

Oxidative Degradation of Parylene C (Poly (monochloro-*para*-xylylene)) Thin Films on Bulk Micromachined Piezoresistive Silicon Pressure Sensors

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High-temperature exposure experiments were performed to determine the upper temperature limitations for parylene-C-coated pressure sensors in air. Significant sensitivity shift ($> 2\%$) and visual cracking of the parylene surface were observed on devices with $\geq 3 \mu\text{m}$ of parylene C when heated above 125°C in air following as little as 1 hour of exposure. The mechanism for this phenomenon has been identified as oxidative degradation of the polymer and is documented in the literature. This mechanism is not noticed with uncoated devices, nor is it noticed with parylene-coated devices in nitrogen or vacuum ambients, nor with parylene-coated devices if oxygen can be isolated from the parylene (for example, by coating the parylene with a secondary encapsulant). As reported in the literature, our observations confirm that this effect is a function of parylene thickness. No cracking and very little sensitivity shift ($< 2.0\%$) were observed for any devices coated with $\leq 16 \mu\text{m}$ of parylene C at 105°C or less. Therefore, 105°C is recommended as the maximum temperature for pressure sensors coated with conventional Gorham process parylene C in air.

1. Introduction

Silicon bulk micromachined piezoresistive pressure sensors are currently used in dry air applications. Figure 1 shows a cross-sectional view of a silicon pressure sensor.⁽¹⁾ A thin diaphragm (approximately $20\text{--}25 \mu\text{m}$) is formed in the silicon by backside anisotropic

etching in KOH. A difference in pressure across the diaphragm causes a deflection which in turn causes a change in strain throughout the diaphragm. The piezoresistors are transducers that react to a change in strain by changing their resistance. Sensitivity, thus, is directly proportional to the stress applied to the sensor chip by pressure. Maximum pressure-induced stresses are located at the top edge of the square diaphragm; therefore, a diffused silicon piezoresistor is placed at that location to maximize sensitivity. Any material added to the single-crystal silicon diaphragm changes the sensitivity, offset and the temperature characteristics of the sensor depending on the modulus, thickness and CTE of that material. Definitions for sensitivity, offset and temperature characteristics for these pressure sensor devices are shown in Figure 2.

While several applications for pressure sensors exist in dry air environments, considerably more are available if the sensor is compatible with pressurized media. Providing media compatibility generally refers to providing long-term reliability for the environment in which the active sensor will be used. For example, the washing machine market requires the sensor to be compatible with alkaline solutions (pH = 7 to 12) at elevated temperatures (0–100°C) for the life of the product.⁽²⁾ Corrosion of the first-level interconnection in aqueous environments is expected to be the primary failure mode for these devices, although etching of the silicon in alkaline environments and swelling of plastic films in organic solvent environments are also potential failure modes. Automotive, industrial and appliance markets currently rely on more costly, bulkier and (potentially) less reliable conventional mechanical pressure sensors, so silicon piezoresistive pressure sensors could provide cost savings if media compatibility could be achieved.

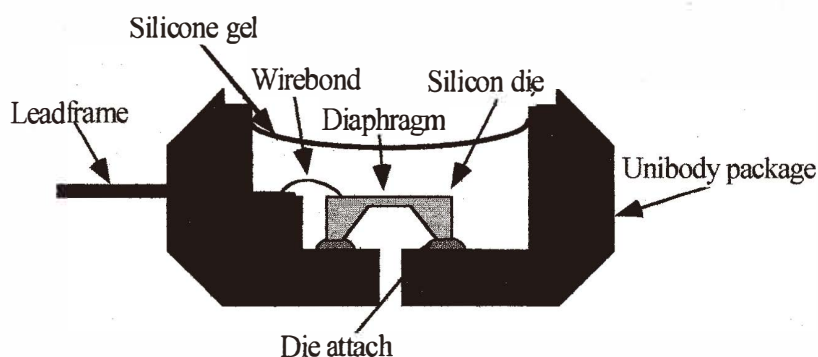
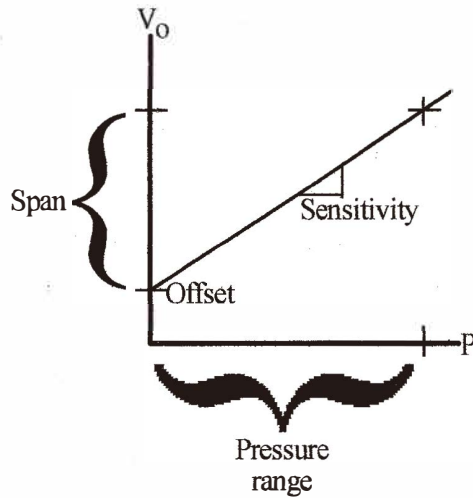
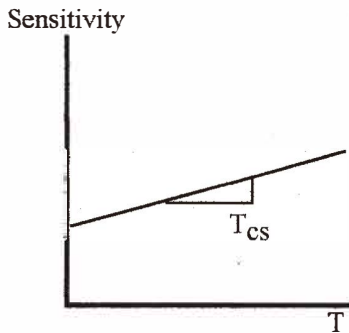


