEXIBLE GAS SENSORS

Sensor material	Sensing gas / gases	Selectivity	Sensitivity / Response	Response / recovery time (sec)	Detection limit	Ref.
SWCNT/Polyimide	DMMP, Methanol, xylene, hexane, chloroform, dichloromethane	DMMP	0.07 (1 % of saturated vapor conc.)	---	1 ppm	[102]
PANI-Polyester fabric	Ammonia	-----	0.93 (10 ppm)	132/234	---	[106]
Graphene/PANI/PET	Ammonia	-----	3 (5 ppm)	36/18	-----	[107]
PANI/CNT-PET film	Ammonia, methanol, ethanol, acetone, pyridine	Ammonia	6 (10 ppm)	85/20	-----	[112]
R _s -MWCNT film	Benzene	-----	2 (500 ppb)	60 /--	below 50 ppb	[113]
PEDOT/PSS-SWCNTS thin film	Ammonia	---	0.4 (2 ppm)	12/18	200 ppb	[114]
Pt-CNT film	CO, CH ₄ , H ₂	CO	0.04 (5 ppm)	-----	-----	[115]
PANI-Nylon 6 fabric	Ammonia	----	1.13 (10 ppm)	117/179	0.79 ppm	[116]
PPY-Nylon 6 fabric	Ammonia	-----	0.85 (10 ppm)	270/324	1.55 ppm	[116]
MWCNT film	H ₂ S, SO ₂	H ₂ S	0.09 (50 ppm)	-----	-----	[117]
PEDOT/PSS-SWCNT film	NH ₃ , CH ₃ OH, TMA, CCl ₄ , Toluene, C ₆ H ₆ , C ₂ H ₅ OH	TMA Ammonia Methanol	0.12 (10 ppm) 0.02 (10 ppm) 0.015 (10 ppm)	60/100 12/18 ---	1 ppb 200 ppb ---	[118]
SWCNT film	Ethanol	-----	1.51 (100 ppm)	54/97	-----	[119]
PPY/graphene-PET film	NH ₃ , NO ₂ , CH ₃ OH, Toluene, C ₆ H ₆ , C ₂ H ₅ OH, Naphthalene	NH ₃ , NO ₂	3.0 (100 ppb) 4.0 (100 ppb)	1/10 2/7	0.04 ppb 0.03 ppb	[120]
p-MWCNT/PANI film	NH ₃	-----	0.18 (100 ppm)	-----	-----	[121]

the stability of the film was observed at a 10-min plasma treatment time. They found that the sensitivity of plasma-enhanced MWCNTs sensors increases with gas concentrations (10, 25, 50, and 100 ppm). Plasma-treated MWCNT films showed better sensitivity compared to pristine MWCNT sensors toward SO₂ and H₂S [109]. Yoo *et al.* [110] have synthesized RF oxygen plasma-functionalized MWCNT/PANI composite film for ammonia gas detection, and they found better sensitivity for pf-MWCNT/PANI composite (1.853 × 10⁻³) than pristine MWCNT (7.5 × 10⁻⁴) and

pristine MWCNT/PANI composite (7.6 × 10⁻⁴). They also reported good linearity (0.996) for pf-MWCNT/PANI composite in the range of 0–100-ppm ammonia concentrations compared to pristine MWCNT (0.865) and pristine MWCNT/PANI (0.925) [110]. Santosh *et al.* [111] have entangled MWCNTs on a flexible foam substrate called polyurethane, and after argon and helium plasma treatment on MWCNTs, they have tested the gas sensing response of this flexible composite toward ethanol. They observed three and five times improvement in sensitivities of the samples, respectively, after helium

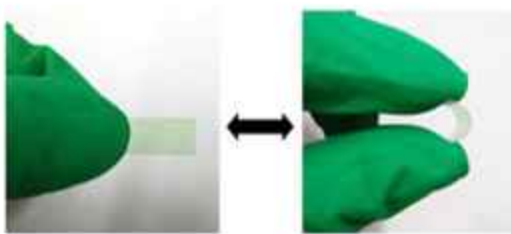


Fig. 10. Photograph of plasma-functionalized PANI/CNT composite deposited on the flexible PET substrate [112].

