

# PERFORMANCE CHARACTERISTICS OF A THIN-FILM ALUMINUM OXIDE HUMIDITY SENSOR \*

Saburo Hasegawa  
National Bureau of Standards  
Washington, D.C. 20234

## Summary

A laboratory study was made of the performance of a miniature-size, thin-film aluminum oxide humidity sensor over a range of ambient temperatures from 0°C to 40°C and encompassing humidities from 40 to 78,000 parts per million by volume (ppm-v). Hysteresis and temperature effects of the sensors were obtained from tests performed at ambient temperatures 40°C, 25°C and 0°C and encompassing relative humidities from 19 to 98 percent. Information on short-term repeatability, sensitivity and storage effects was obtained from tests performed at an ambient temperature of 23°C and encompassing humidities from 40 to 6,000 ppm-v.

## Introduction

There have been numerous investigations<sup>1-6</sup> substantiating the deleterious effects of internal moisture on semiconductor devices. In view of the increasing number of incidents of integrated-circuit failures due to high internal water vapour content, rejection criteria are being implemented in MIL.STD.883 as Method 1018 on "Internal Water Vapour Content". Method 1018 specifies the use of a small humidity sensor within the package or the measurement of the humidity of the emitted gas on puncturing the package, with the latter measurement done primarily by mass spectrometry.

Although present efforts within the industry are leading to increased intralaboratory precision and interlaboratory correlation, there is still a great need for the development of test packages with known humidity which would be suitable for use as a transfer device for interlaboratory measurements. The development of a suitable transfer standard requires the use of a well-characterized humidity sensor, mass spectrometer and test package. This paper describes the study of a commercially available humidity sensor for possible use in the study of the behavior of water vapour inside experimental test packages.

## Description of Sensor

The aluminum oxide sensor is a variable impedance device.<sup>7-8</sup> It consists essentially of an oxidized silicon-wafer base or substrate on top of which is a thin layer of aluminum with a porous surface of aluminum oxide. The latter is overlaid with a thin film of gold. The gold and aluminum serve as electrodes. Water vapour diffuses through the porous gold film to the oxide surface. On sorbing water vapour the oxide changes its impedance.

The sensor was attached to a TO-5 header using epoxy and air cured at room temperature. 0.0025cm (one-mil) gold wires were attached to the electrodes using an ultrasonic bonder.

Two groups of sensors were treated. The sensors designated by Series "A" were purchased in early 1979 and the "B" Series sensors were obtained from Rome Air Development Center in 1978.

The capacitance of the sensors was measured with an impedance bridge with an accuracy of 0.1 percent. A frequency of 770 Hz was used for the tests and the peak-to-peak voltage was less than 1 volt.

\*This effort was supported by the Semiconductor Technology Program of the NBS with funding provided by the Naval Material Command through P.O. N0037781P89010.

## Test Procedure

The National Bureau of Standards two-pressure humidity generator<sup>9</sup> was used for these tests. Briefly, the humidity generator produces atmospheres of known humidity by saturating a gas stream at an elevated pressure and constant temperature and then expanding this gas into a test chamber which is in a separate temperature-controlled bath. The pressure in the test chamber is nominally at atmospheric pressure. The humidity of the gas in the test chamber is calculated from the measured values of the temperature and pressure in the saturator and in the test chamber. The uncertainty of the humidity of the gas produced by the generator does not exceed 0.3 percent of the value (volume ratio, ppm) over most of the range covered in this paper. The carrier gas used for these tests was air.

Seventeen sensors were tested simultaneously, nine sensors of the series "A" and eight of the series "B". To obtain general information on the operational characteristics such as shape of response curve, sensitivity, hysteresis and temperature coefficient, the first series of test was performed at ambient temperatures 25°C, 40°C and 0°C. The second series of runs was made at an ambient temperature of 23°C and at various humidities below 6000 ppm-v to obtain more detailed information on such factors as the sensor stability, storage effects, hysteresis and sensitivity. Between the first and second series of tests the sensors were baked in vacuum at a nominal absolute pressure of 0.7 Pa (5 millitorr) and a temperature of 100°C for a period of eight hours.

Each sensor was electrically connected to the measuring circuit through a selector switch and a shielded cable. Capacitance calibrations were made of the measuring circuit and used to adjust the output readings.

## Results for First Series of Tests

A family of calibration curves (isotherms) is shown in Fig. 1 for sensor A5, which had a sensitivity close to the average of the sensors "A" Series and similarly in Fig. 2 for sensor B3. The output in picofarads has been plotted as a function of the logarithm of the volume ratio (parts per million by volume) for ambient temperature 40, 25 and 0°C.

