Compositional and optical properties of SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions deposited by HFCVD

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Abstract

In this work, non-stoichiometric silicon oxide (SiO$_x$) films and (SiO$_x$/SiO$_y$) junctions, as-grown and after further annealing, are characterized by different techniques. The SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions are obtained by hot filament chemical vapor deposition technique in the range of temperatures from 900°C to 1,150°C. Transmittance spectra of the SiO$_x$ films showed a wavelength shift of the absorption edge thus indicating an increase in the optical energy band gap, when the growth temperature decreases; a similar behavior is observed in the (SiO$_x$/SiO$_y$) structures, which in turn indicates a decrease in the Si excess, as Fourier transform infrared spectroscopy (FTIR) reveals, so that, the film and junction composition changes with the growth temperature. The analysis of the photoluminescence (PL) results using the quantum confinement model suggests the presence of silicon nanocrystal (Si-nc) embedded in a SiO$_x$ matrix. For the case of the as-grown SiO$_x$ films, the absorption and emission properties are correlated with quantum effects in Si-nc and defects. For the case of the as-grown (SiO$_x$/SiO$_y$) junctions, only the emission mechanism related to some kinds of defects was considered, but silicon nanocrystal embedded in a SiO$_x$ matrix is present. After thermal annealing, a phase separation into Si and SiO$_2$ occurs, as the FTIR spectra illustrates, which has repercussions in the absorption and emission properties of the films and junctions, as shown by the change in the A and B band positions on the PL spectra. These results lead to good possibilities for proposed novel applications in optoelectronic devices.

Keywords: HFCVD; PL; FTIR; Transmittance; Thermal annealing; Si-nc

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Background

Silicon is the semiconductor material predominant in the microelectronics industry. However, it has been long considered unsuitable material for optoelectronic applications [1], due to its indirect band gap, which means it is a poor light emitter. Though, after discovery of visible light emission at room temperature in the porous silicon by Canham [2] in 1990, many investigators have studied emission properties of materials with silicon compounds as the non-stoichiometric silicon oxide (SiO$_x$), which has gained increasing interest in the research community due to the formation of silicon nanocrystals embedded in the matrix, implying low-dimensional effects and thus determines an efficient emission of visible light, even at room temperature [3,4]. In the SiO$_x$ films, the absorption and emission properties are correlated with quantum effects in silicon nanoparticles, and also associated with defects [5]. From the technological standpoint, the average size of a silicon nanoparticle (Si-np) offers band gap widths, which opens the possibility to tune the emission of light using nanostructured thin films in novel optoelectronic devices. The goal of this work is to study and investigate the compositional and optical properties of SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions obtained by hot filament chemical vapor deposition (HFCVD) technique, as-grown and after a further annealing, in order to have a broad and solid view of the behavior of the material by varying the growth temperature and mainly its thickness, which opens the possibility for proposed novel applications in a future work.
Methods

SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions were obtained by HFCVD technique in the range of temperatures from 900°C to 1,150°C and deposited on quartz and n-type silicon (100) substrates with 1 to 10 Ω cm resistivity, using quartz and porous silicon as the reactive sources. The deposition time was 5 min for the SiO$_x$ films and of 10 min for the (SiO$_x$/SiO$_y$) junctions due to that the time for each film of the junction was of 5 min. The chemical reaction for the grown non-stoichiometric silicon oxide in the HFCVD technique is pictured in [6,7]. The relationship between the filament temperature (approximately 2,000°C) and the variation of the source-substrate distance (dss) of 4, 5, and 6 mm provides a change in the growth temperature (Tg) of 1,150°C, 1,020°C, and 900°C, respectively, which was measured with a thermocouple in the reaction zone. The SiO$_x$ films were deposited at 1,150°C, 1,020°C, and 900°C. Three samples of each kind have been studied. The (SiO$_x$/SiO$_y$) junctions were made with two films deposited at two different temperatures, obtaining six possible combinations, 1,150°C/1,020°C, 1,150°C/900°C, 1,020°C/1,150°C, 1,020°C/900°C, 900°C/1,150°C, and 900°C/1,020°C. Three samples of each kind have been studied. The changes in the dss and Tg, consequently, modify the silicon excess and defects in the non-stoichiometric SiO$_x$ films. The substrates were carefully cleaned with a metal oxide semiconductor standard cleaning process and the native oxide was removed with an HF buffer solution before being introduced into the reactor. The thermal annealing was made using a nitrogen atmosphere at 1,100°C for 1 h. Several spectroscopic characterization techniques were used. The film thickness was measured using a Dektak 150 profilometer (Veeco Instruments Inc., Plainview, NY, USA). Room-temperature transmittance of the SiO$_x$ films was measured using a UV–Vis-NIR Cary 5,000 system (Agilent

