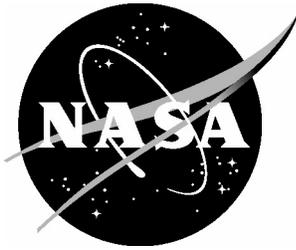


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Characterization of Polymer-Coated MEMS Humidity Sensors for Flight Applications

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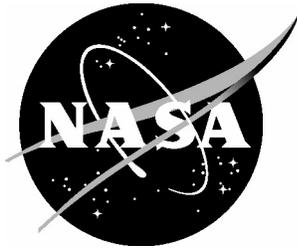
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Abstract

The rapid and accurate measurement of humidity has challenged the scientific community for a long time. Under NASA's Aviation Safety Program (AvSP), in addition to wind velocity and temperature, water vapor is considered a key factor in determining aviation weather, a substantial contributor to many general aviation (GA) accidents. The conventional and reliable humidity measuring methods, such as radiation reflection or absorption, have relatively high cost in addition to highly specialized operating and maintenance characteristics.

During the last 15 years, an emerging Micro-Electro-Mechanical-System (MEMS) technology, coupled with the latest cutting edge technologies for smaller, faster, and inexpensive systems, has opened the way for development of capacitance type and cantilever-based MEMS humidity sensors, which use industrially proven thermoset polymer coatings. Both types of humidity sensors were subjected to ambient environmental conditions, to deicing fluids and other aviation-related chemicals. The data of relative humidity (%RH) vs. output (mV) of these sensors with exposure to different types of deicing fluids and aviation related chemicals was taken at fixed temperatures and is reported in the paper. The 2-D and 3-D Atomic Force Microscope images were also taken to analyze the sensor polymer thin films. The polymer surface thin films were soaked in isopropyl alcohol, and tests were repeated to determine whether these sensors recovered from any damage.

This paper presents a characterization of these sensors for their potential use on aircraft. If installed, these sensors are subjected to ambient environmental conditions as well as to different chemicals and deicing fluids used on aircraft. This paper reports the effect of different deicing fluids and chemicals on these inexpensive humidity sensors. The long-term effects of different chemicals and isopropyl alcohol on polymer coatings are still to be determined.

Introduction

The wind velocity and temperature data have been available on commercial aircrafts since the late 1970s (ref. 1). FAA initiated an aviation weather program in early 1990s (ref. 2). A small subset was Commercial Aviation Sensing Humidity (CASH) (ref. 3). Data on the vertical structure of water vapor in the lower atmosphere is important for a variety of reasons, including aviation safety and weather predictions. One task of this program was to develop sensors to accurately measure water vapor on commercial aircrafts. In addition to their faster response and enhanced accuracy to measure water vapor in the lower troposphere, the sensors were required to perform with unaltered accuracy for at least three months. Other required features for the sensors were their easy installation on the existing aircraft and their unattended performance. The CASH program was completed in 1994 and potential problem areas were documented.

In 1997, a White House Commission led by the then US Vice President Al Gore mandated to reduce accident rates by a factor of 10 within the next 20 years. NASA responded by organizing an Aviation Safety Program, AvSP. As part of this safety program, NASA initiated a program named AWIN (Aviation Weather Information) to develop technologies that will eventually reduce weather-related aviation accidents (ref. 4).

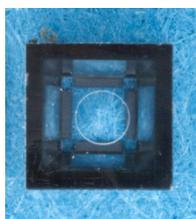
This paper reports, in brief, the background of the evolution of water vapor measurement techniques. Today, a variety of humidity sensors are commercially available. During the last 15 years, an emerging Micro Electro-Mechanical-Systems (MEMS) technology coupled with the latest cutting edge technologies for smaller, faster, and inexpensive systems have opened the way for development of new types of humidity sensors such as cantilever-based MEMS humidity sensors. A small and inexpensive capacitance type humidity sensor has been in the market for the last twenty years. These inexpensive sensors use industrially proven thermoset polymer coatings. This paper presents characterization of these sensors for their potential to be used on aircrafts. When installed, these sensors will be subjected to hostile ambient environmental conditions as well as to different chemicals and deicing fluids used on aircrafts. This paper reports effect of different deicing fluids and chemicals on these low cost humidity sensors.

The practice of measuring relative humidity is quite old. In 179 B.C., the Chinese made the oldest recorded humidity measurement system by using a “balance type” hygrometer. In 1450, Nicholas described this hygrometer in these words: “If someone should hang a piece of wool, tied together on one end of a large pair of scales, the weight of the wool would increase when the air becomes more humid and decrease when the air tends to dryness.” In 1550, the device was improved by substituting a sponge for the wool. When it was stretched between two fixed points with a weight fixed to its center, it responded to the change in humidity either by contraction or elongation depending upon increase or decrease in humidity. Different hygrometer versions evolved afterwards with the substitution of paper, hair, nylon, and acetate. During the seventeenth and eighteenth centuries, there were several opinions about how water dissolves in air. Finally, by 1790, an important principle was established—aqueous vapors have the properties of gases. As data accumulated, scientists started establishing a relationship between humidity and temperature (ref. 5). De Saussure established an extensive table for various humidities at different temperatures. Later in 1803, L.W.Gilbert claimed “The degree of humidity depends on the ratio of the vapor actually present to that which is possible.” Because of growth in the electronics field since then, different types of humidity sensors and measurement techniques have been established.

Several old techniques are still in use, but over the years, several new humidity sensors have been developed. Over the last several decades, mechanical hygrometry has evolved into state-of-the-art strain-gage sensors. The first strain-gage device was built in 1960 by using metallic beams instrumented with discrete silicon gages, which were bent by natural organic structures (ref. 6).

The sensors assembled from organic crystallite elements are reliable but follow a type 2 (nonlinearity and wide hysteresis) adsorption curve of bound water. The next generation strain-gage sensor was developed in 1970 by using a cellulose crystallite element that was held in full shear restraint. These sensors produced a linear output from 0–100 percent RH with very low hysteresis (ref. 7). These sensors, though relatively expensive, are reliable, linear, accurate and capable of withstanding extremely hostile environments.

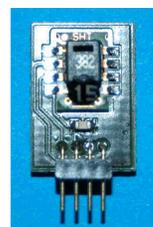
The next generation sensors were developed in the early 1980s and 1990s when capacitance type and cantilever beam-based MEMS sensors were developed respectively. Some of the sensors are shown in figure 1. The cantilever beam-based MEMS sensors and capacitance type sensors both use industrially proven thermoset polymer coatings to detect water vapors. Both these types of sensors are commercially available at a reasonable price (refs. 8–10). The sensors produce low hysteresis and reliable response. The results can be further improved with built-in signal conditioning.



(a) Cantilever based MEMS humidity sensor.



(b) Capacitance type humidity sensor.



(c) Capacitance type humidity sensor.

Figure 1. Commercially available cantilever beam-based MEMS and capacitance type humidity sensors.

Thin Polymer Films, Key Factor For Small-Scale Humidity Sensors

Thin polymer films have found widespread application in silicon-based microelectronic devices due to their unique material properties. Polyimides, polymers that contain recurring amide groups as integral parts of the main polymer chains, have been extensively used in the aerospace and electronics fields because they are thermally stable, mechanically strong, and electrically insulating (refs. 11–13). Interest in these materials is particularly high where miniaturization, large-scale integration, and high-speed signal processing in semiconductor-based components are important factors. Such demands require varied properties of polyimide materials. Accordingly, polyimides of different chain structures are used for different applications (refs. 12 and 13). In case of small-scale humidity sensors, commercially available polyimide is spin coated on cantilever beams. As a result of the spin-coating process and subsequent thermal processing, polymer layers show in-plane orientation (ref. 11). When exposed to a humid environment, these polymer thin films tend to expand due to the absorption of water molecules (refs. 12 and 13). In some of the previous studies, the swelling of thin polyimide films has been found to be anisotropic (ref. 14).

Mechanism of Water Sorption in Polyimide Films

Polyimides have relatively high uptakes of water that has a significant effect on the dielectric constant of polyimides. It causes increased conductivity of dielectric insulators and promotes corrosion of the conducting metal, which leads to potential device failure (refs. 15–17). Properties of these films can be changed by different methods like ion implantation and incorporation of fluorine during deposition. Both the dielectric constant and the water uptake of polyimides can be reduced by incorporating fluorine into the polymer (refs. 18–20).

Decreasing the imide content of the polymer is another way to change film characteristics (ref. 18). Because of high permeability, polyimide films can also be used as membranes for water vapor separation (refs. 21–23). The water vapor sorption and diffusion properties of polyimides are very important for their use in humidity sensors. Investigations of water vapor sorption and diffusion properties of polyimides have been widely reported [24–37].

Solubility (S) and diffusion coefficients (D) are determined with the transient sorption and desorption experiments by measuring water vapor uptake on freestanding polymer films. The process by which vapors are transmitted depends on the structure of the polymer membrane or film. For a polymer film with gross pores, mass transport occurs when there exists a pressure difference across the pores, and by ordinary or Fickian diffusion in the presence of a concentration difference across the pores (ref. 38).

Though polymer thin films are desirable without pores, an activated diffusion type penetrant transport depends upon pores films in which the gas dissolves in the film at higher concentration surface, then migrates through the film towards lower concentration surface under a gradient. The absorbed water vapor mass depends on the mass of the polymer itself. The mass of the absorbed water also depends on the temperature, so it is important that all measurements be performed at a constant temperature. Regarding the annealing conditions of polymer films, it has been reported that the water uptake and expansion as well as the degree of non-linearity is influenced by the annealing conditions (ref. 39).

The amount of sorbed water also depends on the chemical structure and the morphology of the polymer chains. The larger film densities because of higher anneal temperature lead to reduced mass uptake.

Cantilever-Beam-Based MEMS Humidity Sensor

This sensor works on the properties of low linear hysteresis and reliable response of the polymer film spin coated on the silicon cantilever beams. The beams are part of the micro-machined MEMS sensor that has a built-in Wheatstone Bridge. The application of a fully restrained polymer film on a strain gage instrument employs van der Waals forces to produce a linear output in a 4-arm bridge configuration. The sensor is low power and has the potential to be operated in hostile environments. Polymer thin films on strain gages are exposed to the environment. There is also a hole drilled at the center of the 2-mm × 2-mm humidity chip, which helps in rapid response of the sensor as well as contamination disposal when the cantilever beams are washed with liquids such as water or isopropyl alcohol.