The temperature coefficients for two sensors from each lot are given in Table 1. The two selected sensors are sensors which have the highest and lowest sensitivities for their respective lot. The coefficients express the percentage change in the volume ratio per degree Celsius change in ambient temperature for fixed capacitance. They can be used to estimate the error in the indicated volume ratio due to an uncertainty in, or a failure to account for, the ambient temperature. The coefficients are given for the temperature intervals 25 to 40°C and 25 to 0°C.

Table 1

Temperature Coefficient

Sensor	Volume ratio span	Mean temperature coefficient for temp. range	
		40 to 25°C	25 to 0°C
( % of volume ratio per degree C )			
A1	57k to 10.2k	3.8	
A1	30k to 1.8k		5.4
A3	61k to 9.4k	4.3	
A3	29k to 1.9k		5.2
B4	40k to 6.4k	2.9	
B4	28k to 1.0k		5.4
B2	44k to 7.1k	3.7	
B2	27k to 1.5k		5.1
	average	3.7	5.3

Typical hysteresis loops at ambient temperatures 25 and 0°C are shown in fig. 3. Starting the calibration from the low humidity point of the isotherm, the volume ratio was increased in steps to the highest value and then reduced in steps to the initial point. At 40°C, the loop was not completed and therefore no hysteresis data were available. A summary of the average calibration cycle hysteresis for each sensor is given in Table 2. There is a significant difference in hysteresis between the "A" and "B" lot sensors with the "B" sensors exhibiting a larger hysteresis at both the 25 and 0°C calibrations.

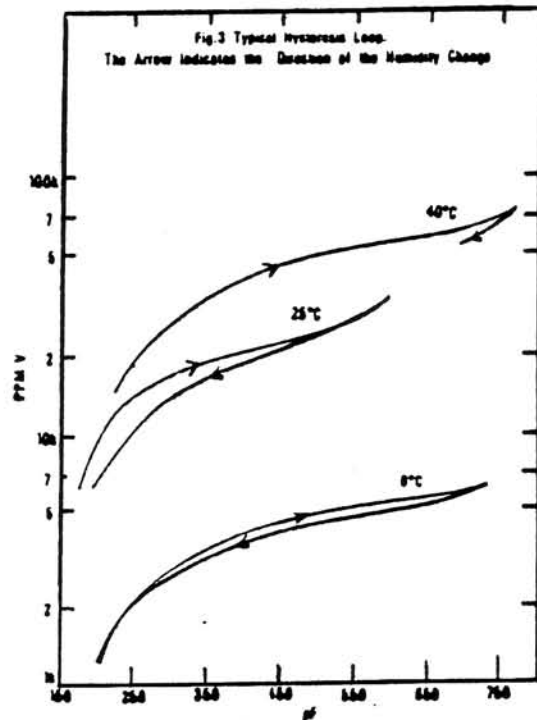
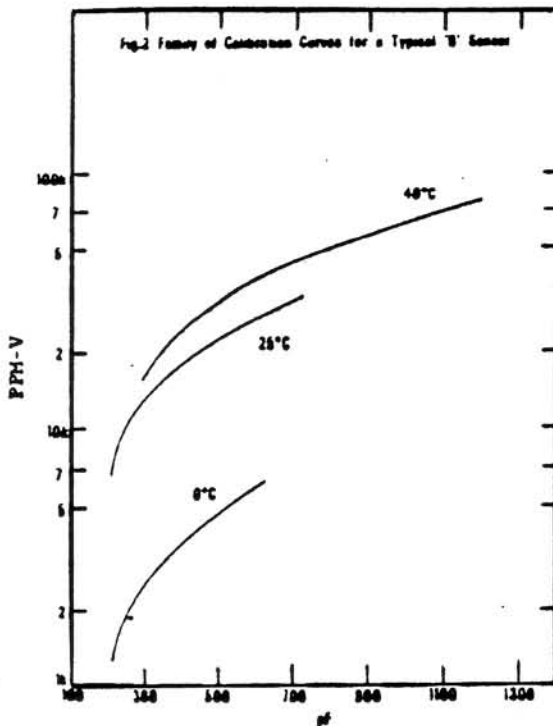
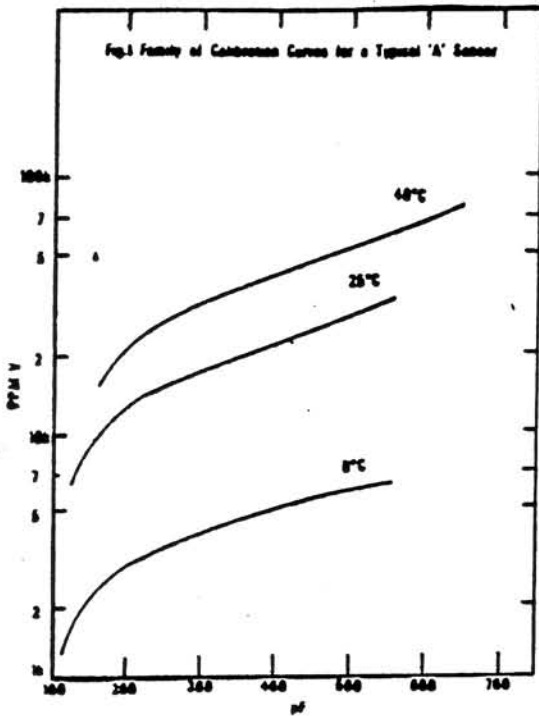