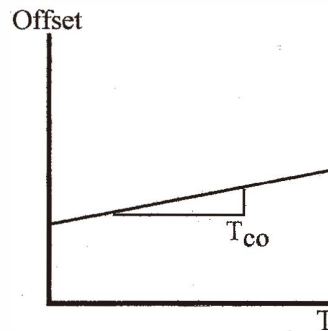
Fig. 1. Cross-section of the Motorola "unibody" pressure-sensor package that was used throughout these experiments. The package contains a bulk micromachined silicon die with a piezoresistive transducer that is die-bonded to the unibody package (housing material) using a (fluoro-)silicone elastomeric die attach material. Gold wires are used to connect aluminum bondpads on the die with the Au-plated, Ni-barrier layer, Cu leadframe. A (fluoro-)silicone gel is used on the topside of the die to protect the die surface, wirebonds and leadframe from moisture in our current production package. Parylene coatings are being evaluated to replace this (fluoro-)silicone gel for applications where media compatibility is necessary.



(a)



(b)



(c)

Fig. 2. Definitions for the electrical parameters of interest for semiconductor pressure sensors. (a) The plot of output voltage as a function of pressure is very linear for these devices. The slope of this line is the sensitivity of the device at a given input bias, and the intercept is the offset. (b) The plot of sensitivity (or span) as a function of temperature is approximately linear for these devices. The slope of this curve is the temperature coefficient of sensitivity (or span), T_{cs} . (c) The plot of the offset as a function of temperature is also approximately linear for these devices. The slope of this curve is the temperature coefficient of offset, T_{co} .

Two general methods for protecting silicon pressure sensors currently exist. First, a solution can be obtained by changing the silicon die configuration to provide a media-compatible die that fits into almost any package.⁽³⁻¹⁷⁾ However, this approach is generally a long-term product development proposition. Alternatively, protective films that are resistant to pressurized media can be applied to the existing package.⁽¹⁸⁻²⁸⁾ While this is a shorter-term solution, at least four difficulties exist with this method: 1) assembled packages often contain low-temperature (< 200°C) polymeric materials, so high-temperature processes and associated materials are not possible with the existing package; 2) the materials must be processed in such a way that they coat the silicon die, bondpads, wirebonds and leadframe within the package cavity; 3) the materials must have either a negligible effect on device sensitivity or (more probably) a minimal, repeatable effect on device sensitivity; and 4) the encapsulation must be manufacturable.

However, most practical attempts at media compatibility for pressure sensors, including those described here, have applied the idea of an additional media-compatible encapsulant. Even though other encapsulants are prevalent in the IC industry, currently, only silicone gels and parylenes have been used successfully with pressure sensor packages.^(1,13,18,28) Therefore, our initial work with barrier coatings studied the possible combinations of these two types of materials. Because parylene does not adhere well to silicone gels, the combination of parylene on silicone gel produces a sensor with non-repeatable temperature characteristics, so this case has not been considered.⁽¹⁸⁾ Several companies, including Motorola, are pursuing the (fluoro-)silicone gel option. However, fluorosilicone gels provide insufficient media compatibility to aqueous solutions when devices are biased. Corrosion often results; therefore, parylene coatings have been pursued for these environments. Work on parylene-coated pressure sensors has been performed by Motorola, Delco, Honeywell, Siemens, Sensym and Bosch. Solutions with a silicone gel on parylene have been suggested by Fujikura, Motorola, and Delco.