Capacitance-Type Humidity Sensor

Capacitance-type polymer sensors use polyamide or cellulose acetate polymer thin films deposited between conductive electrodes. The thin film acts as a capacitor dielectric medium that changes its dielectric constant as the moisture is adsorbed. An alternative construction method uses a porous top metal layer that allows moisture transmittance. There is another polymer layer that protects the capacitor from contamination such as dirt, dust, and oils. Capacitor saturation has been identified as a source of temporary loss of data. When exposed to deicing fluids or other chemicals, the polymer coated thin film is degraded.

Exposure of Humidity Sensors to Deicing Fluids and Other Chemicals

Aircraft deicing is an important function because of potential frost formation on aircraft surface due to extreme cold weather. Ice formation can substantially impair an aircraft's ability to take off and fly. Aside from the obvious safety implications, this can cause reduction in lift, hence increased fuel usage and greater engine wear. Another hazard related to icing is the potential for damage when pieces of ice fly off during takeoff and landing. The purpose of this study is to understand the effects of deicing fluid and other aviation-related chemicals on small size and inexpensive humidity sensors currently available in the market.

Glycols and other deicing chemicals are efficient freezing-point depressants that lower the freezing point of the solvent when mixed with water. Propylene glycol and ethylene glycol have been used as anti-freeze and deicers for years because of their low cost. Ethylene glycol can lower the freezing point of water to approximately $-50\text{ }^{\circ}\text{C}$ while propylene glycol can lower the freezing point of water to about $-60\text{ }^{\circ}\text{C}$. Because of nontoxicity of propylene glycol, its market has grown from 10 percent to more than 70 percent during five years. A single application of type I deicing fluid to the critical surfaces of an

aircraft is the most common treatment method. Typically type I is about 90 percent glycol and 8 percent water, but water percentage can be varied depending on ambient temperature. Deicer performance is measured by holdover time, the length of time an aircraft can wait after being treated with deicing fluid. When a long holdover time is required, a second step is used. This is to treat the aircraft with an anti-icing fluid, like type IV. This coating contains a sufficient amount of polymer thickener.

The effects of type I and type IV deicing fluids on MEMS sensors under different humidity and temperature environments are discussed in this paper. The effect of different aviation related chemicals on sensors was studied and will be reported in this paper. These chemicals include commercially available deicing fluids, such as isopropyl alcohol, water (soaking, frozen in water for 24 hours), diesel fuel, and smoke.

Experimental Setup and Procedure

In this series of experiments that lasted for more than a year, there were several goals: (1) to investigate the practical performance and usability of the cantilever beam-based MEMS sensors, (2) to investigate the performance of capacitance-type humidity sensors, and (3) to study the effect of different deicing fluids and aircraft related chemicals on these sensors.

The MEMS characterization system consists of an Environmental Temperature/Humidity Chamber manufactured by Tenney Inc., a LabVIEW characterization program, a dc Power Supply, a precision 7-digit, 10-channel system multimeter, and the EdgeTech DewPrime II Chilled Mirror Dewpoint Hygrometer to measure the environmental test chamber temperature, dewpoint, and relative humidity. A block diagram of this system is shown in figure 2.

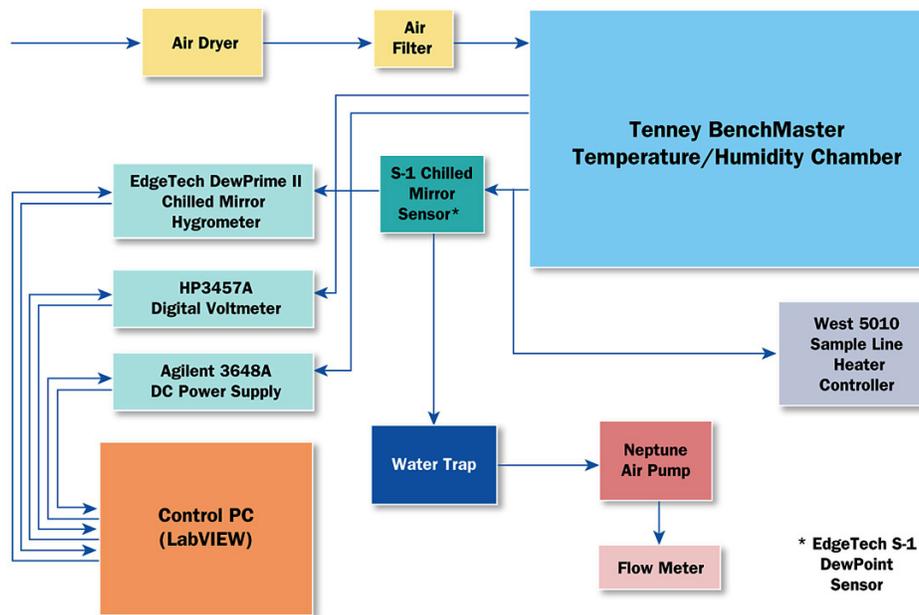


Figure 2. Temperature/Humidity Sensor Characterization System.

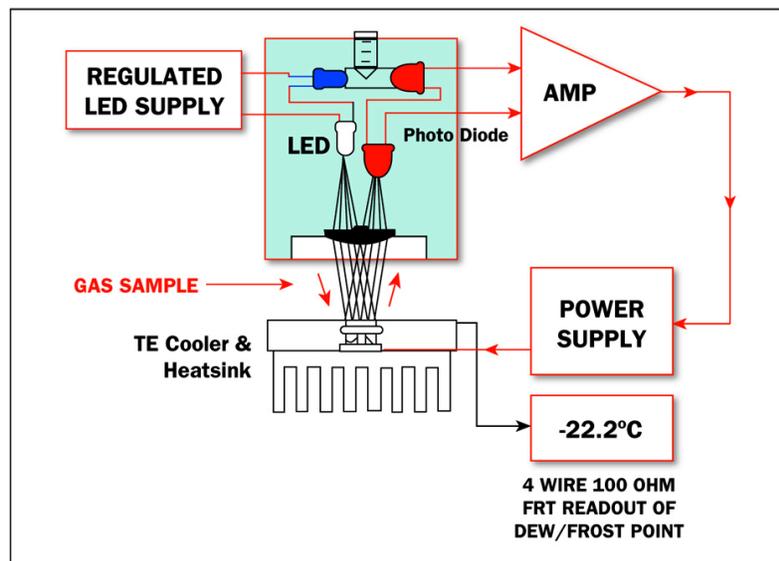
The LabVIEW program was developed in-house to allow flexibility in test requirements. The LabVIEW programming environment was used to develop a semi-automated temperature/humidity characterization system to measure sensor parameters at temperatures between -70° and $+95^{\circ}$ centigrade and at humidity levels from ~ 0 to ~ 100 percent. The LabVIEW program controls the test chamber parameters, monitors actual temperature and dew point inside the chamber, acquires sensor output and power data, records all data results in a spreadsheet format, and saves data under a user-selected file path.

The LabVIEW-based system allows the user to change the chamber humidity and temperature from the computer for measurements to characterize the sensors output voltage. The supply voltage to each sensor is also recorded and written to file because the sensor output voltage is a function of supply voltage, percent relative humidity, and the temperature. All chamber parameters are measured and recorded to assure repeatability and consistent chamber conditions for all tests. A typical test requires temperature changes in the intervals of 15°C over the desired range. Humidity steps are taken at 15 percent RH intervals at each temperature.

The environmental chamber is sealed during the tests to prevent infiltration of external air to ensure stable and accurate conditions for the internal environment of the chamber.

In the chilled-mirror technique, as shown in figure 3, a mirror made from a good thermal conductor, such as silver or copper, is used. The mirror is plated with an inert metal, such as irridium, rubidium, nickel, or gold to prevent tarnishing and oxidation. The mirror is chilled by using a thermoelectric cooler until dew just begins to form. A beam of light, typically from a solid-state light-emitting diode (LED), is aimed at the mirror surface, and a photodetector monitors reflected light.

As the gas sample flows over the chilled mirror, dew droplets form on the mirror surface, resulting in the scattering of the reflected light. As the amount of reflected light decreases due to scattering, the



Basic Chilled Mirror Operation

Figure 3. Basic chilled mirror operation.

photodetector output also decreases. This decrease in output in turn controls the thermoelectric heat pump via an analog or digital control system that maintains the mirror temperature at the dew point. A precision miniature platinum resistance thermometer (PRT), embedded in the mirror, monitors the mirror temperature at the established dew point.

If the mirror is controlled to an equilibrium condition just above the ice point, that is, 0 °C, the sensor measures the dew point. Below 0 °C, the deposit of the dew does not persist for long as liquid water. It is assumed that the deposit is frost and that the sensor measures the frost point. However, if the mirror is kept extremely clean, it is possible for dew to exist below 0 °C. The only sure way to verify that the sensor is controlling on the frost point is to visually inspect the mirror with a microscope. However, especially outdoors, it is typically impractical to maintain a perfectly clean mirror because contaminants, such as spores and other particulates, serve as nodes on which frost deposits can nucleate. Consequently, errors due to dew/frost point confusion at 0 °C are seldom encountered.

Characterization Matrix

A series of tests to characterize MEMS humidity sensors were performed as follows:

1. Characterization of polymer coated cantilever-beam-based MEMS sensors without signal conditioning unit.
2. Characterization of polymer coated capacitance type humidity sensor with built-in signal conditioning.

The environmental chamber was set at 10 °C with sensor excitation voltage of 1.25 (measured 1.2543019V). After temperature was stabilized (in about 30 minutes), 10 sets of data were taken and averaged with the LabVIEW program. Typical data of polymer coated cantilever-based MEMS sensor recorded for 10% RH, are given below:

Chamber °C (Set Point)	RTD °C (Measured)	Chamber %RH (Set Point)	Vaisala %RH (Measured)	Sensor Power V(DC) (Measured)	Output mV (Measured)
10	11.271961	10	6.499826	1.2543019	-5.78

The same procedure was adopted to take data at 20 °C, 30 °C, 50 °C, 70 °C, and 90 °C.

