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A NEW ELECTRICAL HUMIDITY SENSOR

by

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Physical Chemistry Laboratory

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SUMMARY

The preparation of a new humidity sensor is described, and results of a wide range of performance tests on the new device are presented. The sensor responds to changes in atmospheric water content by changes in electrical resistance and is effective throughout most of the relative humidity (RH) range. It is stable under a variety of conditions including immersion in water, humidity cycling, heating to 100°C, and passage of AC current. Several methods for using the new sensor in humidity measurement and control applications are illustrated.

KEY WORDS

humidity, sensor, polyelectrolyte, control, measurement

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A NEW ELECTRICAL HUMIDITY SENSOR

L. V. Interrante and K. W. Browall

INTRODUCTION

A substantial increase in the need for accurate humidity measurement and control devices has occurred during the last decade.(1) Areas of science and technology which have traditionally used humidity sensors, such as meteorology, the processing and storage of foods, textiles, and chemicals, etc., now require increasing numbers of such devices with a greater degree of precision and accuracy in their operation. New awareness of the importance of humidity control for human health and comfort has resulted in the increased use of humidifier and dehumidifier devices. The aerospace and computer technologies have likewise created new demands for humidity sensors.

A wide range of humidity sensors have been developed in recent years in response to these needs. Several excellent review articles provide a detailed description of these devices.(1) In spite of these developments, however, the need for an accurate, reliable, and yet inexpensive sensor for general-purpose humidity measurement and control has not been adequately met.

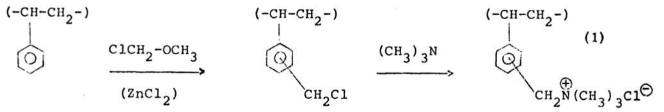
The purpose of this report is to describe a new humidity sensor which has been developed in the Physical Chemistry Laboratory at CR&D. The new sensor is an electrical impedance device, in which changes in relative humidity (RH) are reflected in changes of the sensor's electrical impedance. The device to be described offers distinct advantages over other "electrical humidity sensors" in both cost and performance characteristics.

CONSTRUCTION OF THE SENSOR

The basic components of the new sensor are a humidity sensitive, ionically conductive polymer and an interdigitated electrode support substrate.

The Humidity-Sensitive Polymer

The polymer is an organic polyelectrolyte of controlled composition, prepared by J. F. Klebe of the Chemical Laboratory at CR&D. Its preparation involves the chloromethylation and quaternization of polystyrene, as in Eq. (1):



By controlling the chloromethylation reaction,(2) a sufficient number of ionic groups can be introduced to yield a polymer which has relatively low electrical resistivity and high water permeability, yet is essentially insoluble in water. The electrical resistivity of this material is highly dependent upon the ambient relative humidity. This dependence presumably arises from the tendency of the ionic sites in the polymer to absorb water from the atmosphere, resulting in an increased mobility of the Cl^- charge carriers. Work with polymers of varying chloride ion content has shown that optimum humidity-sensing characteristics are obtained when the polyelectrolyte contains one chloride ion for every three to four monomer units. At this degree of substitution, the material is readily soluble in a 1:1 methanol:chloroform mixture, yet still insoluble in water. This allows convenient application of the polymer to the substrate by spraying, brushing, or dipping; yet, after the coating is dry, repeated immersion in water has no effect upon its electrical or physical properties. The coating also resists abrasion and dust buildup, and does not flake or loosen upon immersion in water or exposure to heat. Several other polyelectrolytes, including sulfonated PPO®, P_3O, and polystyrene derivatives were evaluated for the humidity sensor application, but none exhibited the superior physical properties and performance characteristics of the chloromethylated polymer.

The Electrode Substrate

A pair of interlocking electrodes provides electrical contact and support for the polymer. This electrode substrate is constructed from 1/16-inch copper-clad Textolite, a material with an electrically insulating glass-epoxy base which is commonly used in making printed circuit boards. The interdigitated electrode pattern* shown in Fig. 1 is etched into the copper using standard photoresist methods. The resulting electrode structure is then electroplated with a flash coat (approximately 1000Å) of gold or silver to protect the copper surface from oxidation. Finally, the surface is coated with the polymer using a 2% to 3% solution in 1:1 methanol:chloroform. The optimum quantity of polymer per 1- by 1 1/4-inch element was found to be 3 to 4 mg. Because of the low cost of materials required and the adaptability to large-scale production, this method provides an

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* For the present study, a mask containing 56 such electrode patterns was prepared by means of step and repeat photography and was used in exposing the photoresist.