During our initial parylene evaluation, a significant change in the sensitivity was noted following 100 to 1000 hours of high-temperature storage at 125°C and 150°C on a parylene-coated Motorola XPX 5050D pressure sensor device.⁽¹⁾ Table 1 lists these results. Notably, the parylene-coated devices exhibited a change in sensitivity that was dependent upon thickness, exposure time and exposure temperature in air. Uncoated devices did not exhibit this effect. The results from the -40 to 125°C temperature cycling were similar to the results from the 125°C high-temperature storage test. Visually, cracking damage to the parylene occurred following these experiments. Therefore, a study was necessary to determine the maximum storage temperature in air for parylene-coated pressure sensors designed for 1000-hour reliability.

Similar results have been reported in the literature. Parylene has been observed to suffer from oxidative degradation at temperatures above 125°C.⁽²⁹⁻³¹⁾ Specifically, chain scission (i.e., C-H bond breakage) has been reported in oxygen containing atmospheres at temperatures above 125°C (Fig. 3). Wu *et al.* observed the normalized thickness as a function of exposure time and temperature for parylene N and parylene F films.⁽³⁰⁾ For the parylene N (i.e., poly(*para*-xylylene)) films in air, which will react similarly to parylene C in air, the thickness change became noticeable at 150°C. Severe cracking of the films was observed at temperatures as low as 250°C. In nitrogen or vacuum, thickness change was

Table 1
Evaluation of parylene coated XPX 5050D devices in high temperature storage exposure tests.

	Exposure time (hrs.)	Estimated sensitivity shift (% change from initial)	
		Storage at 125°C	Storage at 150°C
No parylene	0	0	0
	100	—	0
	500	0	+0.5
	750	—	+0.5
	1000	0	—
Nominally 1.5 μm	0	0	0
	100	—	-0.5 \pm 0.25
	500	0	-1.5 \pm 0.5
	750	—	-1.5 \pm 0.5
	1000	-0.5 \pm 0.25	—
Nominally 5 μm	0	0	0
	100	—	-6 \pm 2
	500	-3 \pm 1	-9 \pm 9
	750	—	-11 \pm 11
	1000	-4 \pm 2	—

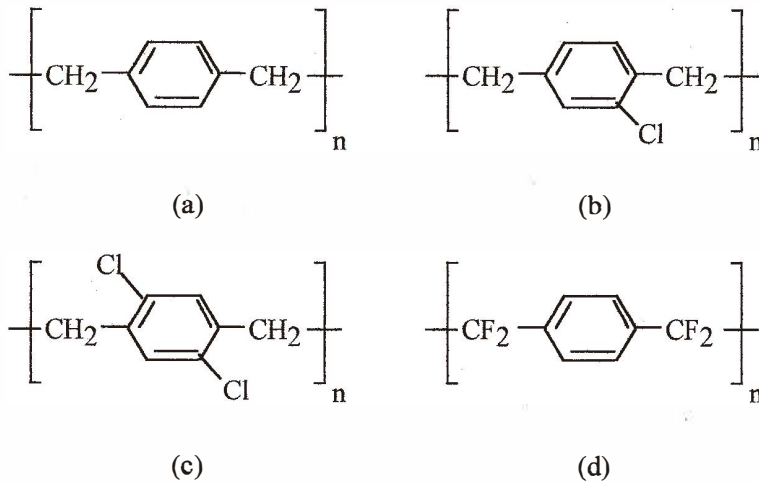


Fig. 3. The chemical structure for the monomers of the parylene materials. (a) Poly (*para*-xylylene) or parylene N. (b) Poly (monochloro-*para*-xylylene) or parylene C. (c) Poly (dichloro-*para*-xylylene) or parylene D. (d) Fluorinated poly (*para*-xylylene), parylene F, or parylene AF-4.

not observed until 300°C.