Table 2

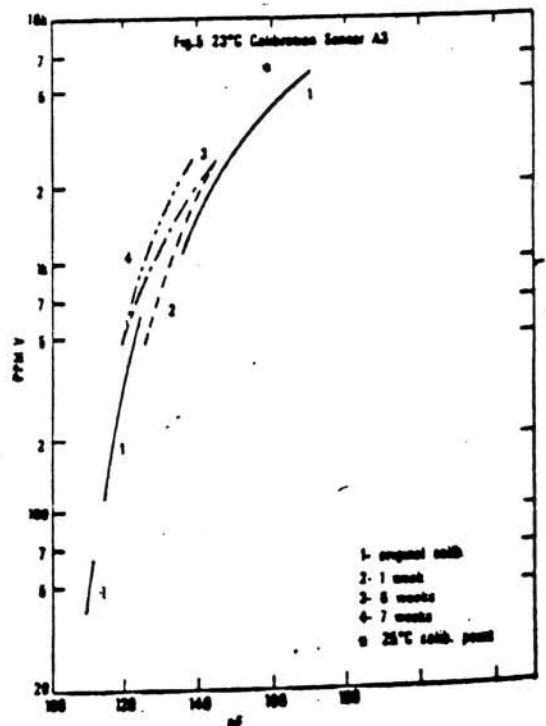
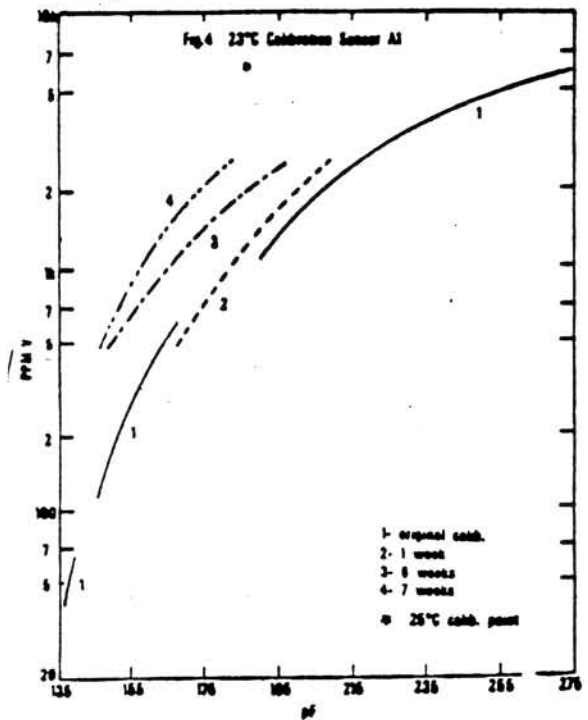
Average Hysteresis for First Test Series