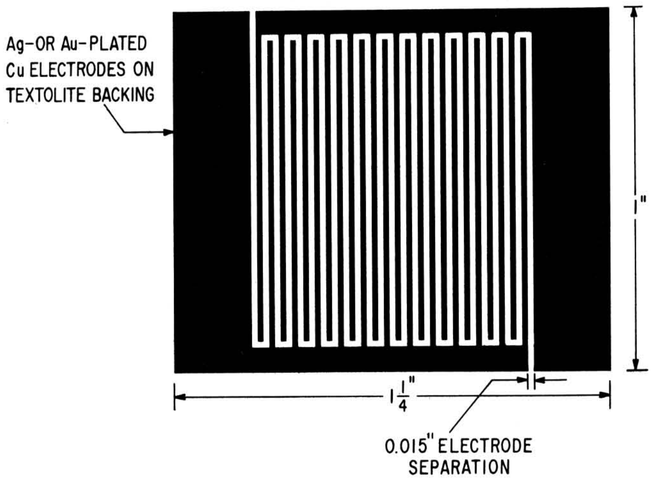

Fig. 1 Interdigitated electrode humidity sensor with 0.015-inch electrode separation.

extremely economical route to the preparation of humidity sensors.*

The spacing between the interlocking electrodes can easily be varied to meet specific requirements of resistance range and RH vs resistance characteristics. In the present study, units with 0.015-, 0.008-, and 0.004-inch electrode separation with approximately the same overall electrode area were used. The 0.004-inch separation is close to the lower limit attainable by the photoetching method.

PERFORMANCE FEATURES OF THE SENSOR

Relative Humidity vs Resistance Characteristics

Experimental Procedure

The variation of electrical resistance with relative humidity was determined using a continuous flow, controlled humidity air stream and an AC conductance bridge. The controlled humidity system(3) employs two constant flow streams of air, one thoroughly dried over H_2SO_4 and drierite, and the other saturated with water vapor at a constant temperature slightly below room temperature by passage through two consecutive gas saturating bottles. The streams are equilibrated at room temperature and then mixed in continuously controlled proportions using bleed-off valves to regulate the flows and two flowmeters to determine the relative amounts being mixed. The flow rate of the mixed flow is also monitored to provide a constant flow into a test chamber containing a thermocouple and holders for making electrical contact to seven humidity sensors.

For experiments at temperatures above or below room temperature, some modifications in the above procedure were necessary. In this case, a single stream of air was saturated with water vapor by passage through two consecutive gas saturating bottles placed in a thermostated bath at temperature, T_1, and then warmed to the test temperature, T_2. This stream was then passed through a glass test chamber, containing a thermocouple and a humidity sensor with electrical connections; the test chamber was immersed in a thermostated bath at temperature T_2.

In both cases the electrical resistance was measured using a Wayne-Kerr Model B224 Universal AC bridge.

Results

Figure 2 shows the equilibrium response and associated hysteresis loop for a sensor with 0.015-inch electrode separation at room temperature (23°C).