The sensor was exposed to different humidity conditions (10, 20, 30, 50, 70, and 90 percent) at different temperatures (10, 20, 30, and 50 °C). Variation of the sensor output (m-V) as a function of relative humidity (%RH) at a constant temperature is shown in figure 4(a). As expected, the output increases with an increase in humidity. Similarly, the sensor output increases with temperature for a constant RH percent. Thin films used for these humidity sensors are photo-imageable polyimide precursors that can be imaged on all exposure tools. They are suitable for a wide range of applications and for different substrates, including silicon, glass, alumina, and gallium arsenide. Properties of the final processed polyimide film are dependent upon the post-deposition temperature (ref. 40). If cured at 350 °C for 60 minutes, these polyimide films show virtually no degradation, even after 1000 hours of testing.

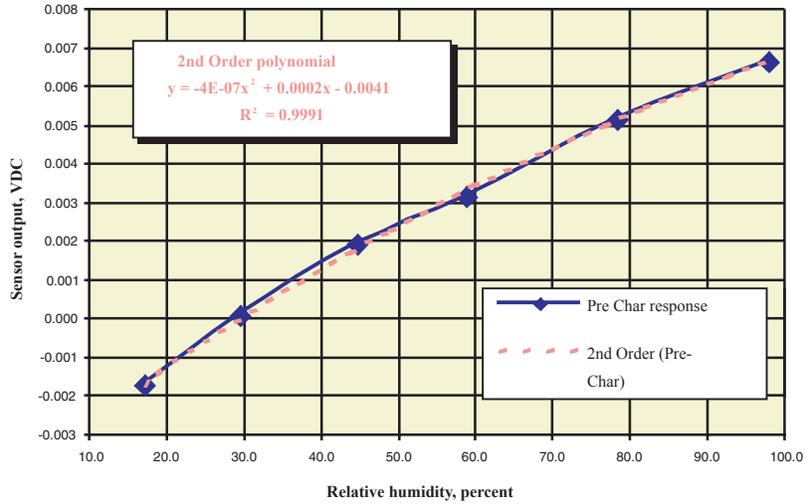


Figure 4(a). Cantilever-beam-based humidity sensor characteristics.

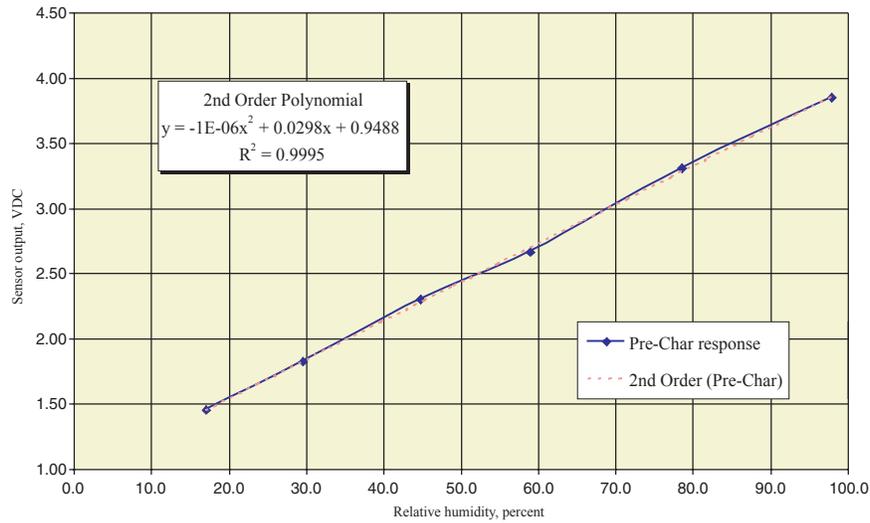


Figure 4(b). Capacitance-type humidity sensors characterization.

The process of spin-coating to deposit thin polymer films and the shrinkage caused by subsequent thermal processing results in an anisotropic expansion of the films, which tends to expand anisotropically when exposed to humidity. As seen in figure 4, the plot of relative humidity (%RH) vs. output (m-V) is nonlinear because of anisotropical expansion and three different sorption processes in a polymer film when it is exposed to humidity (ref. 41): (A) at low humidity levels, water is absorbed in micro-voids; (B) at humidity levels above 50 percent RH, absorption is dominated by cluster formation; (C) the third absorption, also known as Henry dissolution, occurs over the whole humidity range. This process is described by equation, $C_K = C_H + C_L + C_C$ where C_H is Henry sorption, C_L is Langmuir sorption, and C_C is clustering sorption. The first two sorption processes are nonlinear, while the third one responds in linear fashion. At low humidity levels, as shown in figure 4, water absorption can be modeled by means of Langmuir isotherms and causes the convex appearance. This process changes at humidity levels above 50 percent and absorption is dominated by cluster formation and causes the concave shape.

There are some other capacitance-type humidity sensors available in the market that use similar types of polymer coating. In operation, water vapor in the active capacitor's dielectric layer of these devices equilibrates with the surrounding gas, and the change in capacitance is measured. Figure 4(b) shows typical response of a capacitance type humidity sensor (ref. 9).

In this plot, again all three processes, Langmuir isotherm, cluster formation, and dissolution of water are apparent but on a smaller scale as linearity is improved because of built-in signal conditioning.

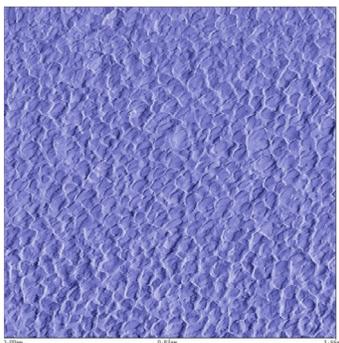
The principle upon which these small size humidity sensors (cantilever-beam-based and capacitance type) work depends upon the coating of polymer thin films. Measurement of RH percent directly depends upon absorption of water vapors by polymer films. The water vapor mass absorbed depends on the mass of the polymer itself, so it will be useful to relate the mass of the absorbed water to the mass of the dry polymer. Mass of the absorbed water, and consequently the saturation concentration, depends on the temperature, so it is important that all measurements be performed at a constant temperature. It has been reported that the maximum value of water uptake and expansion as well as the degree of nonlinearity for humidity measurement is influenced by the anneal conditions (ref. 39).

Thin film structure also affects the influence of sorption behavior. The performance and reliability of the polyimide films can be improved by changing the chemical and physical structures (ref. 42). High degrees of molecular ordering and in-place orientation of the polymer reduce moisture uptake but increase the humidity-induced stress in the film.

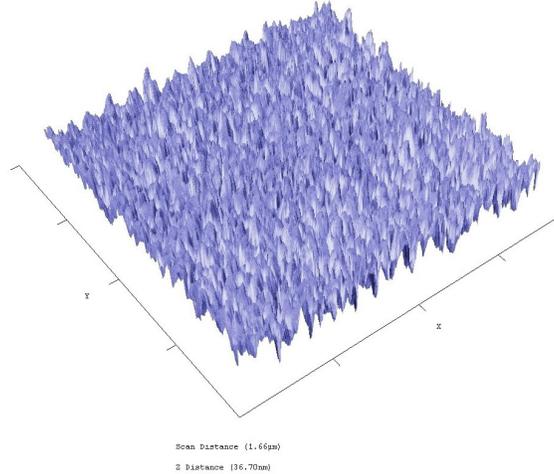
Polymer Surface Characterization

Many techniques are available today for characterizing polymer surfaces, including unique information on the chemistry and surface morphology. The most commonly used technique is the optical microscopy that can resolve $\sim 1 \mu$ images. Higher resolution imaging ($< 1 \mu$) is accomplished with a Scanning Electron Microscope (SEM). The SEM can resolve surface features up to a few nanometers; however, SEM does not provide high contrast images on flat films, needs a vacuum, and often requires a conducting coating on the surface of the test film. The Atomic Force Microscope (AFM) has the advantage that no special sample preparation is required. Images are usually obtained by raster scanning the tip over the surface, while a feedback system adjusts the tip-sample spacing to maintain the force constant. Hence, the image is a constant force contour map whose features often reflect the physical structure of the surface. In the present study, AFM was used to analyze polymer-coated films.

Here are 2-D and 3-D scans of typical polymer coating on humidity sensors



(a) 2-D scan ($1.66 \mu\text{m} \times 1.66 \mu\text{m}$).



(b) 3-D scan (1.66 $\mu\text{m} \times 36.7 \text{ nm}$).

Figure 5. AFM scans of typical polymer-coated surface sections of humidity sensor.

The scans in figures 5(a) and 5(b) provide visual and qualitative information on many physical properties, including size, morphology, surface texture, and roughness. Figures 5(a) and 5(b) show poly-type surface structure with an average roughness of 18 nm. Statistical information on size, surface area, and volume distribution can be obtained from these scans. Such analysis will be reported in another paper.

Roughness of Polymer Films on the Surface of Humidity Sensor

Quantitative measurement of the nanometer scale roughness provides surface texture and helps in measurement of polymer adhesive properties. On one typical polymer coating, shown in figure 6, three different spots were used to determine surface roughness.

Total length R_t , roughness average R_a , root mean square R_q , and 10 points height R_z were calculated as follows.

