

# FUNDAMENTALS AND ADVANCES IN ULTRA-LOW MOISTURE SENSING: FROM NANOSTRUCTURED INTERFACES TO SMART INDUSTRIAL INFRASTRUCTURE

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## Abstract

In applications such as semiconductor manufacturing, lithium-ion battery manufacturing, gas purification lines, and aerospace systems the precise detection of very low relative humidity (< 10%RH) and low moisture content (< 1000 ppm<sub>v</sub>) is very critical. The physisorption-based Grotthuss ionic conduction mechanisms break down at low moisture content because of the absence of continuous networks of liquid-like water molecules. This extensive review provides the reader with a detailed synthesis of the state-of-the-art fundamental physicochemical mechanisms involved in low-moisture sensing, including chemisorption, dielectric modulation, quantum tunneling and interfacial charge transfer. High performance solid-state trace moisture sensors are comprehensively explored in terms of advanced synthesis, deposition and fabrication routes. This encompasses nanostructured metal oxides, defect-engineered metal-organic frameworks (MOFs), 2D material heterostructures and flexible substrates. Sensitivities, hysteresis limits, response/recovery times and long-term stability under severe drying are among the key performance parameters that are critically evaluated. Lastly, we bring an integrated analytical view on the use of low moisture sensors combined with a calibration model based on artificial intelligence (AI) and automatic edge-computing nodes for the next generation of a smart industrial infrastructure.

## 1. INTRODUCTION

Applications for humidity sensors in environmental control and industrial processes are growing [1] [2]. Industry and research labs make extensive use of humidity sensors based on water-phase protonic ceramic materials. Protons and adsorbed water will condense on the material's surface and conduct in the created aquatic layers. The conductivity of ionic sensing

materials decreases and the dielectric constant rises with increasing humidity [3] [4].

Moisture control is one of the most important measurements in modern, high-tech, industrial settings. Ambient relative humidity (40% to 70% RH) plays an important role in human comfort and commercial HVAC applications, while ultra-low relative humidity (< 10% RH or 1000 ppm<sub>v</sub>)

water concentration) is essential in ensuring catastrophic failures do not occur in advanced manufacturing workflows [4].

In the production of lithium-ion batteries, ambient air needs to be carefully controlled in order to avoid the reaction between water and lithium hexafluorophosphate,  $\text{LiPF}_6$ , to produce

hydrofluoric acid,  $\text{HF}$ , which is toxic and corrosive. Trace water molecule contamination is crippling the wafers yields in semiconductor lithography clean rooms and gas purification pipelines by causing irreversible performance degradation and surface oxidation. Figure 1 shows detection of trace water leak