![Figure 1](http://www.nanoscalereslett.com/content/9/1/422)

**Figure 1** Thickness as function of Tg of the SiO$_x$ films (a) and (SiO$_x$/SiO$_y$) junctions (b).

![Figure 2](http://www.nanoscalereslett.com/content/9/1/422)

**Figure 2** Transmittance spectra as function of Tg of the SiO$_x$ films (a) and (SiO$_x$/SiO$_y$) junctions (b).
Technologies Inc., Santa Clara, CA, USA). The transmittance signal was collected from 200 to 1,000 nm with a resolution of 0.5 nm. FTIR spectroscopy measurements were done using a Bruker system model vector 22 (Bruker Instruments, Billerica, MA, USA). Photoluminescence (PL) response was measured at room temperature using a Horiba Jobin Yvon spectrometer model FluoroMax 3 (Edison, NJ, USA) with a pulsed xenon source whose detector has a multiplier tube, which is controlled by a computer. The samples were excited using a 250-nm radiation, and the PL response was recorded between 380 and 1,000 nm with a resolution of 1 nm.

Results and discussion
The thicknesses of the SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions as a function of the growth temperature (T$_g$) are shown in Figure 1 as box statistic charts, where the mean, maximum, and minimum values are shown, the width of the box denotes the standard deviation of the measurements. The T$_g$ and the deposition time affect the thickness of the SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions. When T$_g$ was decreased and the deposition time was less, thinner samples and a more uniform deposition were obtained due to lesser amount of volatile precursors likely to deposit. Figure 1b shows that the junctions have a better uniformity than the films (Figure 1a) because the standard deviation (width of the box) is less. This could be due to a possible ‘annealing’ of the first SiO$_x$ film deposited. In this case, the first layer has received annealing twice, which would certainly lead to a different formation. Then, these results show that the double annealing procedure and the order of layers have a very strong impact on the thickness. The tendency is that the more annealing, the thicker the layer. For the SiO$_x$ films, average thicknesses were obtained of 500 to 100 nm and for the (SiO$_x$/SiO$_y$) junctions of 750 to 200 nm.

Figure 2 shows the UV–Vis transmittance spectra of SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions as-grown deposited on quartz. All the samples exhibited a relatively high transmittance (>70%) between 600 and 1,000 nm. The change in the growth temperature produces a change in the stoichiometry of the SiO$_x$ films and thickness, which produces a shift of the absorption edge. In a previous work, where the SiO$_x$ films were characterized by XEDS [7], the information on change in stoichiometry of SiO$_x$ were reported; where $x$ for the film deposited at 1,150°C was 1.27, at 1,020°C was 1.81, and at 900°C was 1.66; these values are approximates. Figure 2a shows a similar absorption edge for 900°C and 1,150°C, while 1,020°C is shifted; therefore, a random result is obtained. Figure 2b shows that there is an extremely large difference...
between junctions; this seems to imply that the double annealing procedure and the order of layers have a very strong impact on optical properties. The overall trend seems as the more annealing is received, the less transparent the sample is in visible range - i.e., annealing is facilitating formation of absorbing/emitting states (defects or NCs) [8].