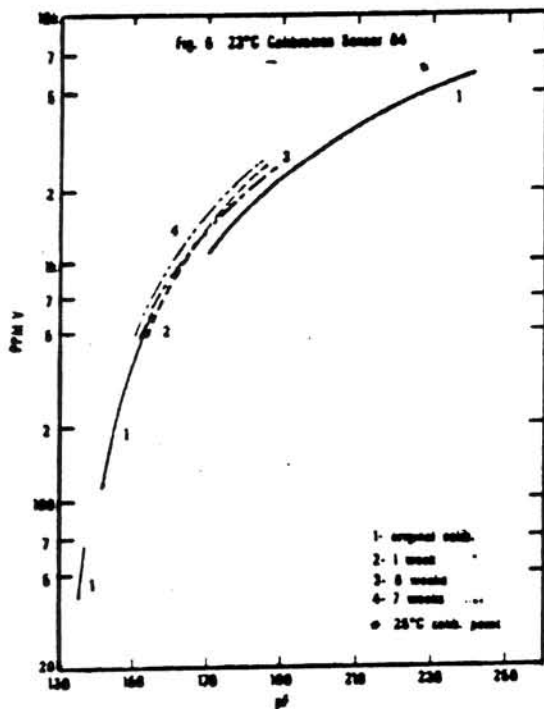
Ambient Temperature (°C)	Humidity Range (ppm-v)	Sensor Number									
		A1	A2	A3	A4	A5	A6	A7	A8	A9	Mean
25	8000 to 30000	Average Hysteresis, ppm-v									
		1600	1700	1100	600	800	700	600	800		990
0	1500 to 6000	220	240	530	400	380		410	250	400	350
		Sensor Number									
25	8000 to 30000	Average Hysteresis, ppm-v									
		B1	B2	B3	B4	B5	B7	B8			2900
0	1500 to 6000	2600	1800	3200	3700	3400	2800	3000			550

Results for Second Series of Tests

One of the factors which affect the sensitivity of a sensor is the number of available sites on which the water molecule can sorb on the surface. Since maximum sensitivity of the sensor was required for the investigation of test packages, the following procedure was used in an attempt to clean the sensor surface before beginning the second test series. The sensors were put in a vacuum chamber (approximate absolute pressure of 0.7 Pa) and baked at a temperature of 100°C for a period of 8 hours. After this bake-out procedure, the sensors were stored in air at moderate vacuum (absolute pressure of approximately 19kPa) for 3 weeks and then for 2 weeks at room humidity (not exceeding 50% RH) before starting the second series of tests.

The 23°C calibration curves for the sensors with the highest and lowest sensitivities from each of their respective lots are shown in Figs. 4-7. The "star" in Figs.4-7 indicate the 25°C calibration point of the sensor at 6000 ppm-v prior to the first bake-out. The curves labelled "1" (solid line), covering a humidity range of 40 to 6000 ppm-v, represents the initial calibration in this series of tests. Three consecutive days of testing were required to cover this range. The sensors were then stored at room humidities for one week before performing the first recalibration tests. The results for this recalibration are represented by curve "2" in Figs. 4-7.





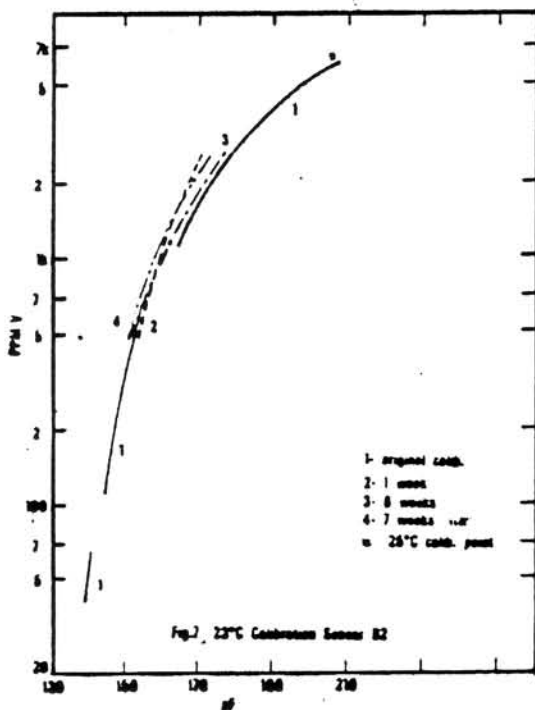
After the first calibration, the sensors were again heated to 100°C under vacuum for 8 hours to determine whether the drifting of the calibration curve was due to trapped water molecules on the sensor surface. After this rebaking, the sensors were stored in dried nitrogen atmosphere for a week before performing the second recalibration. The results for this second recalibration are represented by curve "3" in Figs. 4-7.

After the second recalibration, the sensors were stored for a week in a dried nitrogen atmosphere before performing the third recalibration. The results of the third recalibration are represented by curve "4" in Figs. 4-7. The numbers of weeks of elapsed time since the original calibration for each of the recalibration runs are also listed in Figs. 4-7.