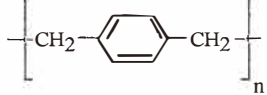
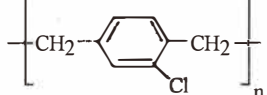
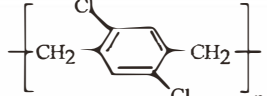
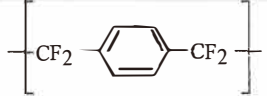
Beach⁽³²⁾ has reported the elongation at break, secant modulus and tensile strength as a function of exposure at elevated temperature in air. For parylene C, these results suggest that exposure at 150°C in air is similar to that at 265°C in vacuum in terms of the change in these mechanical properties. Furthermore, it appears that increasing the chlorination increases the temperature stability. This result has also been reported by Mahoney⁽³³⁾ and Joesten.⁽³⁴⁾

Joesten⁽³⁴⁾ observed the weight loss as a function of temperature for four different parylene materials via thermogravimetric analysis (TGA) in both air and N₂. From these results (Table 2), it is apparent that the four different parylene materials have relatively similar thermal stability in nitrogen but widely different temperature resistances in air. Dabral *et al.*⁽³⁵⁾ reported similar TGA results for parylene AF-4 in N₂. They also performed differential scanning calorimetry (DSC) and observed an exothermic peak at approximately 480°C that corresponds to the dramatic increase in weight loss that was observed with TGA. Loss of weight during TGA analysis in air is the result of breaking C-H bonds (99 kcal/mole⁽³⁵⁾ or 80.9 kcal/mole in a diatomic molecule⁽³⁶⁾). These bonds break at much lower energy than C-F bonds (116 kcal/mole⁽³⁵⁾ or 132 kcal/mole in a diatomic molecule⁽³⁶⁾) (Table 2).

It should be noted that the oxidative degradation effect is different from the annealing effect observed in parylene. Annealing is the result of a conformational change in the polymer chains from the as-deposited metastable state to a thermodynamically preferred state during the initial heating above the glass transition temperature.⁽¹⁸⁾ Oxidative degradation involves chain scission during high-temperature exposure in oxygen atmospheres.

The goal of the current work was to determine the temperature limitation for parylene-

Table 2
Initiation of rapid weight loss in TGA for the parylenes.

Tradename	Structure	Air	Nitrogen
Parylene N		260	490
Parylene C		260	490
Parylene D		320	460
Parylene AF-4		500	510

coated pressure sensors. This limitation was determined by observing the change in sensitivity during exposure and the appearance of any cracking of the parylene films, which will degrade the harsh media protection afforded by the films.

2. Materials and Methods

Two experiments involving pressure sensor packages coated with different parylene thicknesses were performed. Table 3 shows the two experimental matrices for the parylene-coated devices.

In the first experiment, 24 Motorola XPX2010D pressure-sensor devices⁽¹⁾ were exposed to high-temperature oxygen storage following parylene deposition and annealing: eight were exposed at 85°C, eight were exposed at 105°C, and eight were exposed at 125°C. Half of each of these groups contained 4–5 μm parylene-C-coated devices, and half had 7.5–9.5 μm parylene-C-coated devices. Sensitivity was measured at room temperature after each 24-hour interval for 192 hours for sensors biased to 10 Vdc.⁽¹⁾ It was also measured following 504 and 1008 hours at room temperature. Visual observation of the devices was also performed at these intervals.

In the second experiment, 20 Motorola XPX12D pressure-sensor devices⁽¹⁾ were exposed to high-temperature oxygen storage following parylene deposition and annealing; 10 were exposed at 85°C and 10 were exposed at 105°C. Half of each of these groups were 2.5–3.5 μm parylene-C-coated devices, and half were 10–16 μm parylene-C-coated devices. Sensitivity was measured after each 24-hour interval for 192 hours at room temperature for sensors biased to 3 Vdc.⁽¹⁾ It was also measured following 504 and 1008 hours at room temperature. Visual observation of the devices was also performed at these intervals. No bias was applied during high-temperature storage for either of these experiments, although it was applied during device electrical characterization at each of the prescribed measurement intervals.

Table 3

Experimental matrix for high temperature exposure of parylene-C-coated devices in air. Figures in the table indicate the number of devices.