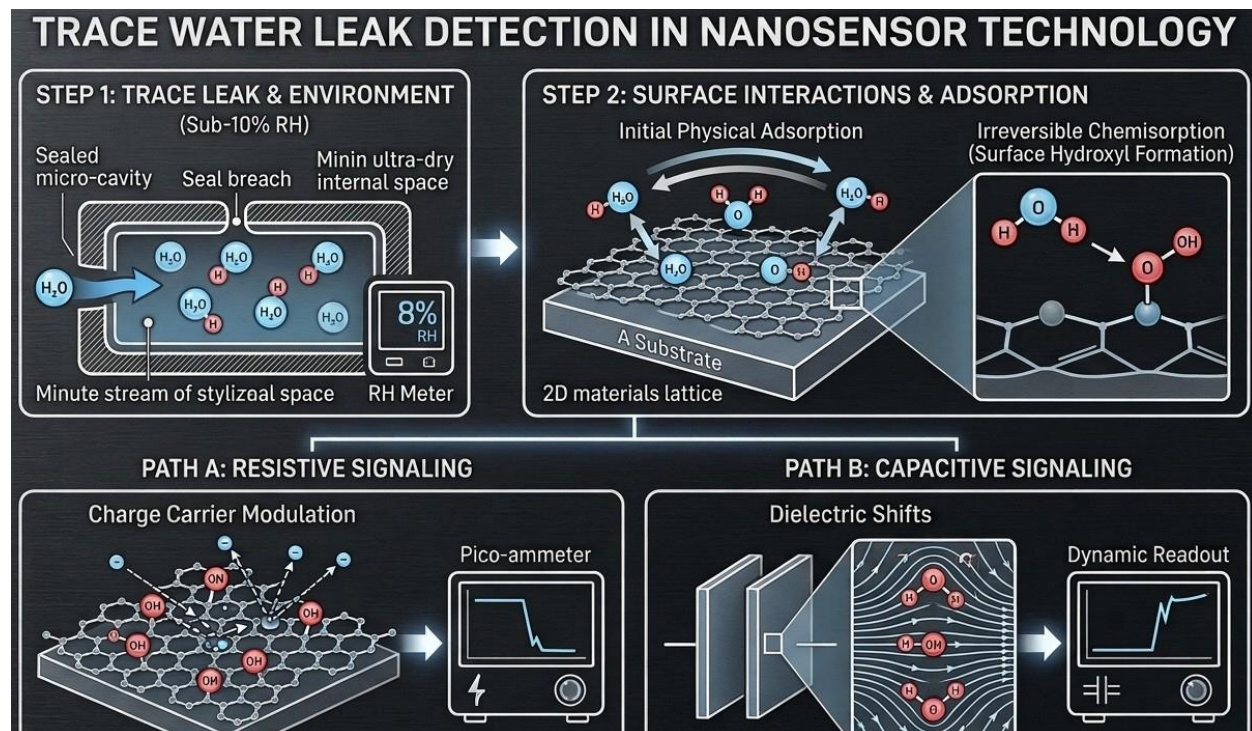


Figure 1: Detection of trace water leak

In the mid- to high-humidity field, traditional humidity sensor measures the humidity through multi-layered physical adsorption (physisorption) of water molecules. This process causes fast ionic transport through proton hopping, the so-called Grotthuss mechanism [5]. But at ultra-dry or trace levels, the continuous network of hydrogen-bonding between water molecules collapses [6-10]. Under these constraints, sensing requires advanced structural interfaces that are both highly chemically reactive and have a high surface area or are quantum mechanical responsive to individual molecules of water [4]. Capacitive humidity sensors [11-15] based on anodic aluminum oxide

(AAO) film are widely used in many applications but here they are not focused on review.

## 2. Sensing Mechanisms and Fundamental Physics at Ultra-Low Humidity

The shift from the bulk properties of the liquid to discrete interactions on the surface is needed for developing ultra-low moisture sensors. In most cases, adsorption of water is restricted to the chemisorbed (Figure 2) or localized physisorbed single-layer adsorption ( $\text{RH} < 10\%$ ) [5]. There are four major mechanisms that dominate within this regime:

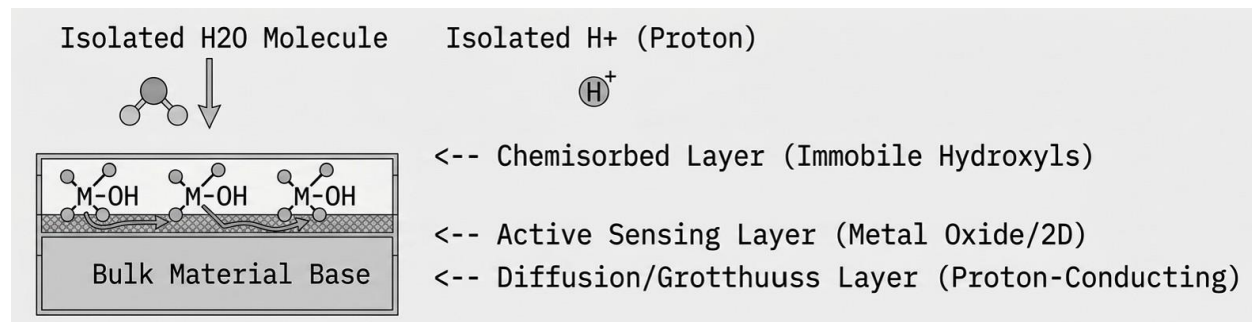
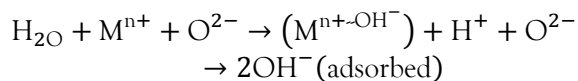


Figure 2: Chemisorbed layer with hydroxyl groups

### 2.1 The chemisorption and dissociative adsorption process

When a completely dry surface of a sensor (such as a metal oxide) is exposed to first exposure of trace moisture, the water molecules initially dissociate with very high energy.