Table 3

23°C Calibration Shifts

Sensor no.	Duration of Storage		
	1 week (average calibration shifts, percent of the indicated humidity)	6 weeks	7 weeks
A1	23	56	76
A3	29	32	45
B4	19	15	22
B2	26	9	20



In general, the shift in the calibration was to give a lower capacitance reading for the same humidity which has the effect of indicating a lower humidity for the same capacitance reading. The average shifts in the calibration for the four sensors illustrated in Figs. 4-7 are given in Table 3. The average shifts are given in units of percentage change of the indicated volume ratio over the humidity span of 500 to 2500 ppm-v and were obtained by comparing the recalibration results with the original calibration. After storage for 1 week, there appears to be no significant difference in behavior between the two lots of sensors and the repeatability (more precisely, the lack of repeatability) is approximately 25 percent. However, after rebaking the sensors, the sensors in the "A" lot appear to have a significantly larger shift than those in the "B" lot. It should be emphasized that the values listed in Table 3 should not be used to compute the rate of shift of the sensor with duration of storage because the sensors were rebaked prior to the time when the reading listed under 6 weeks were taken. A comparison of the values listed under 1 week and the difference between the values listed under the 7 weeks and 6 weeks columns would indicate the effect of storage for a week under room humidity conditions as compared to a week storage in dried nitrogen atmosphere. The bake-out procedure did not produce a recovery of the original calibration characteristics of the sensor. The sensor shows a continual drift towards lower capacitance reading for a given humidity. Storage of the sensors in a dried nitrogen atmosphere did not stop this drifting of the calibration curve.

Table 4  
Sensitivity, 23°C Calibration

Sensor	Calib. Curve	Humidity, ppm-v				
		2500	2000	1000	600	500
		Sensitivity, ppm-v / pF				
A1	Original	64	57	36	28	28
	1st recal.	59	62	47	33	29
	2nd recal.	46	50	40	29	25
	3rd recal.	52	67	56	41	36
A3	Original	145	129	81	68	66
	1st recal.	124	131	99	71	62
	2nd recal.	94	101	78	56	49
	3rd recal.	109	132	109	79	70
B2	Original	128	115	73	82	74
	1st recal.	126	144	115	83	73
	2nd recal.	96	101	77	55	48
	3rd recal.	90	111	93	68	59
B4	Original	73	66	42	48	48
	1st recal.	40	70	65	48	43
	2nd recal.	43	61	54	40	35
	3rd recal.	42	69	63	46	41

Table 4 is a compilation of the sensitivities of the four sensors illustrated in Figs. 4-7 and given in units of ppm-v / pF. The sensitivities were obtained by differentiating the calibration equations and solving the differentiated equations at capacitances corresponding to the indicated humidities. The calibration data for each sensor by means of a least squares regression to a second order polynomial. In Table 4, a smaller number indicates a more sensitive sensor. In general, the sensitivity of the sensor is increased after undergoing the bake-out procedure.

Due to the fact that these sensitivities are based on limited numbers of calibration points subject to various influences and errors, no great significance can be placed on individual values.

Fig. 8 shows the hysteresis loops for sensor A1 and it illustrates the type of hysteresis the sensor exhibits when the sensor is exposed to a maximum humidity of 6000 ppm-v at 23°C. A summary of the average hysteresis for each sensor is given in Table 5. The average hysteresis of the "B" sensors is approximately 50 percent smaller than the lot "A" sensors. Prior to subjecting the sensors to the bake-out procedure the sensors in the "B" lot had significantly larger hysteresis than the "A" lot sensors.

Table 5  
Average Hysteresis for Original Calibration Second Test Series

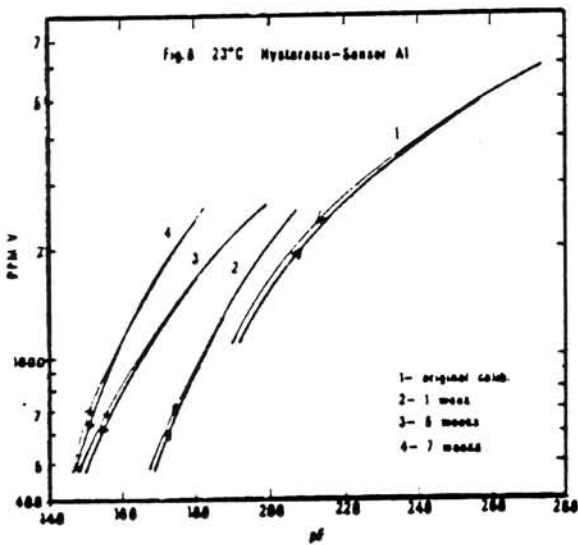
Humidity Range (ppm-v)	Sensor Number									Mean
	A1	A2	A3	A4	A5	A6	A7	A8	A9	
1200 to 6000	60	130	150	160	110	100	130	180	130	130
Average Hysteresis, ppm-v										
	20	20	64	60	70	82	84	70		59
1200 to 6000	Sensor Number									
	B1	B2	B3	B4	B5	B6	B7	B8		
	Average Hysteresis, ppm-v									
	20	20	64	60	70	82	84	70		59
1200 to 6000										