Parylene thickness (μm)	Exposure temperature ($^{\circ}\text{C}$)		
	85	105	125
2.5–5	4*	4*	4*
	5†	5†	
7.5–9.5	4*	4*	4*
	5†	5†	–

* First experiment.

† Second experiment.

3. Results and Discussion

Figure 4 shows the results of the first experiment comparing 4–5 μm parylene-C-coated and 7.5–9.5 μm parylene-C-coated XPX 2010D devices. Each set of data represents the average of four devices. The same devices without parylene C coating did not exhibit any significant sensitivity shift at any of the high temperature exposure conditions investigated. Very little change was observed for either thickness at 85°C or 105°C (circles and squares in Fig. 4). At 125°C, a significant change in sensitivity (i.e., 2–3%) was observed following the first 24-hour exposure interval in both the thin and thick parylene-coated sensors. The sensitivity continued to vary as a function of time when the parylene-coated devices were exposed at 125°C in air for 1008 hours. Furthermore, cracking of the parylene-coated devices was observed when they were exposed at 125°C but was not observed for devices exposed at 85°C or 105°C (Fig. 5). The observation of cracking is indicative of oxidative degradation.⁽³⁰⁾

It is possible that some continued annealing occurred during the first 24 hours of exposure at 125°C. However, the continual sensitivity shift following 24 hours through 1008 hours suggests that oxidative degradation was occurring. From modeling work used to determine the effect of parylene on the device sensitivity, the decrease in sensitivity occurs because of an increase in the effective parylene modulus/thickness product.⁽³⁷⁾ For example, the sensitivity shift caused by the addition of parylene is a function of the parylene modulus (E_p), the silicon modulus (E_{Si}), the parylene thickness (t_p), and the silicon thickness (t_{Si}):

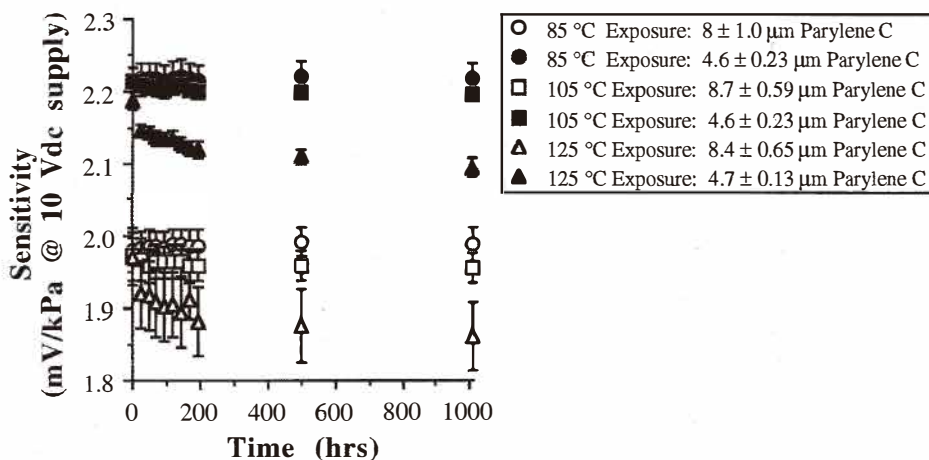


Fig. 4. A plot of the sensitivity as a function of time for parylene-C-coated XPX2010D pressure-sensor devices during high-temperature exposure. Sensitivity is measured with a 10 Vdc supply voltage at room temperature after specific time intervals. Thin (4–5 μm) and thick (7.5–9.5 μm) parylene-coated pressure sensors were tested at 85°C, 105°C and 125°C in air. Four devices per parylene thickness per temperature were used.