Molecules are broken at active metals ( $\text{M}^{n+}$ ), and at localized oxygen vacancy defects to form stable and immobile surface hydroxyl groups ( $\text{OH}^-$ ). This chemically bonded chemisorbed monolayer is not mobile and proton conduction across the monolayer is severely restricted [5].

Rather, this layer modifies the overall work function of the material and serves as a very polar surface template for the subsequent physical adsorption. With high binding energy, however, these chemisorption bonds can generally be broken only at high activation energies, resulting in significant dry-down hysteresis [4].

### 2.2 Dielectric Modulation

Capacitive trace moisture sensors [16-20] are based on the changes in real and imaginary parts of the permittivity of the sensor material caused by the accumulation of water molecules. The total capacitance  $C$  of a parallel-plate structure or an interdigitated electrode (IDE) array is given by:

$$C = \epsilon_0 \epsilon_r \frac{A}{d}$$

The vacuum permittivity,  $\epsilon_0 = 8.854 \times 10^{-12}$ , the active geometric surface area,  $A$  and the electrode gap,  $d$  are parameters of the system,

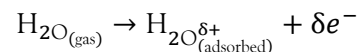
while the relative permittivity of the composite sensing film matrix,  $\epsilon_r$ , is a parameter of the sensing film. Relatively "small" dielectric footprint of clean, dry nanostructured ceramics or polymers ( $\epsilon_r \approx 2$  to 10).

The overall material matrix permittivity is greatly affected by the presence of even trace amounts of water molecules due to the high value of the static relative permittivity of water ( $\epsilon_r \approx 80$  at 20°C). This change is mainly attributed to the polarization of individual, localized water dipoles in an alternating current (AC) electric field at ultra-low RH [4].

The charge carrier modulation and interfacial charge transfer will be described in this section. This section describes the charge carrier modulation and the interfacial charge transfer.

### 2.3 Charge Carrier Modulation and Interfacial Charge Transfer

Water molecules act as direct electron donors or acceptors in semiconducting metal oxides (SMOs) and 2D nanomaterials (2-D materials, such as graphene, MXenes, transition metal dichalcogenides). In an n-type semiconductor, the lone pair electrons of water molecules can be donated to the conduction band of the semiconductor:



The effect of this electron donation is to reduce the electron depletion layer at the surface, and hence to reduce the electrical resistance measurably. Majority holes, recombine with this electron creating additional resistance in p-type materials.

This charge transfer occurs mainly at local areas of active edge configurations, structural step defects and oxygen vacancies and is highly localized at low humidities. This localization gives a high sensitivity but also brings a baseline drift with pinned electronic states [4].

## 2.4 Quantum Tunneling

Conduction in advanced nanomaterial systems, such as functionalized carbon nanotube, graphene oxide sheets, and discontinuous metallic nanoparticle arrays, is very sensitive to the inter-particle and inter-planar nano-gaps. The tunneling current density  $J$  as a function of the physical distance  $s$  between neighboring conducting channels is exponential:

$$J \propto \exp\left(-\frac{4\pi s}{h}\sqrt{2m\Phi}\right)$$

Here,  $h$  is Planck's constant,  $m$  is the mass of the carriers and  $\Phi$  is the potential energy barrier height. The presence of trace moisture creates the nanoscale gap if the polymer/nanocomposite matrix swells, or if the height of the local dielectric barrier ( $\Phi$ ) changes, the probability of tunneling dramatically changes [4]. This is a structural response, which allows for high sensitivity to minute moisture content changes.

## Conclusion

To conclude, the accurate detection of ultra-low moisture (< 10%RH, < 1000 ppm,) levels is critical in many high-tech manufacturing processes such as Semiconductors and Lithium-Ion Battery production plants. At these low concentrations, conventional proton-hopping processes are no longer reliable and high-performance solid-state sensors must rely on the use of advanced nanomaterials (nanostructured metal oxides, 2D heterostructures) that can benefit from localized chemisorption, dielectric modulation, modulation of charge carriers and quantum tunneling. Finally, combining these highly sensitive sensor architectures with AI-enabled calibration models and edge computing nodes will pave the way for future smart industrial automation systems.

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