#### Conclusions and Discussions

The sensitivity of individual sensors varied by a factor of at least two. The variability of the sensors require individual calibration for each sensor. The sensors are also sensitive to ambient temperature and at temperatures near 25°C, the temperature coefficient is approximately 4 to 5 percent of the indicated volume ratio (ppm-v) per degree Celsius.

The sensor decreased in sensitivity with time and the procedure of baking the sensor at 100°C and under vacuum increased the sensor's sensitivity. Storing the sensor in dry nitrogen atmosphere did not stop the shift in calibration with time. The bake-out procedure reduced the hysteresis of the "B" lot sensor.

The response time characteristic was not investigated; however, it was found that the sensor took a much longer time to reach equilibrium when subjected to high humidity (40 and 25°C calibration) as compared to the conditions during the second test series. For the first test series performed at high water vapour content, the sensor exhibited a normal hysteresis curve, i.e. for a fixed humidity, the capacitance reading was larger for the descending curve than for the ascending curve. However, for the second test series, the hysteresis characteristic was reversed and the capacitance reading was larger for the ascending curve. Combined with the observations of faster response time at the low water vapour content, overall drifting of the sensor to lower capacitance and a shift in the calibration curve after an elapsed time of 1 day, it is surmised that this reversed hysteresis curve is a result of the sensor undergoing a shift in calibration after an elapsed time of less than an hour. After storage in a dry nitrogen atmosphere, the sensors still continued to drift towards lower capacitance.



However, the hysteresis curve returned to normal indicating a possible reduction in the rate of the calibration shift with storage in dry nitrogen atmosphere. Unfortunately, control sensors were not set aside during the bake-out procedure to test whether the procedure used to increase the sensor sensitivity was detrimental or beneficial for the over-all performance characteristic of the sensor.

The results of the test indicate that, although the sensitivity and hysteresis characteristics may be adequate, the drift in the calibration curve is too large for these sensors to be useful to study the behavior of water inside experimental test packages over extended time periods.

#### Reference

1. D.S. Peck and C.H. Zierdt, Jr., "Temperature - Humidity Acceleration of Metal-Electrolysis Failure in Semiconductor Devices", 11th Annual Proceedings Reliability Physics Symposium, April 1973.
2. H. Koelmans, "Metallization Corrosion in Silicon Devices by Moisture-Induced Electrolysis", 12th Annual Proceedings Reliability Physics Symposium, 1974.
3. A. Shumka and R.R. Piety, "Migrated-Gold Resistive Shorts in Microcircuits" 13th Annual Proceedings Reliability Physics Symposium, April 1975.
4. N. Sbar, "Bias-Humidity Performance of Encapsulated and Unencapsulated Ti-Pd-Au Thin Film conductors in an Environment Contaminated with Cl<sub>2</sub>", Proceedings 26th Electronic Component Conference, 1976.
5. A. Der Marderosian and C.R. Murphy, "Humidity Threshold Variations for Dendrite Growth on Hybrid Substrates", 15th Annual Proceedings Reliability Physics Symposium, April 1977.
6. D. Stroehle, "On the Penetration of Gases and Water Vapour into Packages with Cavities and on Maximum Allowable Leak Rates", 15th Annual Proceedings Reliability Physics Symposium, April 1977.
7. M.G. Kovac, "A New Moisture Sensor for 'In-Situ' Monitoring of Sealed Packages", 15th Annual Proceedings Reliability Physics Symposium, 1977.
8. M.G. Kovac, "Performance Characteristics of Al<sub>2</sub>O<sub>3</sub> Moisture Sensor Inside Sealed Hybrid Packages, Proceedings of the 1977 International Microelectronics Symposium, 1977.
9. S. Hasegawa and J.W. Little, "The NBS Two-Pressure Humidity Generator, Mark 2", J. Res. Nat. Bur. Stand. (U.S.), 81A, No. 1, 81-88 (Jan.-Feb. 1977)