The approximate values of the energy band gap (Eg) were obtained by the relationship known as Tauc plot [9-11] as shown in Figure 3. The methodology for obtaining these approximate values of Eg has been described in a previous work [7], where the absorption coefficients \( \alpha(\lambda) \) were determined from transmission spectra and was obtained in the order of \( 10^3 \) to \( 10^4 \) cm\(^{-1}\). The Eg decreases as the growth temperature increases; the optical energy band gap of the SiO\(_x\) films is in the range of energies of 2.15 to 1.8 eV and of the (SiO\(_x\)/SiO\(_y\)) junctions is in the range of energies of 2.25 to 1.8 eV. When \( x \) decreases from 2.0 in a-SiO\(_x\), the valence band edge moves up, as the increased Si-Si bond states are gradually overlapped with the oxygen nonbonding states (ONS) and finally spread out into the Si valence band. Simultaneously, the conduction band edge also moves down. The final result is that the optical band gap decreases nonlinearly when Si concentration continually increases [5]. Therefore, we may assume that the Si excess increases as the growth temperature increases.

Figures 4 and 5 show the FTIR spectra from SiO\(_x\) films and (SiO\(_x\)/SiO\(_y\)) junctions as-grown and after a further annealing, respectively. The IR vibration bands are listed in Table 1 for the SiO\(_x\) films and in Table 2 for the (SiO\(_x\)/SiO\(_y\)) junctions. All the spectra show the absorption peaks characteristic of SiO\(_2\) which correspond to the vibration modes of stretching (1,082 cm\(^{-1}\)), bending (812 cm\(^{-1}\)), and rocking (458 cm\(^{-1}\)) of Si-O-Si [12,13].

Before thermal annealing, these peaks show a shift to lower wavenumber with decreasing of the growth temperature (Tg), indicating the change in the silicon excess. After thermal annealing, these shifts disappear and the position of the vibration modes corresponding to SiO\(_2\), thus indicating a phase separation. Before thermal annealing, a vibration band approximately at 885 cm\(^{-1}\), assigned to Si-H bending and Si-O of Si-O-H [14-16], appeared with a shoulder at 810 cm\(^{-1}\) related to the Si-O bending vibration band. When the Tg was higher, the intensity of the Si-O bending vibration band increased and shift at the same time that the peak at 870 cm\(^{-1}\) decreased, and the Si-O bending vibration band became more apparent. These bonds are present in the films due to the incorporation of hydrogen in the experimental process, but after the films were thermally annealed, the band at 885 cm\(^{-1}\), disappeared due to the desorption of hydrogen to high temperature and the characteristic absorption peaks of SiO\(_2\) were more noticeable. The Si-O stretching vibration band shifted to higher frequencies after thermal annealing indicating a phase separation and its width reduced due to an increment in the oxygen concentration.

### Table 1: IR vibration bands [12-17] of the SiO\(_x\) films before and after thermal annealing

| Vibration mode | Peak position (cm\(^{-1}\)) |
|---------------|---------------------------|
|               | As grown | Annealed |
| Si-O rocking  | 444      | 436      | 429      | 458      | 458       |
| Si-O bending  | 797      | 800      | 810      | 812      | 812       |
| Si-O stretching | 1,064   | 1,055    | 1,048    | 1,082    | 1,082, 1,082 |
| Si-H wagging  | 654      | 649      | 645      | 645      | 645       |
| Si-H bending  | 885      | 879      | 875      | -        | -         |
concentration. Also, all samples exhibit a peak around 645 to 664 cm$^{-1}$, which has been associated with Si-H wagging bonds or neutral oxygen vacancies [17]. These bonds are almost imperceptibly present in both cases.

Figures 6 and 7 show the PL spectra from SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions as-grown and after further annealing, respectively.

A wide PL spectrum with a similar shape to a Gaussian curve is observed for all the samples. For this reason, the deconvolution was applied to the PL spectra, with which several peaks were obtained as shown in Figure 8; each of the peaks represent the emission individually, which are the components of the PL spectra. Therefore, each peak has different origins.