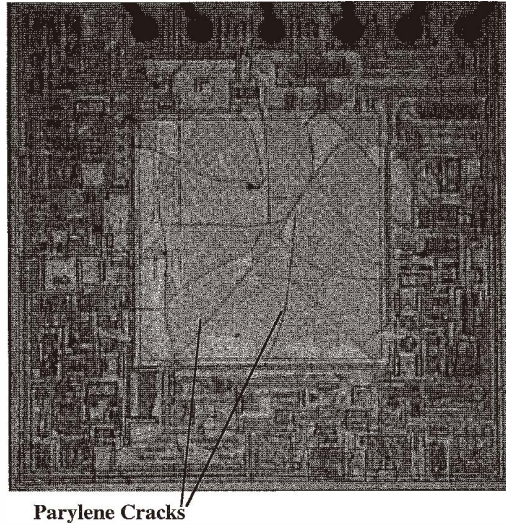


Fig. 5. Cracking at the parylene surface following high-temperature exposure in air. For parylene C, cracking on the surface of the parylene is observed at 125°C in as little as 1 hour. This is an example of a parylene-C-coated surface following 1008 hours of exposure at 125°C.

$$\frac{S_{\text{post}} - S_{\text{pre}}}{S_{\text{pre}}} \propto \frac{2 \left(\frac{E_p}{E_{\text{Si}}} \right) \left(\frac{t_p}{t_{\text{Si}}} \right) + \left(\frac{E_p}{E_{\text{Si}}} \right)^2 \left(\frac{t_p}{t_{\text{Si}}} \right)^2}{1 + 2 \left(\frac{E_p}{E_{\text{Si}}} \right) \left(\frac{t_p}{t_{\text{Si}}} \right)}, \quad (1)$$

where S_{post} is the sensitivity following deposition and S_{pre} is the sensitivity before deposition. This result assumes that the change in sensitivity is proportional to the length of the diaphragm divided by the effective thickness of the diaphragm, t_{eff} :

$$t_{\text{eff}} = t_{\text{Si}} + \left(\frac{E_p}{E_{\text{Si}}} \right) t_p. \quad (2)$$

A similar analysis can be performed to determine the approximate sensitivity shift that results from a high-temperature storage process of a parylene-coated pressure sensor:

$$\frac{S_{\text{post}} - S_{\text{pre}}}{S_{\text{pre}}} \propto \frac{2(E_{\text{pre}} t_{\text{pre}} - E_{\text{post}} t_{\text{post}})}{E_{\text{Si}} t_{\text{Si}}}, \quad (3)$$

where the subscript 'pre' represents the time before the high-temperature process and the subscript 'post' represents the time after the high-temperature process. Therefore, the sensitivity shift observed is a function of the parylene thickness and modulus changes that result from oxidative degradation.

These cracks in the parylene do not appear to propagate through the entire parylene thickness onto the silicon; therefore, the surface of the diaphragm remains covered with parylene. However, the cracks do alter the stress profile within the film. Because the pressure-sensor output is affected by the packaging stresses, this phenomenon causes a permanent change in electrical parameters, specifically sensitivity.⁽³⁷⁾ As the crack grows, the effective parylene thickness and/or modulus change results in the continuous decrease in the sensitivity of the devices (per eq. 3).

Figure 6 shows the results of the second experiment comparing 2.5–3.5 μm parylene-C-coated and 10–16 μm parylene-C-coated XPX 12D devices. Again, the same devices without parylene C coating did not exhibit any shift in sensitivity at any of the high-temperature exposure conditions investigated. Very little change was observed for the thin (2.5–3.5 μm) parts at 85°C or 105°C. A slight ($-2.0 \pm 0.19\%$) change in the sensitivity of the thick (10–16 μm) parylene-coated devices was observed during the first 24-hour interval for the thick parts at 105°C. Because this occurred only during the first interval, it is postulated that this is the result of insufficient annealing of these thick films. None of the sensitivity measurements on any of the devices shifted following 24 hours through 1008 hours of exposure at 85°C or 105°C in air. Therefore, for parylene-coated pressure sensors with a thickness of less than 16 μm , little change in sensitivity (i.e., $-1.0 \pm 0.19\%$