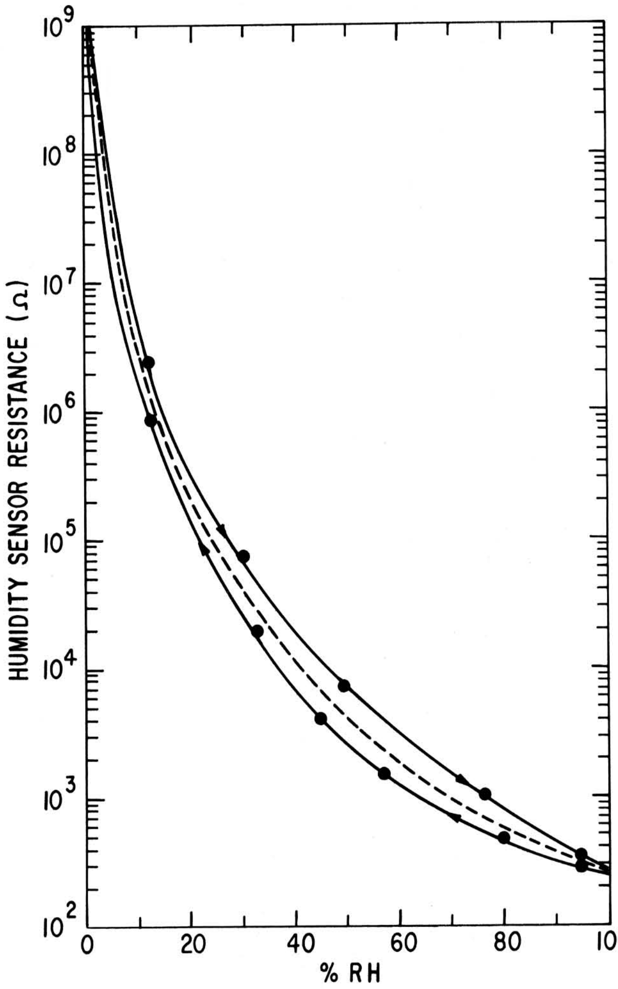

Fig. 2 Typical resistance vs %RH curve for a sensor with 0.015-inch electrode separation.

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* The estimated cost for sensors prepared for this work in groups of 56 units is 25¢ per unit. Costs, including materials and labor, should be well under 10¢ per unit for larger scale production.

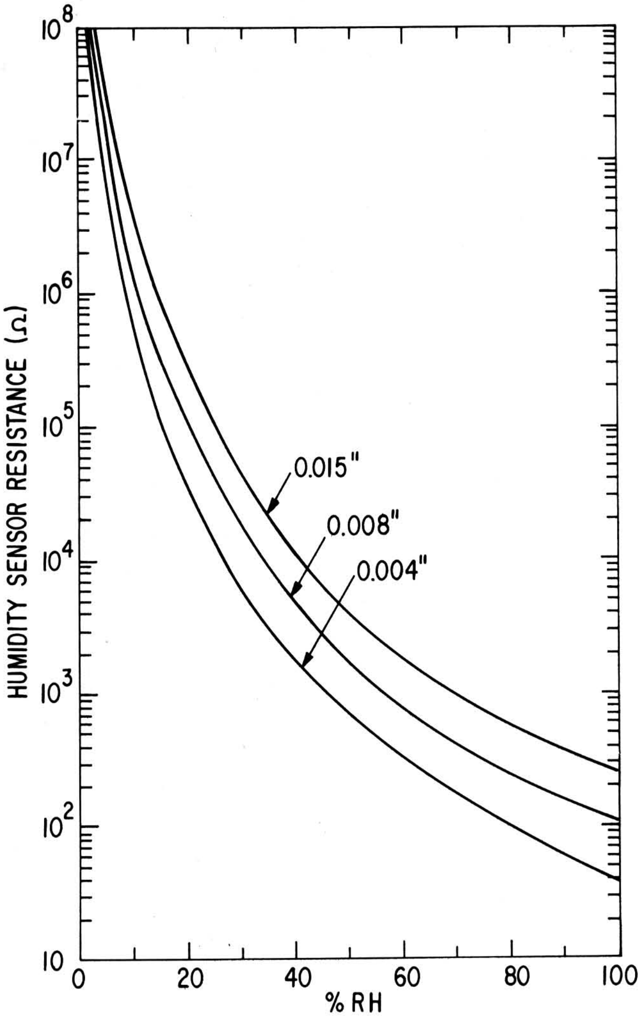

Fig. 3 Resistance vs %RH for sensors with different electrode separations.

When the humidity is varied from 0 to 100%, a change in resistance of approximately six orders of magnitude takes place. The equilibrium response curve (dotted line) in Fig. 2 was derived from the two experimentally determined curves for high-to-low and low-to-high humidities, respectively. The hysteresis is most significant in the middle humidity range, where deviations of up to 6% RH occur. This hystersis effect is observed for all types of humidity sensors and results from the kinetics of the absorption and diffusion of water in the sensor material. As such, its magnitude is highly dependent upon the experimental conditions employed. The large variation in relative humidity and short time (~15 minutes) between the resistance measurements employed herein undoubtedly lead to a hysteresis considerably larger than would be observed in most applications. Therefore, except for those applications which require very high accuracy at rapidly varying humidities, this effect may be entirely neglected. In the following discussion and diagrams the equilibrium response curves, as defined here, will be used.

The effect of changing the electrode separation in the sensor element is shown in Fig. 3. Decreased electrode separation causes a corresponding decrease in the resistance readings throughout the humidity range. The electrical resistance at high humidity is almost an order of magnitude lower for the 0.004-inch spaced electrodes than for the 0.015-inch separation.