The average roughness is the integral of the absolute value of the roughness profile height over the evaluation length.

$$R_a = \frac{1}{L} \int_0^L |r(x)| dx$$

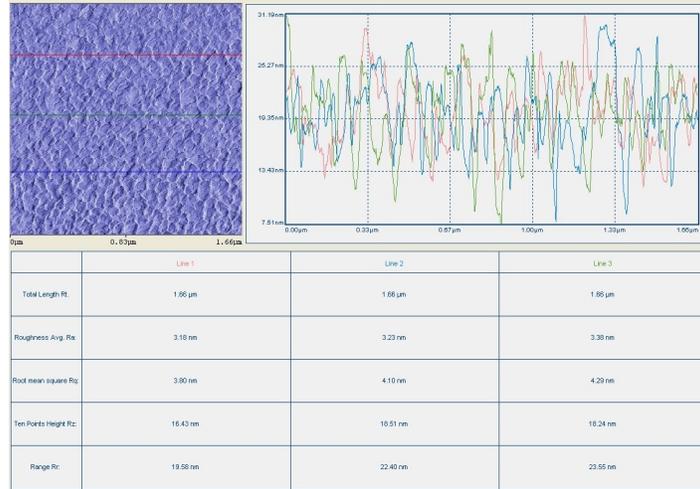
The root-mean-square (rms) average roughness of a surface is calculated from integral of the roughness profile:

$$R_q = \sqrt{\frac{1}{L} \int_0^L r^2(x) dx}$$

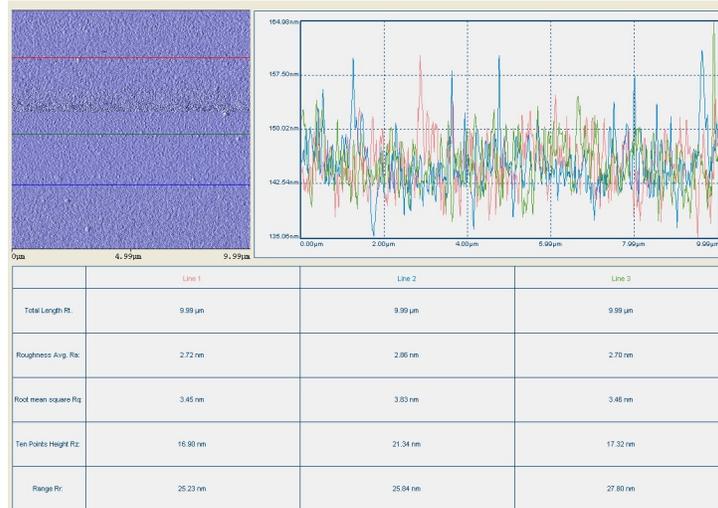
R_z is the sum of the height of the highest peak plus the lowest valley depth within a sampling length.

The calculated roughness values of typical polymer coated film are

$R_t = 11.65 \text{ nm}$, $R_a = 3.37 \text{ nm}$, $R_q = 4.25 \text{ nm}$, and $R_z = 20.84 \text{ nm}$.



(a) 1 μm.



b) 10 μm × 160 nm.

Figure 6. Average surface roughness for typical polymer coated humidity sensor.

Measuring the surface texture is critical. For example, surface texture can alter the optical properties of materials, control adhesive properties of polymers, and control the density of absorption. The relationship between surface roughness and absorption of water vapors will be reported in another paper.

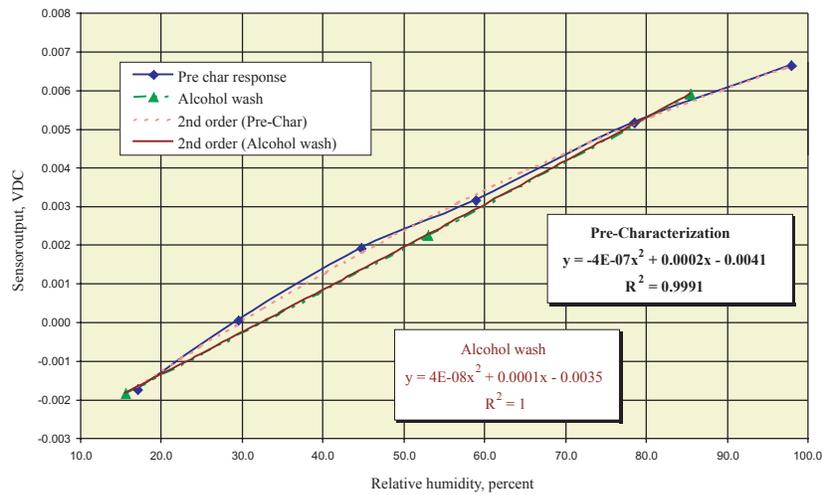
Effect of Isopropanol Alcohol on Humidity Sensors

Isopropyl alcohol is used for a wide variety of industrial applications, such as a solvent for resins, pigments, and many inorganic salts. It is also used as an extraction agent to purify natural gas and as a diluent in perfumes and paints. Both types of humidity sensors (cantilever-beam-based and capacitance type) were flooded with isopropyl alcohol for this test. This process involved soaking the sensors for approximately 30 minutes to ensure saturation in isopropyl alcohol and then drying them for 24 hours in the test chamber at 10 °C and 15% RH. The following plots show output data of both types of humidity

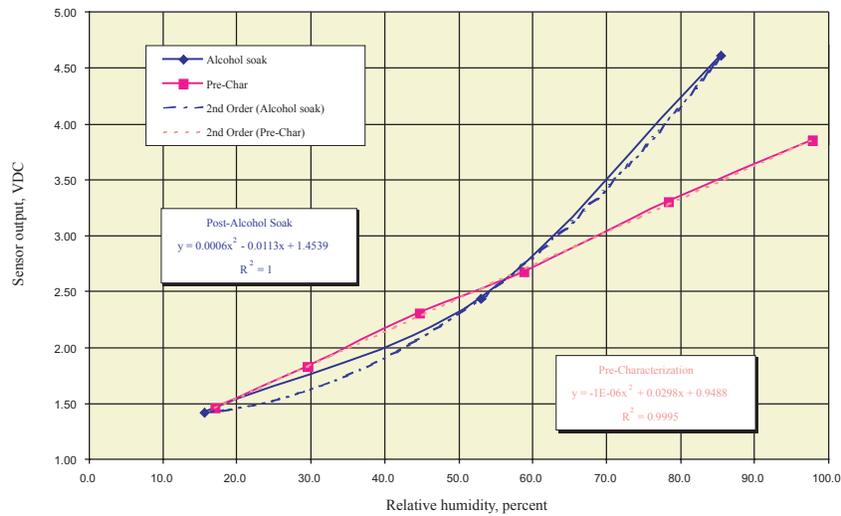
sensors before and after the isopropyl alcohol wash. Surprisingly, the linearity of a cantilever-beam-based humidity sensor, as shown in figure 7(a) is improved. The long-term effect of isopropyl alcohol on humidity sensors is unknown at this time and will be reported after results over a long period of time are available from these studies.

Figure 7(b) shows the response of capacitance type humidity sensor. After being soaked in isopropyl alcohol, the performance of the capacitance type humidity sensor deteriorates as shown in the figure.

Preliminary results show that when the cantilever-beam-based humidity sensor is washed with isopropyl alcohol, it improves the surface roughness—hence the linearity of the sensor. However, results are different for capacitance-type humidity sensors. Capacitance-type humidity sensors deteriorate when washed with isopropyl alcohol. One possible cause could be that isopropyl alcohol is absorbed in the dielectric material between the capacitance plates and causes degradation of the dielectric material.



(a) Cantilever-beam-based sensor.



(b) Capacitance-type humidity sensor.

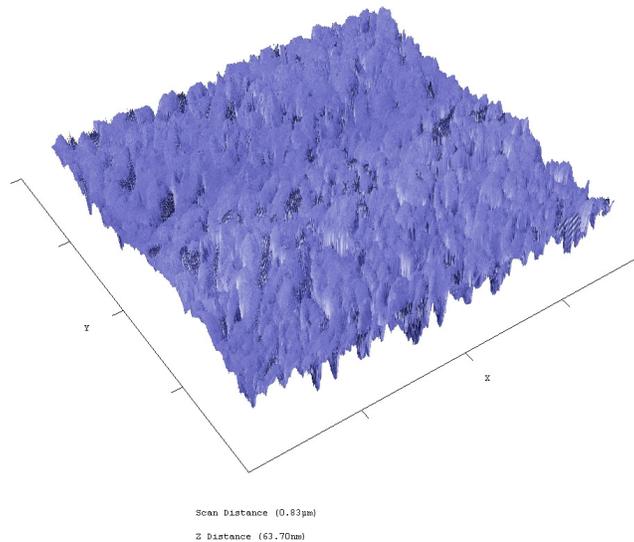
Figure 7. Sensors after soaking in isopropyl alcohol.

2-D and 3-D Images of Polymer Coated Films After Isopropyl Alcohol Wash

Surface roughness of polymer coated films improved after the isopropyl alcohol wash, as shown in figures 8(a) and 8(b). The reason could be attributed to the removal of contamination from the film that had accumulated since its fabrication. Also, some etching could have occurred that resulted in the smoothness of the surface to some extent. In any case, the linearity improves only for cantilever-beam-based humidity sensors and it deteriorates for capacitance type sensors. The reason could be that contamination on all four polymer coated cantilever beams are cleaned by the isopropyl alcohol wash and that they are completely dried up in 24 hours. Hence, polymer film responds to water vapors in an improved way, but that is not the case for capacitance type humidity sensors. The protective polymer layer on the capacitance type humidity sensor provides mechanical protection for the porous platinum layer as well as from contaminants such as dirt, dust and oils. When this sensor is washed with isopropyl alcohol, it cleans the first polymer layer and filters into the second polymer layer. This filtered isopropyl alcohol does not dry up in 24 hours (after which time the sensor was tested again). Hence the sensor characteristics deteriorates. Both these sensors will be tested for long-term stability, and the results will be reported on completion of those tests. The results reported in this paper are preliminary and not conclusive.



(a) 2-D AFM scan.



(b) 3-D AFM scan.

Figure 8. AFM scan of cantilever-beam-based sensor after soaking in isopropyl alcohol.

Effect of ABC-S Deicing Fluid

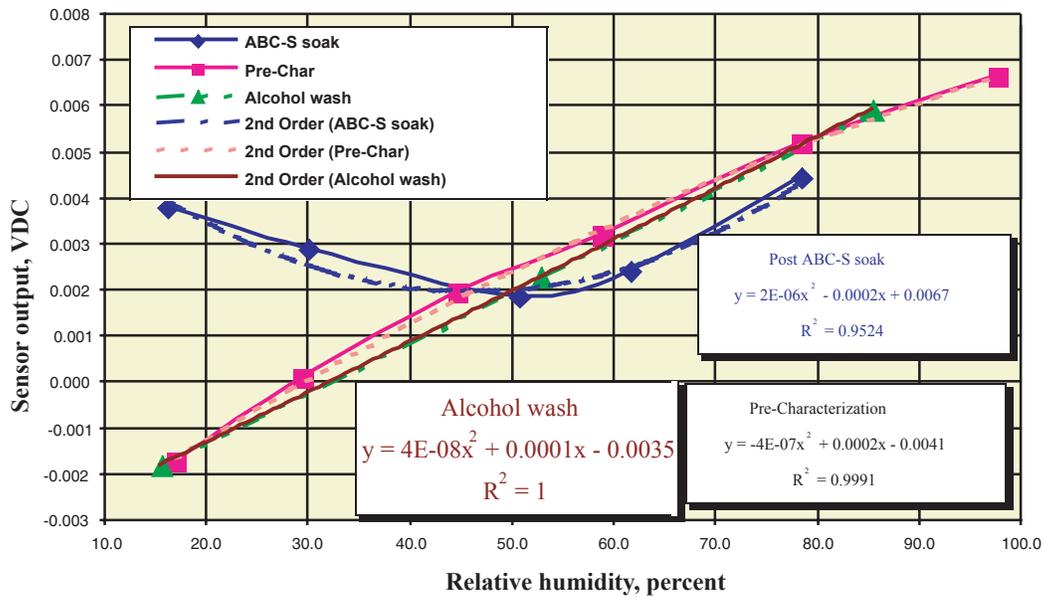
ABC-S deicing fluid is sprayed at the aircraft (as well as on the runway and taxi ways in some cases) to assist in the removal of frozen accumulations (snow and ice) that cannot readily be removed by mechanical means. As discussed earlier, the chemical deicing does not melt that ice but only breaks the bond between the ice and the surface it is sprayed on. Mechanical means are used to remove the accumulation once the ice/snow bond is broken. In the present experiments, ABC-S deicing fluid was sprayed on the cantilever-based and the capacitance-type humidity sensors. The sensors were then dried and characterized under different humidity environments. After each test, deicing sprayed sensors were soaked and cleaned with isopropyl alcohol.