In general, in the PL spectra of the SiO$_x$ films and (SiO$_x$/SiO$_y$) junctions as-grown, there are several emission bands covering a wide spectral range from 380 to 850 nm (violet-near infrared range). After further annealing, the PL spectra there are two main bands, band A of 380 to 495 nm (violet-blue range) with a relatively weak PL intensity and band B of 590 to 875 nm (orange-near infrared range). In this case, the green-yellow band disappears. The PL spectra of the SiO$_x$ films, as-grown (see Figure 6a) exhibit a higher PL intensity with respect to the PL intensity after further annealing (see Figure 6b). The PL spectra of (SiO$_x$/SiO$_y$) junctions as-grown (see Figure 7a) illustrate a relatively weak PL intensity with respect to PL intensity after further annealing (see Figure 7b). The origin of the PL emission is still object of debate; two models have been proposed. The first model relates the PL to quantum confinement effects (QCE) [18,19] in Si-nc’s. The second model attributes the PL to defects in the matrix associated with oxygen vacancies or defects in the interface SiO$_2$/Si-nc [16,19].

Figure 6a shows that the SiO$_x$ films, as-grown, have a maximum emission peak that suffers a blue shift and an increase in the intensity as the growth temperature decreases. This behavior could be related to the presence of Si-nc’s as was observed in a previous work, where the SiO$_x$ films were characterized by HRTEM [7], and the average size of Si-nc was obtained, which reduced along with the decreasing growth temperature. Therefore, in

| Vibration mode  | 1,150°C/1020°C | 1,150°C/900°C | 1,020°C/1,150°C | 1,020°C/900°C | 900°C/1,150°C | 900°C/1,020°C |
|-----------------|----------------|---------------|----------------|---------------|---------------|---------------|
| As grown        |                |               |                |               |               |               |
| Si-O rocking    | 447            | 444           | 442            | 442           | 440           | 440           |
| Si-O bending    | 808            | 803           | 800            | 800           | 797           | 796           |
| Si-O stretching | 1,067          | 1,063         | 1,063          | 1,055         | 1,055         | 1,054         |
| Si-H wagging    | 664            | 659           | 645            | -             | 652           | 651           |
| Si-H bending    | 883            | 883           | 880            | 879           | 879           | 877           |
| Annealed        |                |               |                |               |               |               |
| Si-O rocking    | 458            | 458           | 458            | 458           | 458           | 458           |
| Si-O bending    | 812            | 812           | 812            | 812           | 812           | 812           |
| Si-O stretching | 1,082          | 1,082         | 1,082          | 1,082         | 1,082         | 1,082         |
| Si-H wagging    | 645            | 640           | -              | 637           | -             | -             |
| Si-H bending    | -              | -             | -              | -             | -             | -             |

Figure 6 PL spectra from SiO$_x$ films as-grown (a) and after further annealing (b).
In this case, the PL spectra are analyzed in terms of a quantum confinement model [18,19]:

\[
\lambda(\text{nm}) = \frac{1.24 \, \mu\text{m}}{E_N(\text{eV})} = \frac{1.24 \, \mu\text{m}}{[1.12 + (3.73/d^{1.39})]}
\]

It corresponds to the radiative recombination of electron–hole pairs in the Si-nc, where \(d\) and \(E_N\) are the diameter and energy of the Si-nc, respectively, and \(\lambda(\text{nm})\) is the wavelength of the Si-nc emission. Table 3 shows the theoretical values of average sizes of Si-nc calculated from the main peak of the PL spectra, where the size of the Si-nc reduces and the energy band gap increases with decreasing the growth temperature. Table 3 also shows the values of average sizes of Si-nc obtained by HRTEM [7]. So the blue shift of PL with decreasing Si concentration and growth temperature, i.e., decreasing Si-nc size, is in good agreement with the QCE. Besides, in these SiOₓ films, the absorption and emission are correlated with quantum effects in Si-nc because the theoretical values of \(E_N\) are similar to the approximate values of the optical band gap (\(E_g\)) that were obtained by the relationship known as Tauc plot (see Figure 3).

However, PL spectra are very wide, which indicates that two possible mechanisms are involved. Then, the second mechanism of light emission that was considered in the SiOₓ films is related to some kinds of defects produced during the growth process, as shown in the FTIR spectra, such as, weak oxygen bonds (WOB), neutral oxygen vacancy (NOV), non-bridging oxygen hole center (NBOHC), positively charged oxygen vacancy (E’ center), interstitial oxygen molecules, and peroxy radicals [19-24]. Some of these defects, such as NOV and NBOHC are the principal radiative recombination centers or the luminescence centers. So, the different peaks that were defined by the deconvolution of the PL spectra are related to different kinds of defects, as listed in Table 4.