The thickness of the polymer coating also affects the resistance vs %RH characteristics. The sensors used for the present study each contained 3 to 4 mg of polymer, but otherwise no attempt was made to control or determine the actual thickness of the coating. As a result, small differences in characteristics were observed among sensors of a particular type. In a continuous production facility, where both the polymer thickness and the electrode dimensions could be readily controlled, excellent reproducibility between sensors is anticipated, obviating the need for individual calibration.

The effect of temperature upon sensor calibration is shown in Fig. 4. Measurements at 15°, 23°, and

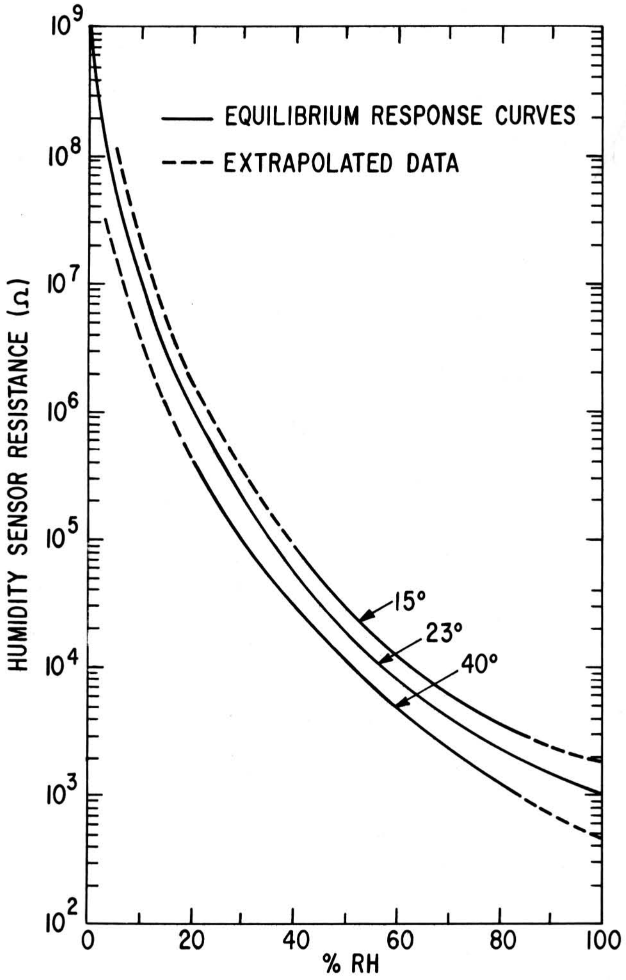

Fig. 4 Resistance vs %RH at 15°, 23°, and 40°C for a sensor with 0.015-inch electrode separation.

40°C for a sensor with 0.015-inch electrode separation show a general decrease in resistance with increasing temperature. Calibration at 15° and 40° was carried out in the variable temperature apparatus described above, while the 23° calibration was carried out using the standard mixed flow system. The temperature effect increases the %RH at a given resistance by about 0.5% RH per degree centigrade increase in temperature over most of the humidity range.

Response Time Measurements

Experimental Procedure

The time required for the sensor to respond to changes in relative humidity was determined using a specially designed flow system. This system employed two air streams at 33% and 75% RH each equilibrated by means of three consecutive gas saturating bottles. For the 33% RH stream the first saturator was filled with 50% H_2SO_4 and the others with saturated $MgCl_2$ solution; for the 75% RH stream, 30% H_2SO_4 in combination with saturated NaCl solutions were used. These air streams were led into a test chamber through a two-way stopcock. The chamber was designed to accomodate the sensor and a holder with minimal excess volume. The atmosphere in the chamber was alternated between 33% and 75% RH by turning the stopcock. The resistance of the sensor was determined as a function of time using the Wayne-Kerr AC bridge. This was accomplished by setting the bridge for a balance at a predetermined set point and measuring the time required to reach this balance point after the stopcock was turned.

Results

The results of response time measurements for a sensor with a 0.015-inch electrode separation are shown in Fig. 5. The abrupt change in %RH causes an exponential change in electrical resistance with time; the sensor reaches 63% of full response in seconds. Response is faster for increasing than decreasing RH, as is typical for absorptive type humidity sensors.