Electrical characteristics of cantilever-beam-based and capacitance-type humidity sensors are shown in figures 9(a) and 9(b), respectively. It is interesting to note that the sensors do not respond after being sprayed with ABC-S deicer. The probable reason could be that micro-voids on polymer films are filled with propylene glycol and water vapors and are not absorbed through the surface of polymer films. When ABC-S deicer is washed away with isopropyl alcohol, the cantilever-beam-based MEMS humidity sensor not only recovered but also improved in linearity. It could not only be due to ABC-S deicer but also to the contamination on all four polymer-coated cantilever beams during washing off of the isopropyl alcohol and its subsequent drying within 24 hours. Thus, the polymer film responds to water vapors in an improved way. The capacitance type humidity sensor does not recover, even after wash with isopropyl alcohol. When this second sensor is washed with isopropyl alcohol, it cleans the first polymer layer and filters into the second polymer layer. This filtered isopropyl alcohol, mixed with contamination, does not dry within 24 hours, after which the sensor is retested. Sensor characteristics deteriorate instead of improving. An extended time testing of the two types of sensors is underway. The results will be reported upon completion of the tests.

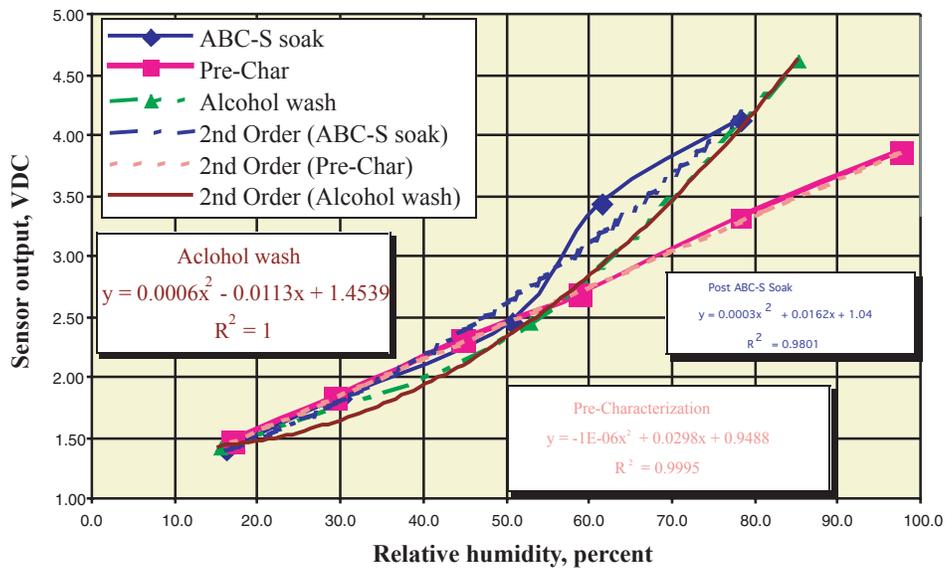
The 2-D and 3-D scans of polymer coated films after their cycling through 70 percent humidity are shown in figure 10. It appears that water vapors cause some of the deicing fluid to be washed away from the surface. Both 2D and 3D AFM scans, shown in figures 10(a) and 10(b) cover almost 50 percent of the polymer coated surface with sprayed deicing fluid. The rest of the polymer surface remains uncovered and is used as a humidity sensor. This feature results in both the sensors demonstrating humidity sensing characteristics.

Effect of DFT-63 Deicing Fluid

Both types of sensors were soaked in second type of deicing fluid, called DFT-63, and characterized under different humidity conditions. Both sensor types did not behave as linear humidity sensors after spraying of DFT-63 deicing fluid, as shown in figures 11(a) and 11(b). However, the effect was not as drastic as it was in the case of ABC-S deicing fluid. When these sensors were washed with isopropyl alcohol, the cantilever-beam-based humidity sensor not only recovered but also gave improvement in its humidity response. The second type of humidity sensor did not recover, even after washing with isopropyl alcohol. This behavior could be reasoned by the same argument as before in case of other deicing fluids. The isopropyl alcohol cleans the first polymer layer and filters into second polymer layer. This filtered isopropyl alcohol does not dry up during the 24-hour waiting period for a rerun leading to the sensor response. Long-term testing of both these sensors is underway. The final results will be reported later in a separate publication.

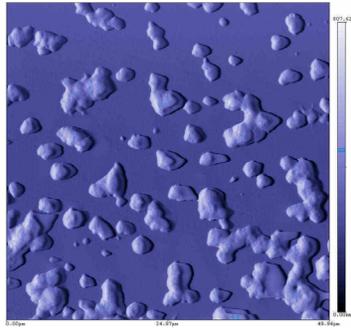


(a) Cantilever-beam-based sensor.

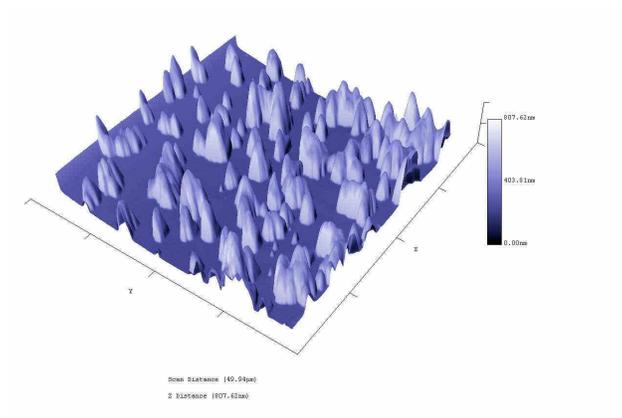


(b) Capacitance-type humidity sensor.

Figure 9. Sensors soaked with ABC-S Deicer, and then washed with isopropyl alcohol.

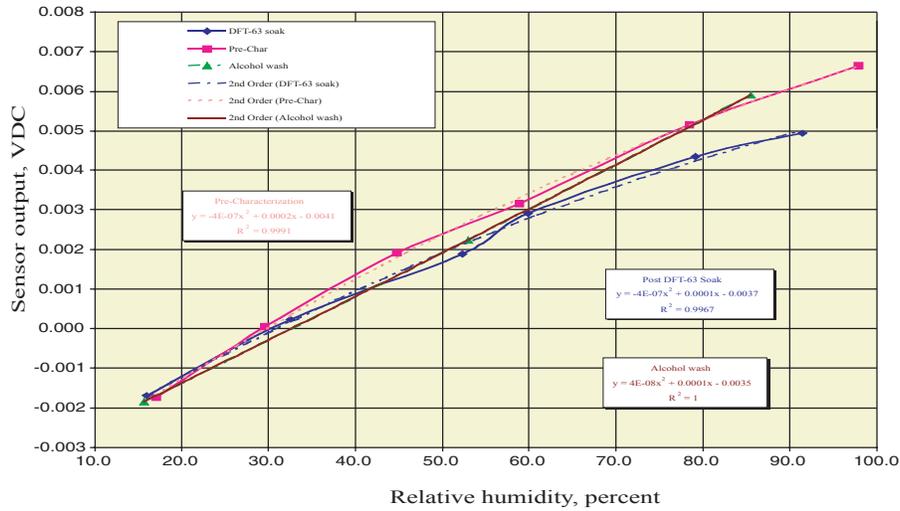


(a) 2-D AFM scan.

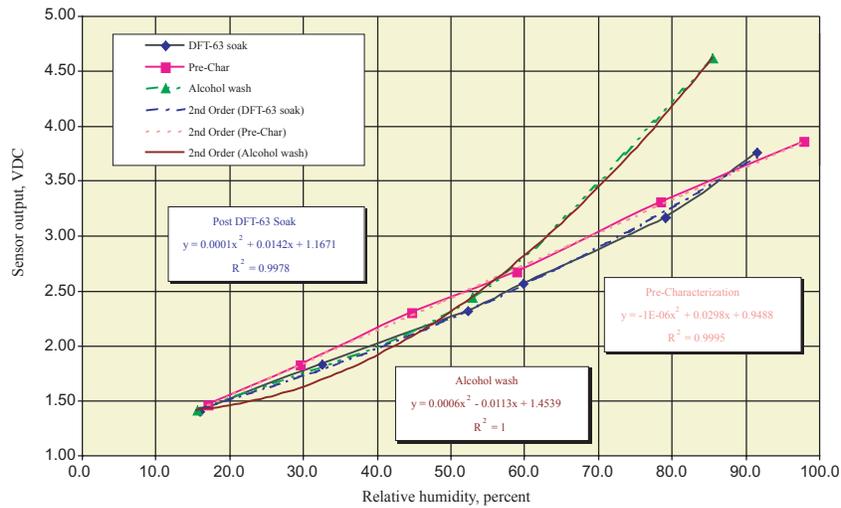


(b) 3-D AFM scan.

Figure 10. AFM scans of cantilever-beam-based humidity sensors with ABC-S Deicer. The sensor was dried before taking AFM scan.



(a) Cantilever-beam-based humidity sensor.