Figure 7a shows that the PL spectra of the (SiOₓ/SiOᵧ) junctions, as-grown, emit in a wide spectral range. This is due to the junction of two films with different properties and stoichiometries, so these spectra have a behavior mixed with respect to the SiOₓ films (see Figure 6a), where again the first film deposited dominates the behavior of the junction. For this case, the PL spectra are not analyzed in terms of a quantum confinement model, because the PL spectra have not a maximum emission.

**Table 3 Theoretical and experimental values**

| Tg (°C) | Gap \(E_N\) of Si-nc (eV) | Diameter of Si-nc (nm) | Diameter of Si-nc (nm) by HRTEM [7] |
|--------|--------------------------|------------------------|--------------------------------------|
| 1,150  | 1.67                     | 3.96                   | 5.5                                  |
| 1,020  | 1.89                     | 3.11                   | 4                                    |
| 900    | 2.28                     | 2.31                   | 2.5                                  |

Values of the \(E_N\) and the diameter of Si-nc calculated from the PL spectra of the SiOₓ films as-grown as a function of the Tg.
peak that suffers a blue shift. So only the second mecha-
nism of light emission was considered in the (SiO_x/
SiO_y) junctions, which is related to some kinds of defects
produced during the growth process, as shown in the
FTIR spectra and listed in Table 5.

Figures 6b and 7b show the PL spectra of the SiO_x
films and (SiO_x/SiO_y) junctions with thermal anneal-
ing. It is observed that when the thermal an-
nealing is applied to the samples, a restructuring occurs
(as shown in the FTIR spectra) because the temperature
causes a phase separation and growth of Si-nc’s. For the
case of the SiO_x films, the defect-related PL is almost
eliminated and the near-infrared PL intensity (band B) is
a little enhanced after annealing, i.e., the PL is domi-
nated by the Si-nc’s embedded in the SiO_x matrix. For
the case of the (SiO_x/SiO_y) junctions, what is most inter-
esting is that they emit not only in the orange-near in-
frared range (band B) but also in the violet-blue range
(band A), and the green-yellow band disappears. In this
case, the PL intensity of band A slightly declines with re-
spect to the PL intensity of the (SiO_x/SiO_y) junctions as-
grown. So the dominant radiative defects have changed
from E’δ or NBOHC defects to Si-O species (i.e., WOB
and NOV defects). With respect to the PL intensity of
band B, this is greatly enhanced after thermal annealing.
It is reasonable to consider that for the (SiO_x/SiO_y)
junctions, the two mechanisms are not contradictory but run
parallel in the samples. In summary, two PL bands, band
A and band B, have been observed after thermal anneal-
ing. According to the PL spectra, the former band A is
ascribed to Si-O-related species [19,20,24], and band B is
ascribed to the Si-nc’s embedded in the SiO_x matrix
[9]. Annealing seems to advance the formation of crys-
talline silicon (c-Si) as well as the formation of (defect)
structures responsible for PL emission. The difference in
behavior of the PL intensity before and after annealing
between the SiO_x films and junctions is interesting; this
difference could be due to the fact that the single layers
have a combination of defects and formed Si-nc’s which
generates intense PL. When annealing SiO_x films, the
defects are desorbed and generated, leading to the de-
crease in PL intensity. In double layers, there are several
factors, one is the double annealing procedure and the
other is the order of layers, which have a very strong im-
 pact on optical properties, where the defects of the first
layer are preserved due to the growth of the second lay-
er. These layers have a higher concentration of defects
and excess silicon; when annealing (SiO_x/SiO_y) junc-
tions, some defects are desorbed or modified, mainly on
the second layer, where the PL is improved in the red.
According to the analysis of the compositional and
optical properties of the SiO_x films and (SiO_x/SiO_y)
junctions, we can correlate the evolution of the stoichi-
ometry (obtained by FTIR and XEDS) with the reduction
of the Eg (obtained by transmittance), where the oxygen
content decreased along with the increase in growth
temperature and in turn with the reduction of the Eg.
So, we may assume that the Si excess increases as the
growth temperature increases.