Stability

The stability of the sensor's resistance vs relative humidity characteristics toward a number of physical and environmental effects was examined. Stability upon storage, heating, contact with water, passage of current, and long-term usage are, of course, of utmost importance for the satisfactory employment of the sensor in measurement and control applications.

Long-Term Storage

Sensors were calibrated and then stored for extended periods under room humidity conditions, and in sealed containers under both high and low humidity conditions. The sensors were recalibrated periodically. The electrical properties of the sensors were not affected by long-term storage under any of these conditions.

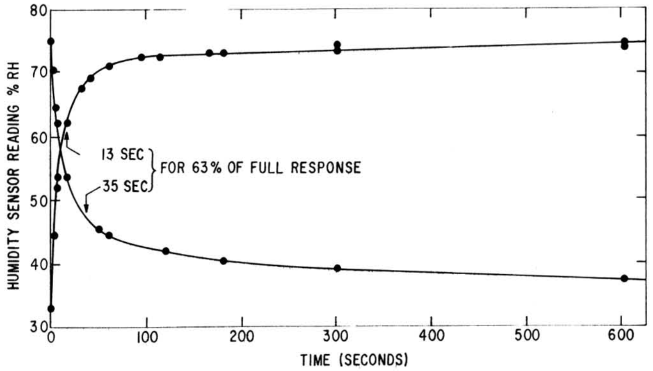

Fig. 5 Response time for a sensor with 0.015-inch electrode separation.

Contact with Water and Other Substances

Contact with water, either from atmospheric condensation, or by direct immersion, has no effect upon the sensor's electrical characteristics or physical appearance. Contact with organic solvents in which the polyelectrolyte is soluble, such as alcohol, chloroform, or acetone would, of course, cause immediate damage. Exposure to acids and ionic salt solutions should similarly be avoided. Detailed studies of the effect of various gases upon the sensor characteristics have not been carried out; however, preliminary tests using high concentrations (10%) of corrosive gases such as SO_2 and NH_3 in air indicate that significant changes in resistance can occur under these conditions. However, the much lower concentrations of the various impurities found in normal air environments are not expected to significantly change the sensor impedance even with prolonged exposure.

Humidity Cycling

Using apparatus similar to that described above for response time measurements, a sensor was cycled repeatedly between humidity extremes of nearly 0 and 100% RH, while the resistance was monitored by an AC bridge. Figure 6 shows the first four of seven complete cycles in a five-hour period. After each cycle the sensor returned to the same resistance value at approximately 100% RH, indicating high stability toward rapid and repeated cycling of the humidity. A sensor which had not been plated with gold or silver (i. e., the polymer was applied directly to the Textolite-backed copper electrodes) exhibited a significant increase in the resistance after each cycle. In this case, a green deposit on the electrodes indicated the formation of $Cu(OH)_2$.

Heat

The thermal stability of the sensor was determined by storing an initially calibrated sensor in an oven for 15 to 24 hours, followed by recalibration.

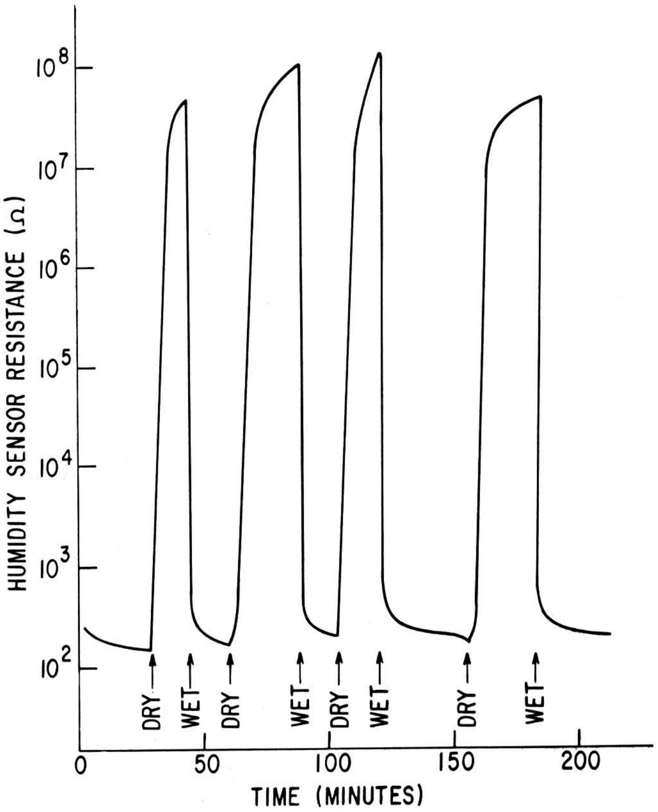

Fig. 6 Humidity cycling test.