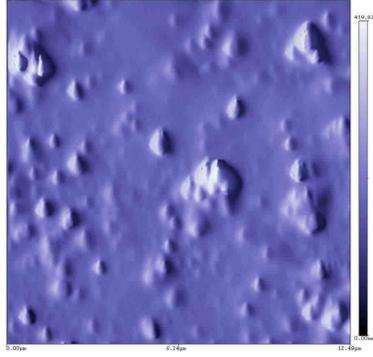
(b) Capacitance-based humidity sensor.

Figure 11. Characterization of humidity sensors with DFT-63 percent deicer sprayed and then washed with alcohol.

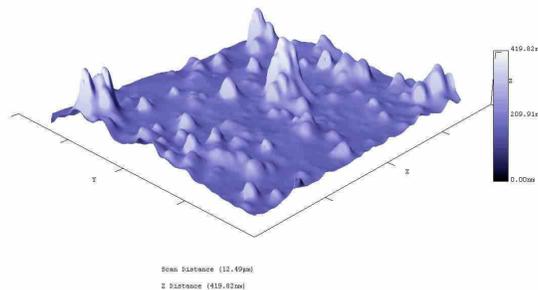
Figures 12(a) and 12(b) are scans of polymer coated films after being cycled through 70 percent humidity. It appears that water vapors help some of the deicing fluid to melt and be washed away from the surface.

Water Soak Experiment

Humidity sensors, when installed on the test object (aircraft), are likely to be soaked in water during the washing of the aircraft. Tests were conducted to examine the performance of soaked humidity sensors. To perform water soak tests, the sensors were dropped into a 4 oz bottle of water and sealed for 96 hours to ensure that they were totally saturated. The sensors were re-inserted into the test fixture and allowed to dry at 100C/15% RH for a period of 2 days. The sensors were then retested to check for any shift in the calibration.



(a) 2-D AFM scan.



(b) 3-D AFM scan.

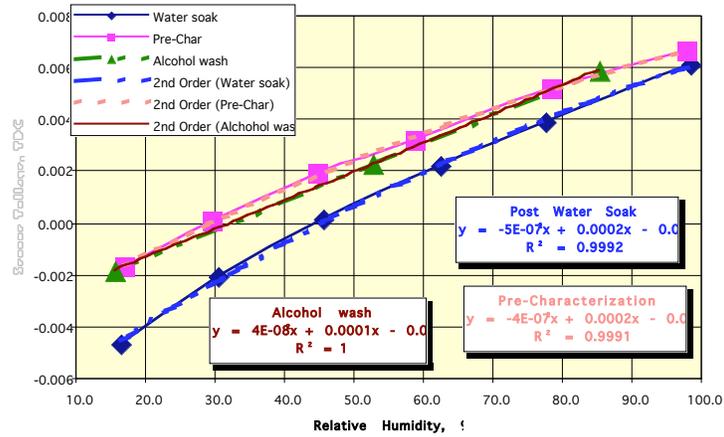
Figure 12. Scans of cantilever beam-based humidity sensors after soaking with DFT-63 percent deicer and then characterizing at 70% RH.

For cantilever-beam based humidity sensor, output characteristics line shifts toward lower values as shown in figure 13(a). Soaking changes the conductivity of the test arm of the Wheatstone bridge resulting in such characteristics. The capacitance type humidity sensor stops acting as a humidity sensor because the active capacitor's dielectric layer is merged in water and does not behave as a dielectric layer any more. See figure 14.

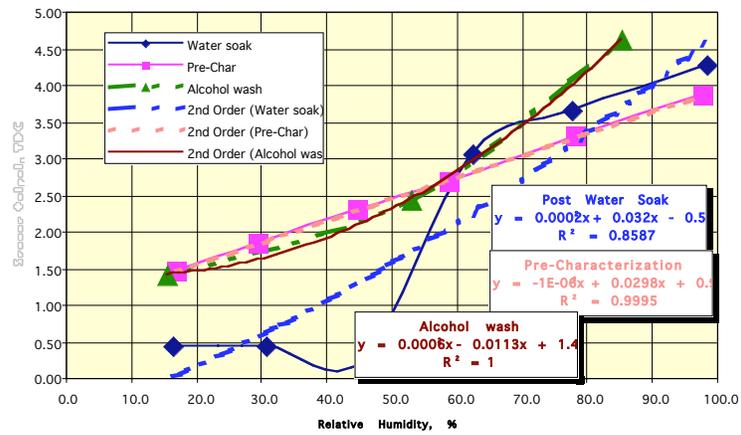
From these scans, it appears that the water reacts with polymer thin film and forms clusters that deteriorate the performance of humidity sensors.

Roughness of Water-Soaked Humidity Sensor Surface Polymer Films

Quantitative measurements of nano scale roughness provide surface texture and help in the measurement of polymer surface properties. On one typical polymer coating, shown in figure 15(a), three different spots were used to determine surface roughness. The surface texture evaluation is on the previously mentioned 96-hour water-soaked polymer surface. The surface roughness measurements also depict cluster formation at different spots.

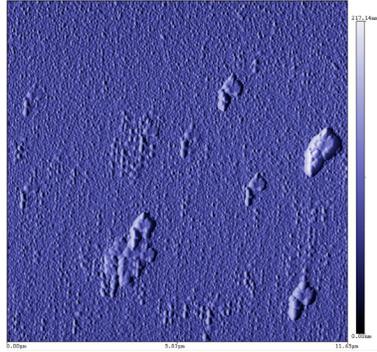


(a) Cantilever-beam-based humidity sensor.

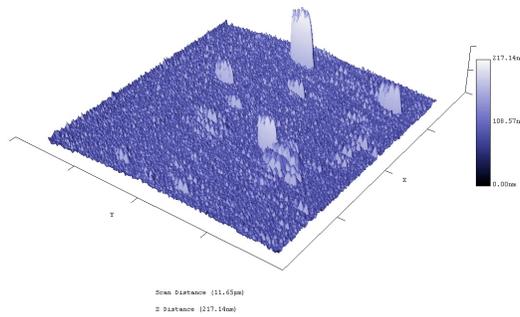


(b) Capacitance-type humidity sensor.

Figure 13. Characterization of humidity sensors after water soaking. Sensor output, VDC is shown.



(a) 2-D AFM scan.



(b) 3-D AFM scan.

Figure 14. Scans of cantilever-beam-based humidity sensor after soaking in a 4-oz bottle of water and being sealed for 96 hours. These sensors were dried for 48 hours afterwards.

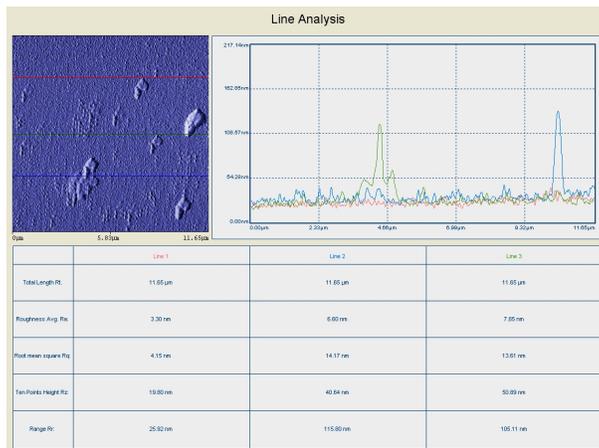


Figure 15. Average surface roughness for typical polymer coated humidity sensor.

Humidity Sensors Frozen in Water for 24 Hours

When the temperature drops below freezing, and especially when it stays there for long hours during flight or when the aircraft is on the ground, water droplets on the surface of the MEMS humidity sensor freeze. Whatever causes water to freeze on the surface of humidity sensors, it is interesting to see its effect on sensor performance. The MEMS humidity sensors were frozen in water for 24 hours. The sensors were allowed to dry at 10 °C/15% RH for 2 days. The sensors were retested for any shift in calibration. The results are shown in figures 16(a) and 16(b).

Sensors were cleaned with methyl alcohol after exposing MEMS humidity sensors to frozen water for 24 hours (fig. 17). The test results indicate that whenever MEMS sensors with frozen water are soaked and/or washed with alcohol, the polymer films are ready to absorb water vapors through micro-voids (as would virgin films) and helped the MEMS cantilever-based humidity sensor to retain its linearity.