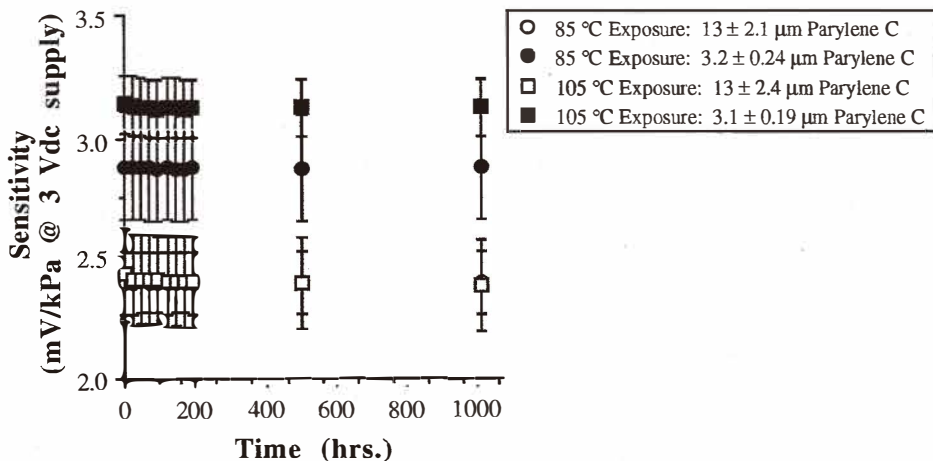


Fig. 6. A plot of sensitivity as a function of time for parylene-C-coated XPX 12D pressure-sensor devices during high-temperature exposure. Sensitivity is measured using a 3 Vdc supply voltage at room temperature after specific time intervals. Thin (2.5–3.5 μm) and thick (10–16 μm) parylene-coated pressure sensors were tested at 85°C and 105°C in air. Five devices per parylene thickness per temperature were used.

maximum) as a function of time was observed when exposing the samples at 105°C in air. It is assumed, because of the results from our initial evaluations and the results from the first experiment in this study, that exposure of these devices at 125°C would cause a sensitivity shift and visual cracking.

4. Conclusions

Table 4 shows the resulting sensitivity measurements from the three high-temperature exposure experiments on parylene-coated pressure sensors. From these results, it appears that 105°C is the maximum temperature for exposure of parylene-C-coated pressure sensors in an oxidizing atmosphere (i.e., exposure at 125°C causes enough oxidative degradation to be observed as a long-term sensitivity shift) to satisfy existing specifications. The oxidative degradation that occurs at higher temperatures causes cracking of the parylene films. The cracks are initiated at the surface of the films and propagate toward the underlying substrate. This changes the effective thickness and/or modulus of the film. Because sensitivity of the pressure sensor is a function of the parylene thickness and modulus, oxidative degradation of the films results in a permanent change in the sensitivity of the sensor. Furthermore, cracks in the parylene films provide easier access for harsh media to the underlying substrates, particularly electrolytic solutions, to biased underlying metal surfaces. Therefore, exposure of parylene films in oxidating atmospheres above 105°C will result in shorter reliability lifetimes and permanent electrical parametric shifts.

Re-evaluation of the high-temperature limit is necessary if parylene is to be used without being exposed to an oxygen atmosphere; for example, if the parylene-coated

Table 4

Summary of the change in sensitivity results (%) for high temperature exposure (>750 hrs.) of parylene-coated pressure sensors.

Parylene thickness (μm)	Exposure Temperature ($^{\circ}\text{C}$)			
	85	105	125	150
< 1.5			$-0.5 \pm 0.25^*$	$-0.5 \pm 0.5^*$
3 - 5	0.32 ± 0.021^1	-0.744 ± 0.0038^1	$-4 \pm 2^*$	$-11 \pm 11^*$
	0.0 ± 0.22^2	-0.6 ± 0.12^2	-4.21 ± 0.01^1	
7 - 10	0.49 ± 0.021^1	-0.84 ± 0.024^1	-5.54 ± 0.044^1	-
10 - 16	0.0 ± 0.13^2	-2.0 ± 0.19^2 total or -1.0 ± 0.19^2 (without annealing)	-	-

* DGAG073 Motorola internal parylene reliability evaluation (16 devices per temperature).

¹ First experiment described above (4 devices per temperature).

² Second experiment described above (5 devices per temperature).

sensors are used in nitrogen or vacuum. Furthermore, if additional encapsulation is placed over the parylene (e.g., (fluoro-) silicone gels), the high-temperature limit must be re-evaluated.

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