The oven temperature was increased by approximately 10°C after each recalibration. As is shown in Fig. 7, the sensor undergoes no significant change in electrical characteristics upon storage at temperatures up to 100°C. Humidity-sensitive properties are retained after exposure to temperatures above 100°C, but a gradual permanent increase in electrical resistance occurs. There are no changes in the physical appearance of the sensor or polymer coating even after exposure to 122°C.

AC Current Tests

AC current from a 60-cycle variable voltage source was used to determine the maximum power output and maximum current level which sensors with both 0.015- and 0.004-inch electrode separations could sustain without damage. The current was determined by measuring the voltage drop across a decade resistor in series with the sensor by means of a null-type AC voltmeter. The previously described mixed flow system was used to maintain constant 50% and 90% RH for the tests. In each case, ohmic behavior is observed up to an applied voltage of 1.5V to 15V, depending on the relative humidity and the electrode separation of the sensor. At higher voltage, significant deviations from ohmic behavior are observed; this is shown in Fig. 8 for a sensor with 0.004-inch electrode separation at 50% RH.

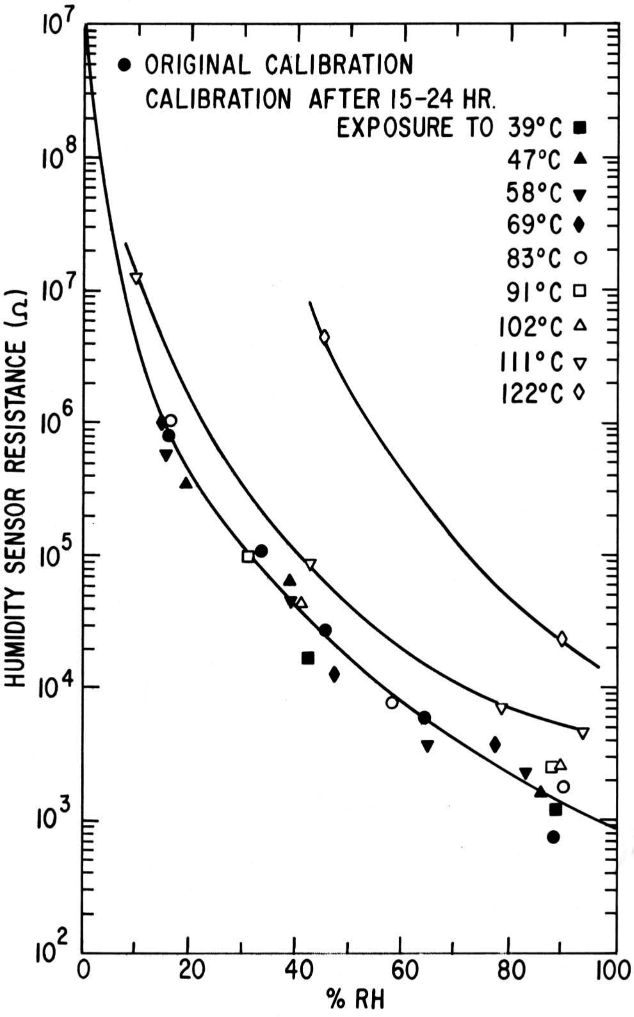

Fig. 7 Stability of the sensor to heat.

An empirically determined "limiting wattage" of 0.015W may be used to locate the approximate voltage at which deviations from Ohm's law occur, from Eq. (2),

$$V = \sqrt{.015R}, \qquad (2)$$

where R is the resistance of the sensor obtained from the resistance vs %RH calibration curve. This limiting wattage was found to be independent of both electrode separation and RH. Localized heating occurs at the surface of the sensor when it is operated at greater than 0.015W; indeed, the limiting wattage may be in part determined by such surface heating, since the polyelectrolyte would tend to become dehydrated and raise the sensor resistance. Appropriate heat-sinking of the sensor may increase this limiting wattage.