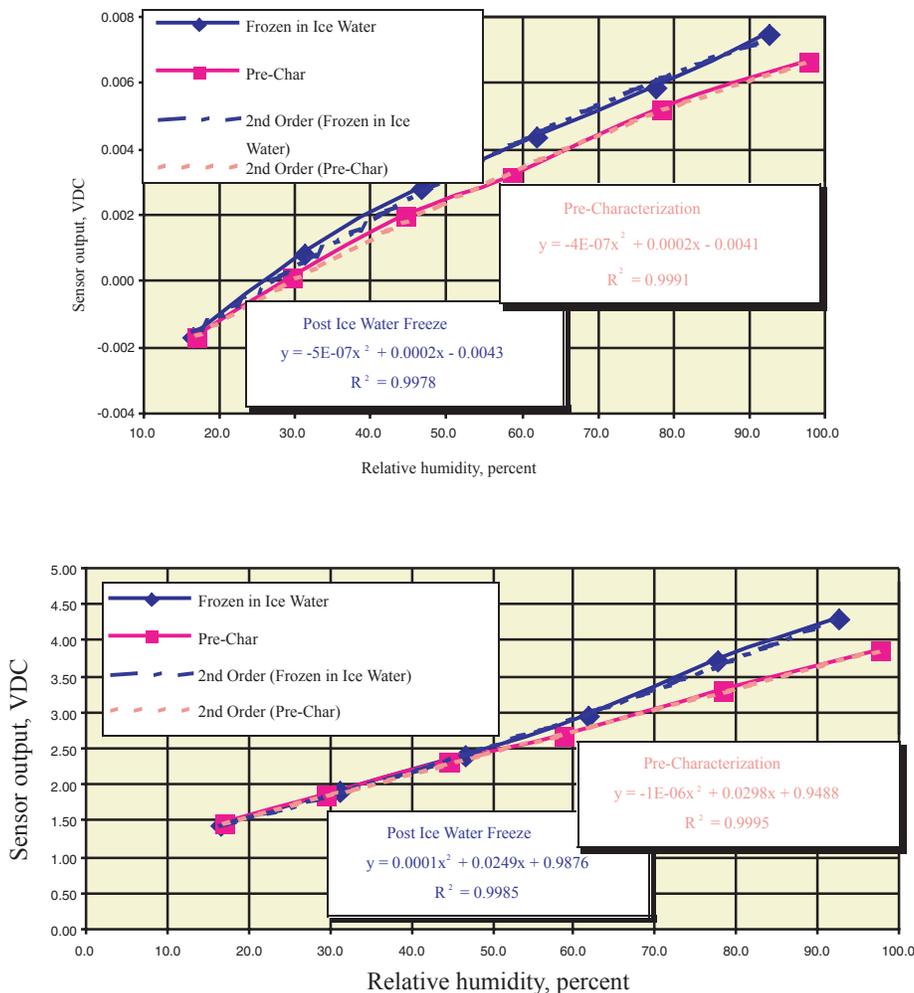
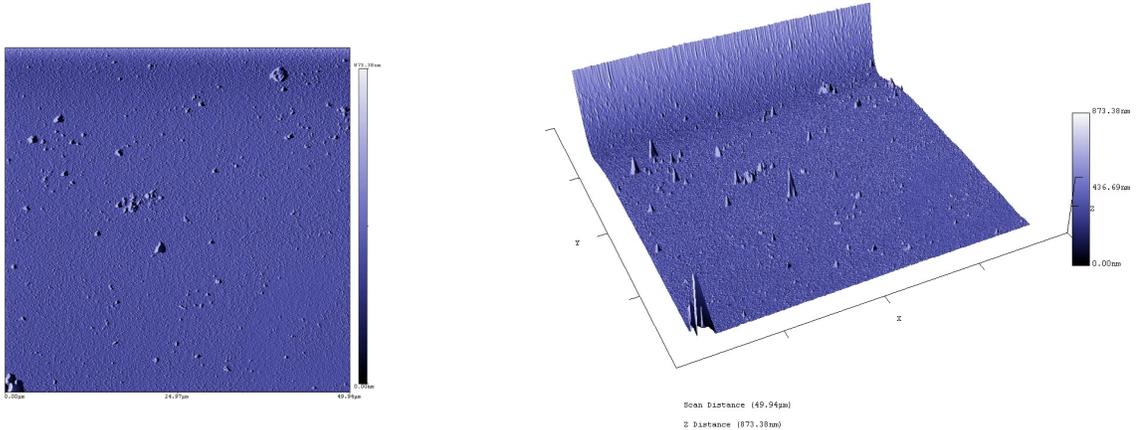


Figure 16. Sensors frozen in water for 24 hours results in absorption of water vapor trapped in micro-voids becoming more dense with higher output of frozen water.



(a) 2-D AFM scan.

(b) 3-D AFM scan.

Figure 17. Scans of cantilever-beam-based humidity sensors after being frozen in water for 24 hours.

Roughness of Humidity Sensor Surface Polymer Films Frozen for 24 Hour

Quantitative measurement of nano scale roughness provides surface texture and helps in measurement of polymer surface properties. On one typical polymer coated cantilever-beam, three different spots were used to determine surface roughness. This surface texture shows humidity sensor surface polymer films frozen for 24 hours. The results are shown in figure 18.

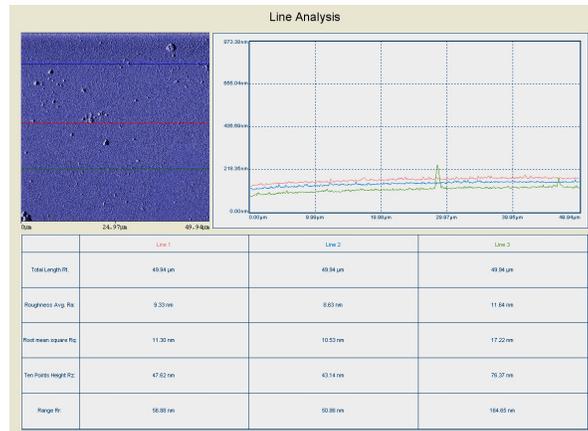
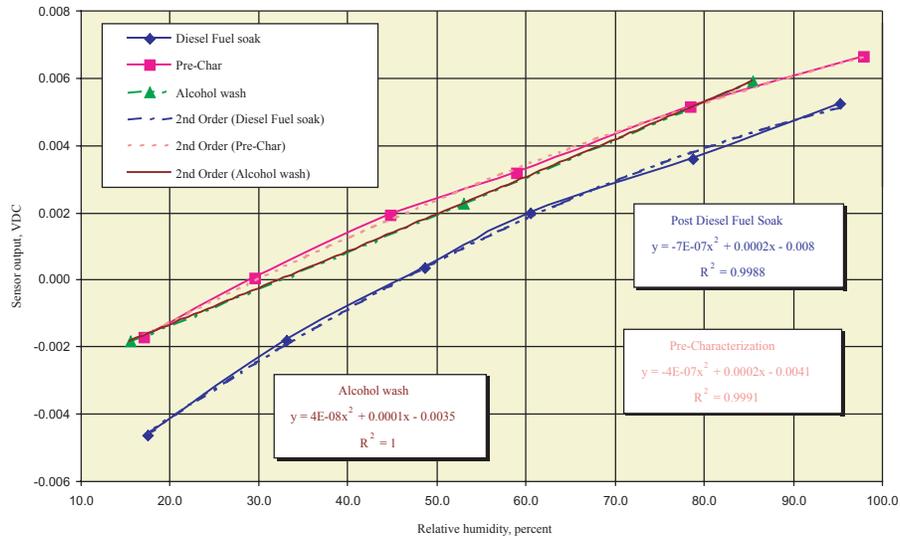


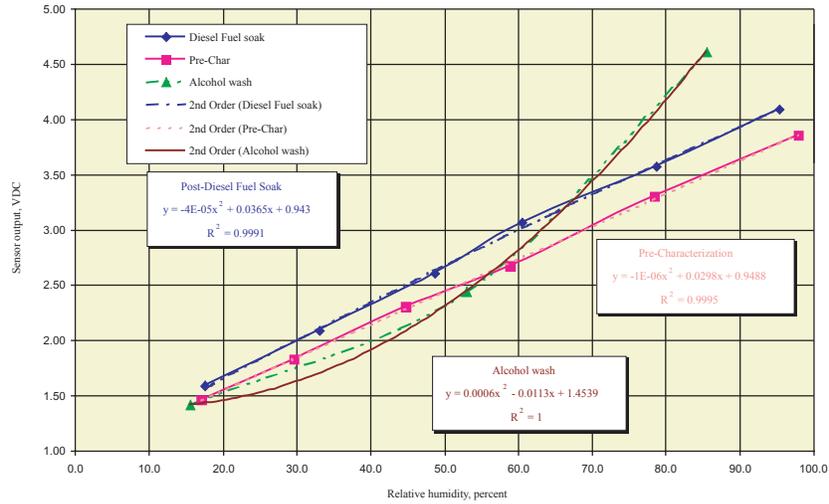
Figure 18. Average surface roughness of polymer coated humidity sensor frozen for 24 hours.

Diesel Fuel Soak

Effect of diesel fuel on sensors was measured by first soaking them in commercially available diesel fuel for 4 hours and then cleaning them with methyl alcohol. The sensors were allowed to dry at 10 °C/15% RH for 2 days. The sensors were retested to examine for any aftereffects on the polymer coated thin films. The results are shown in figures 19(a) and 19(b). Diesel, sprayed on the sensor polymer film, provides a barrier to the water vapor sorption and slows the response time. The sensor response



(a) Cantilever-beam-based sensor.

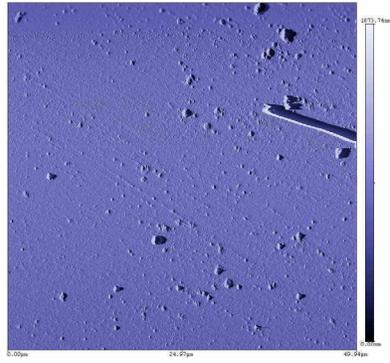


(b) Capacitance-type sensor.

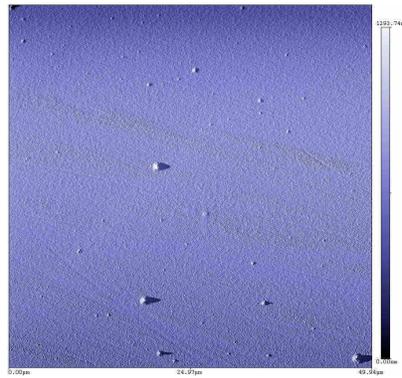
Figure 19. Polymer coated humidity sensor response after diesel fuel soak.

shifts at low voltages as a result of the reduction in percent of water vapors on the polymer films. When the polymer film is washed with isopropyl alcohol, diesel is washed away and the sensor recovers its original characteristics in the cantilever-beam humidity sensor. In the capacitance type sensor, the diesel fuel filters into a second polymer layer. The diesel does not deteriorate the polymer film but responds to the water vapor in a different way. When the capacitance type sensor is washed with isopropyl alcohol, it also filters into the second polymer layer and does not behave as a linear humidity sensor. It could also be due to incomplete evaporation of the isopropyl alcohol. The long-term stability of the sensor response is being tested and the results will be presented later in a separate paper.

Spray of diesel fuel and its subsequent drying creates visible contamination spots on the test surface. Most contamination spots are cleared from the surface upon washing with isopropyl alcohol, thus improving the result of cantilever beam-based sensors. See figures 20 and 21.

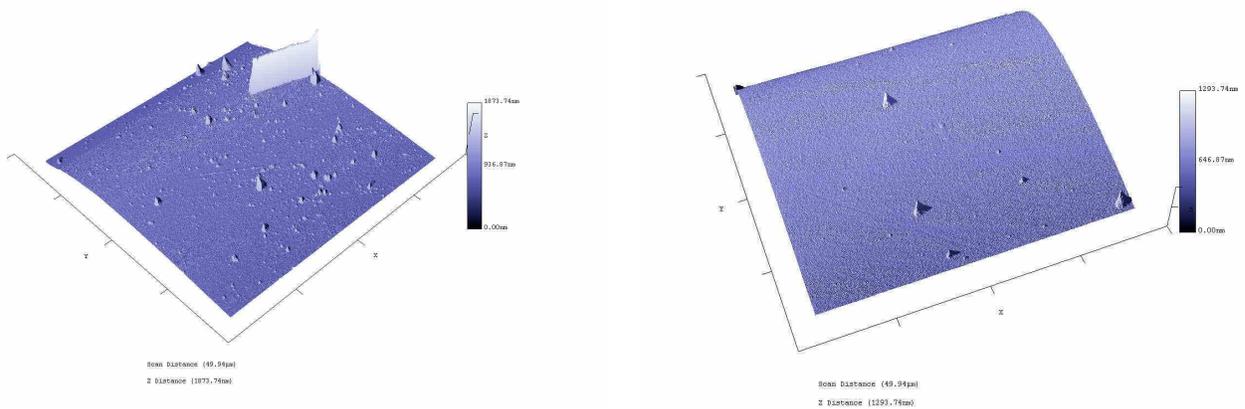


(a) After exposure to diesel fuel.