**Conclusions**

In the present work, SiO_x films and (SiO_x/SiO_y) junc-
tions were obtained by HFCVD technique with different
growth temperatures; their compositional and optical
properties were studied as-grown and after further an-
nealing. For the case of the SiO_x films, as-grown, the ab-
sorption and emission are correlated with quantum
effects in silicon nanoparticles and also associated with
defects. For the case of the (SiO_x/SiO_y) junctions, as-
grown, only the second mechanism of light emission

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**Table 4 Defect types [19-24] linked with the peak position**

| Defect type | Peak position (nm) |
|-------------|-------------------|
|             | As grown          |
|             | 1,150°C  | 1,020°C  | 900°C    |
| WOB         | 400      | 410      |          |
| NOV defect  | -        | 436      |          |
| E’δ defect  | -        | 540      | 543      |
| NBOHC       | -        | -        | 658      |
| None identified | 739 | 758      | -        |

Position obtained by deconvolution from PL spectra of the SiO_x films as function of the T_g.

**Table 5 Defect types linked [19-24] with the peak position**

| Defect type | Peak position (nm) |
|-------------|-------------------|
|             | As grown          |
|             | 1,150/1,020°C  | 1,150/900°C | 1,020/1,150°C | 1,020/900°C | 900/1,150°C | 900/1,020°C |
| WOB         | -                  | -                  | 408 | 406 | 414 |
| NOV defect  | 449 | 438 | 432 | - | - | - |
| E’δ defect  | 532 | 534 | - | 559 | 542 | 514 |
| NBOHC       | 648 | 622 | 667 | 674 | 672 | 644 |
| None identified | 805 | 782 | 795 | 789 | 770 | 780 |

Peak position obtained by deconvolution from PL spectra of the (SiO_x/SiO_y) junctions as function of the T_g.
was considered, which is related to some kinds of defects produced during the growth process. After thermal annealing, the PL spectra of the SiO\textsubscript{x} films only show band B (740 nm), and the PL spectra of the (SiO\textsubscript{x}/SiO\textsubscript{y}) junctions show bands A (375 to 450 nm) and B (740 nm), where the PL band A is ascribed to Si-O-related species, and the PL band B to the quantum size effect of the Si-nc’s embedded in the SiO\textsubscript{x} matrix. Vibration bands related to Si-H was observed in SiO\textsubscript{x} films and (SiO\textsubscript{x}/SiO\textsubscript{y}) junctions before annealing (as shown in the FTIR spectra), which disappeared after thermal annealing. The behavior of the change in intensity and shift of the FTIR and PL spectra is ascribed to changes of phase in this material, associated with several defects and Si-np’s. The (SiO\textsubscript{x}/SiO\textsubscript{y}) junctions showed an interesting behavior due to the double annealing procedure and ‘order’ of layers, which have a very strong impact on optical and compositional properties. This opens the possibility of tuning the spectra range for a specific application.

Competing interests

The authors declare that they have no competing interests.

Authors’ contributions

DEV conducted the SiO\textsubscript{x} growth. DEW and JALL carried out the FTIR and PL measurements and drafted the manuscript. JCL coordinated the FTIR study. GGS coordinated and participated in the growth of the SiO\textsubscript{x} films. ABL conducted the thermal annealing. NDET conducted the UV measurements. JALL provided the idea and supervised the study. All authors read and approved the final manuscript.

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DEV is currently a PhD student in the Science Institute - Center of Investigation in Semiconductors Devices (IC-CIDS) from Autonomous University of Puebla, Mexico. She started to work on the growth and characterization of non-stoichiometric silicon oxide obtained by HFCVD. Her research interests include experiments and structural, optical and electrical characterization of SiO\textsubscript{x} films and MOS structures. JALL is currently a researcher and professor in the Science Institute - Center of Investigation in Semiconductors Devices (IC-CIDS) from Autonomous University of Puebla, Mexico. He started to work on the growth and characterization of non-stoichiometric silicon oxide. His current research interests include metallic oxides obtained by the HFCVD technique, GaN obtained by the metal organic CVD technique and porous silicon gas sensor devices.

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