and argon atmospheric pressure plasma treatment compared to untreated samples for different concentrations of ethanol. They showed better stability in sensitivity of plasma-treated samples than untreated samples. They also reported an increment in response time but speedy recovery of plasma-treated samples. Enhancements in sensitivity and stability were attributed to plasma-assisted surface modification and the creation of polar functional bonds on the nanotube's surface, which is responsive to ethanol [111]. Xue *et al.* [112] have fabricated a flexible gas sensor by depositing PANI/CNT film on oxygen plasma-assisted PET substrate (see Fig. 10). They have studied sensing characteristics of this sensor for various gases (ammonia, methanol, ethanol, acetone dichloromethane, ethylene glycol, isopropyl alcohol, and pyridine), and they found this sensor to be highly selective for ammonia gas and the observed remarkable response of 600% for 10 ppm of ammonia concentration with the response and recovery times of 85 and 20 s, respectively [112]. Various plasma-assisted gas sensors with the comparison of their sensing properties are presented in Table II.

Hence, the CNTs, graphenes, metal nanoparticles, and conducting polymers are very useful materials for various gas sensing purposes, especially at room temperature in their pristine form, and to produce smart and flexible polymeric composites. It can be seen that gas sensing properties, such as sensitivity, selectivity, response, and recovery time, of the sensor can be enhanced by chemical functionalizing and decorating these materials with each other. Gas sensors based on pristine CNTs have disadvantages such as lack of selectivity and irreversibility. Such limitations can be overcome by the chemical functionalization of CNTs with conducting polymers, metal oxides, and metal nanoparticles [122]. Flexible gas sensors using zinc oxide, iron oxide, palladium, conducting polymer, and reduced GO-functionalized CNTs have been reported for the detection of ammonia, hydrogen, ethanol, nitrogen dioxide, methanol, and VOCs with improved gas sensing properties [123], [124]. Jeong *et al.* [125] have synthesized flexible CNT/graphene film to detect NO_2 gas. They reported 19% sensitivity for this film, whereas it was only 2% for the pristine CNT layer [125]. Conducting polymers have good sensing abilities at room temperature. Gas sensors based on conducting polymers generally have a lack of selectivity, and high response and recovery times. The sensing properties of conducting polymers are improved by functionalizing them with other materials. Wan *et al.* [105] reported enhancement in sensitivity (30%) of PANI-FMWCNT film for ammonia

gas (10 ppm), while the sensitivity of pristine PANI and pristine FMWCNT was observed to be 5% and 1%, respectively. Various flexible gas sensors using conducting polymers functionalized with many materials, such as reduced GO, iron oxide, tin oxide, CNTs, and platinum, are reported in the literature to improve gas sensing properties [38], [107], [112], [126]–[129]. It can also be seen that plasma treatment on flexible substrate gives better interfacial bonding of sensing material with the substrate to produce stable composites and also enhances gas sensing properties of a flexible sensor.

V. CONCLUSION

The reviewed study delineated different types of plasmas used for surface modification, their impact on the surface of polymeric flexible substrates, and the use of such plasma-enriched flexible polymers for various hazardous gas sensing applications. The potential of a gas sensor is assessed on the basis of its characteristics, such as high sensitivity, selectivity, flexibility, low detection limit, and fast response and recovery times. Still, there is no such sensing material that fulfills all these properties for an ideal gas sensor. It can be seen that plasma surface modification is an innovative method to improve these properties. Not much work has been done on the fabrication of plasma-modified gas sensors. This review would definitely be benefited the fabrication of plasma-induced smart and flexible gas sensors, which could be useful to detect various hazardous gases in the surroundings to protect the environment.

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