The sensor may be safely operated at wattage levels considerably higher than the above limiting

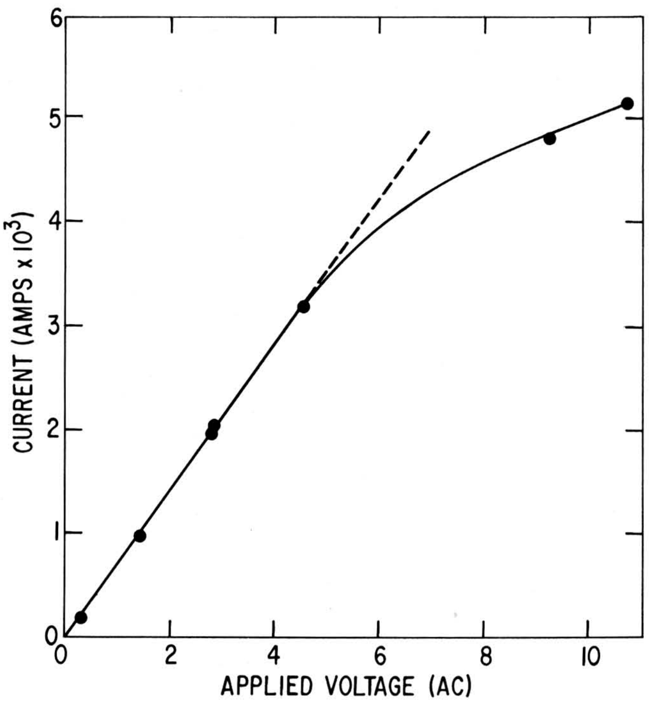

Fig. 8 Current-voltage relationship at 50%RH for a sensor with 0.004-inch electrode separation.

value. For example, a sensor with 0.004-inch electrode separation has been operated at 12 ma at 4V applied for extended periods with no permanent change in characteristics. However, in this region, due to the nonohmic behavior which occurs, significant deviations from the reported resistance vs %RH characteristics should be anticipated. The maximum wattage consistent with long-term stability of the sensor is 0.05W. The maximum current is dependent upon the electrode spacing and is approximately 8 ma for the 0.015-inch sensor and 12 ma for the 0.004-inch sensor.

AC current has been used in testing the new sensor to avoid the polarization and other effects which occur due to ion migration in the presence of direct current. The sensor should be operable with direct current at low applied voltages, but the measured resistance will depend upon the applied voltage and may require some time to reach a steady-state value. Tests with sensors of an earlier design showed that 10 μa DC could be used for extended periods with no permanent damage to the sensor.

HUMIDITY MEASUREMENT AND CONTROL USING THE NEW SENSOR

The performance features of this sensor and its low cost make it attractive for both humidity measurement and control applications. Its low impedance throughout the range of relative humidity greatly simplifies the required associated circuitry and permits use even at very low relative humidities.

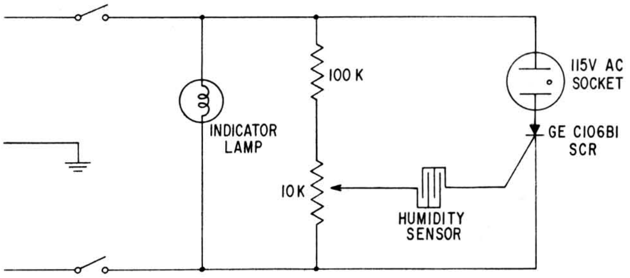

Fig. 9 Humidity control circuit I.

Humidity measurement can be accomplished most effectively by using the humidity sensor as the unknown resistance in an AC bridge circuit. However, a DC ohmmeter may be used to measure the sensor's resistance if measurements are made at low current levels; in this case, the above precaution regarding direct current polarization effects should be noted.

Considerable attention has been given to the sensor's use in control applications, whereby changes in the sensor resistance due to changes in relative humidity are used switch a load, such as a heater or a fan motor. A circuit diagram for a very simple and inexpensive control unit which has been used for demonstration purposes in this Laboratory is shown in Fig. 9. In this circuit, the sensor is used to gate an SCR, which can provide up to 3 or 4 amp of half-wave rectified AC current. The variable resistor allows the humidity at which AC current is switched to be varied. A somewhat more complex device, in which the sensor is used to activate separate relays for both a "low" and a "high" humidity response, is shown in Fig. 10. A "dead" zone of ±5% RH around the variable set point provides a condition where neither relay is closed; this avoids excessive rapid switching at the relay. This device was designed and built by the Engineering Model Shop at CR&D for use in experimental plant growth chambers. In this application the relay on the "high humidity" side is used to open a solenoid valve in a refrigerant system to lower the humidity, and the one on the "low" humidity side to turn on a heater in a water bath to raise the humidity.