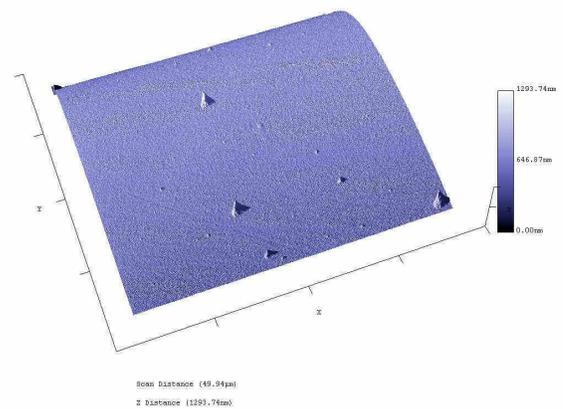


(b) After isopropyl alcohol soaking.

Figure 20. 2-D AFM scans of cantilever-beam-based humidity sensors.



(a) After exposure to diesel fuel.



(b) After soaking in isopropyl alcohol.

Figure 21. 3-D AFM scan of cantilever beam-based humidity sensors.

Again, the result is same from the 3-D view. We see several contaminant spots on the surface of the cantilever beam sensor that are washed away after isopropyl alcohol soaking.

Smoke Tests

Both sensors were exposed to intense smoke produced by burning cotton sash cord in a semi-enclosed container for 45 minutes. Thickness of the smoke deposits on the sensor can be approximated from the container walls. Sensors were tested before and after cleaning with isopropyl alcohol and the results are shown in figures 22(a) and 22(b). The sensors were allowed to dry at 10 °C/15% RH for 2 days after cleaning with isopropyl alcohol and were retested for any shift in the calibration. The results are shown in figure 22(a).

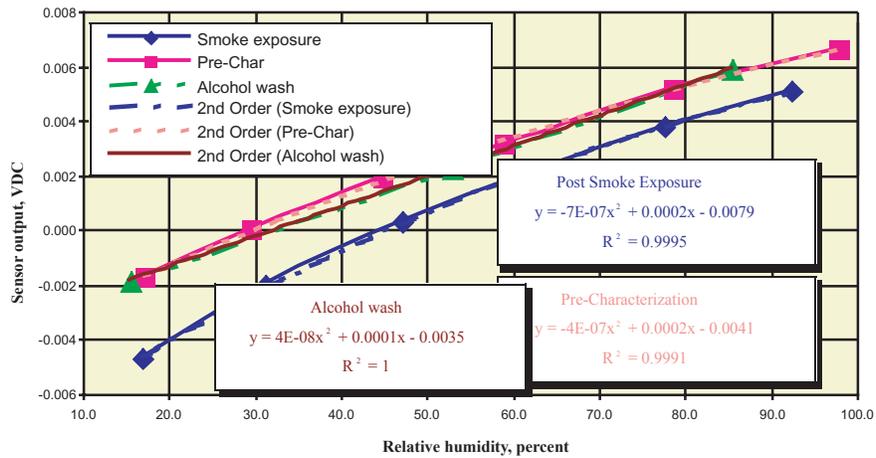


Figure 22(a): Smoke deposits on cantilever-beam-based humidity sensor slow the response of the sensor due to micro-voids that remain trapped with soot.

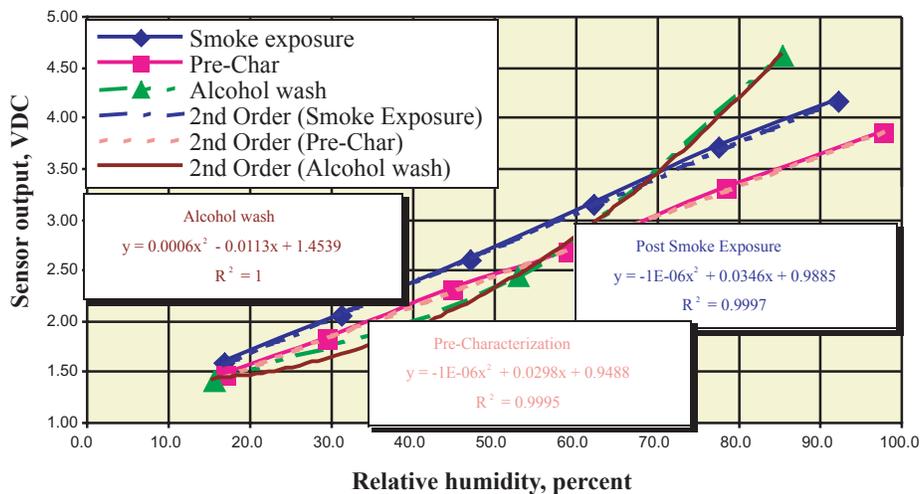


Figure 22(b): Smoke layer on capacitance-type humidity sensor results in trapping of the micro-voids in soot, resulting in lack of response of polymer films.

It appears that the soot fills the micro-voids, resulting in reduction of sensor response. After the surface is washed with isopropyl alcohol, soot is cleared and the sensor retains its original response.

Capacitance type sensor stops responding as a linear humidity sensor when washed with isopropyl alcohol, probably because of the lack of time given to allow isopropyl alcohol to evaporate. The results for long-term testing of the sensors for smoke deposits will be reported later after completion of the experiments.

2-D and 3-D AFM Scans of Smoke-Exposed Polymer Coated Films

The 2-D and 3-D AFM scans for smoke-exposed polymer films coated on the cantilever-beam-based humidity sensor are shown in figures 23(a) and 23(b), respectively.

After exposure to intense smoke produced by burning cotton sash cord, several spots deposited with soot are visible in both the 2-D and 3-D scans. Isopropyl alcohol wash cleans spots and results in the recovery of the cantilever beam sensor response but not that of capacitance-type sensor.

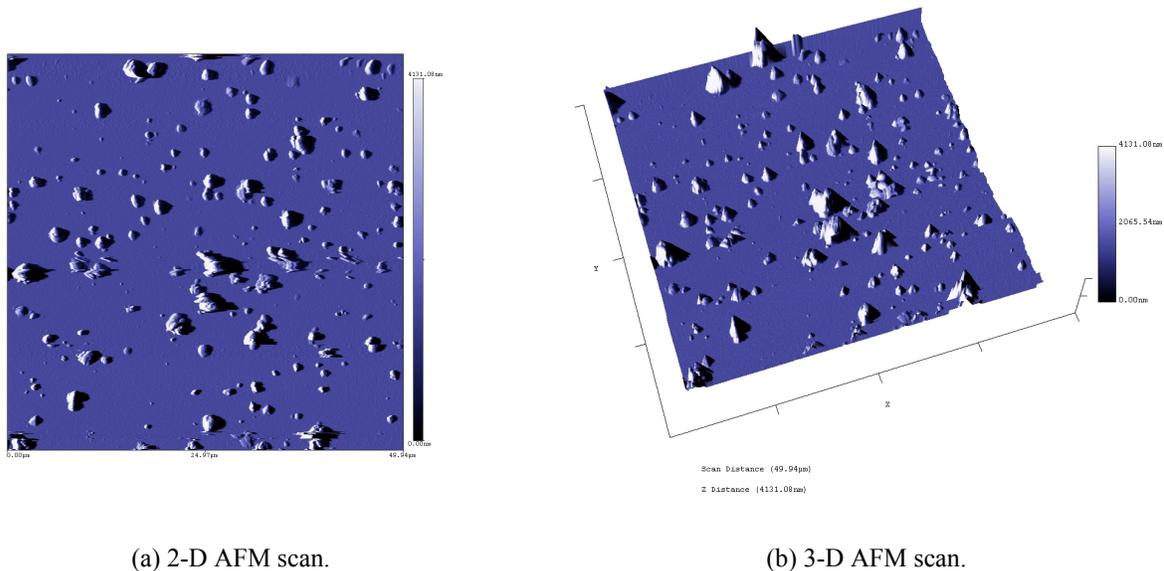


Figure 23. AFM scans of cantilever-beam-based humidity sensors after exposure to smoke.

Concluding Remarks

Two different types of humidity sensors, cantilever beam-based Micro-Electro-Mechanical System (MEMS) sensors and capacitance types with built-in signal conditioning were characterized for their possible use on aircraft. These sensors, coated with industrially proven thermoset polymer thin films were subjected to ambient environmental conditions as well as to common deicing fluids and other aviation-related chemicals. The sensors were also characterized after being soaked in isopropyl alcohol after each experiment with deicing fluids, chemicals, and other related fluid exposures. The sensor surface was analyzed with and without deicing fluids as well as after soaking with different aviation related chemicals. Preliminary results show that polymer coatings can withstand different deicing fluids and chemicals under prevailing flight conditions. Furthermore, these chemicals can be washed away with isopropyl alcohol to enable repeated use of the sensors. Long-term effects of different chemicals and isopropyl alcohol on polymer coatings are still to be determined.

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14. ABSTRACT Under NASA's Aviation Safety Program (AvSP), in addition to wind velocity and temperature, water vapor is considered one key factor in determining aviation weather, which is a substantial contributor to many general aviation (GA) accidents. The conventional and reliable humidity measuring methods such as radiation reflection or absorption have relatively high cost in addition to highly specialized operating and maintenance characteristics. This paper presents characterizations of inexpensive MEMS and capacitance type humidity sensors for their potential use on aircraft. If installed, these sensors are subjected to ambient environmental conditions as well as to different chemicals and deicing fluids used on aircraft. This paper reports the effect of different deicing fluids and chemicals on these inexpensive humidity sensors.					
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