COMPARISON WITH OTHER SENSORS

The most widely used types of sensors for humidity measurement and control applications are the mechanical, wet-bulb and electrical types. Of these, the mechanical sensors, which depend upon the dimensional changes produced in certain materials by changes in humidity, are by far the most popular. The materials employed here are generally either hair or nylon fiber. Their elongation and contraction can be used to move a pointer on a dial, activate a microswitch, or open a valve for humidity measurement or control purposes. The relative simplicity and low cost of the mechanical devices have contributed greatly to their popularity. However, they leave

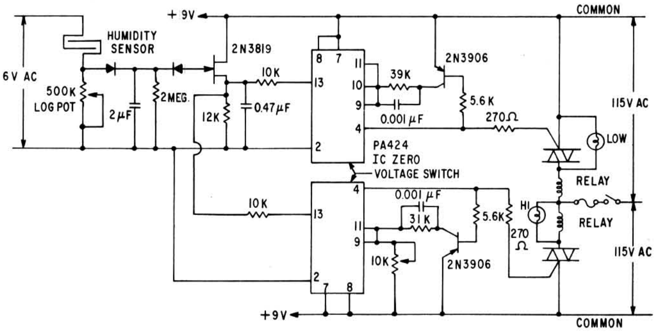

Fig. 10 Humidity control circuit II.

much to be desired in terms of accuracy, sensitivity, and reliability. Severe hysteresis, slow response, and a drift in calibration due to stretching of the fibers are characteristic of these devices.

Wet-bulb type sensors depend upon the lowering in temperature which occurs upon evaporation of moisture from a surface; the magnitude of the lowering in temperature is a function of the relative humidity. These devices generally use simple mercury thermometers as the temperature sensors and are widely used for humidity measurement. Modifications using thermistors as the temperature sensors have also been used for control purposes. However, the relatively high cost and frequent maintenance required for wet-bulb sensor control devices limit their usefulness in this respect.

Electrical sensors have also found widespread use for the measurement of relative humidity, particularly in scientific applications such as meteorology, where they are employed for remote measurements of the atmospheric moisture content. These sensors commonly consist of an ionic salt, such as LiCl, in an organic binder or a porous substrate; appropriately spaced electrodes are used to make electrical contact. Their response characteristics are quite dependent upon the nature of the salt, the substrate, and the electrode system used. In general, they are more sensitive, more accurate, are less subject to drift, and have a more rapid response than the mechanical sensors. On the other hand, they are generally more expensive, and are easily contaminated and damaged by foreign substances in the air and by condensation of water on their surface. The high impedance of most of these devices, particularly at low RH, their limited humidity range, and their high cost have restricted their usage for control applications.

Several other electrical sensors have been introduced which overcome some of the difficulties of the ionic salt sensor. One of these employs a polystyrene plate which has been treated with chlorosulfonic acid to produce an ionically conductive layer on the surface.(4) Electrodes, which are apparently carbon-based, are added to the surface by a silk-screening process. Although quite different in construction and composition, these sensors are reported to have response characteristics and stability similar to those measured for the new sensor described herein. However, tests carried out in this Laboratory on several of these sensors have indicated that the reproducibility of the resistance vs RH characteristics among sensors and hysteresis effects are considerably less satisfactory than reported. The chief drawback to this sensor, however, is cost as at the current selling price of $15 per sensor in quantities of 1000 or more, it is prohibitively expensive for many applications. In another type of electrical sensor a ceramic bead is used as the humidity sensitive component.(5) This sensor is reported to be stable toward water immersion and calibration drift under normal operating conditions, and has the added advantage of temperature stability to 150°C. However, the reported degree of reproducibility between sensors is rather poor (±10%), requiring individual calibration of the sensors for reasonable accuracy. Also, the rather high resistance range, 10^5 to 10^{10} Ω for 100% to 0% RH, limits its usefulness for control applications and for measurements in the low %RH range.

Of the wide variety of other devices currently available for general-purpose humidity measurement and control applications, none exhibit the unique combination of rapid response, high sensitivity, excellent stability toward a wide variety of environmental conditions, and low cost, which are characteristic of the new sensor described herein.

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