

Anodic Spark Deposition of Porous α -Al₂O₃ Films and Its Importance in Humidity Sensor Fabrication: A Review and Perspective

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Abstract

Humidity and moisture sensors play important roles in industrial process control, environmental monitoring, aerospace, pharmaceutical manufacturing, energy infrastructure, and semiconductor fabrication applications. The properties of the humidity-sensitive dielectric layer are important to the performance of these sensors. Anodic Spark Deposition (ASD) is a specialized fabrication technique for producing a highly porous, chemically stable α -Al₂O₃ film, which has been shown to be useful for fabricating a high-performance moisture sensor. ASD is a technique that enables direct electrochemical oxidation to form porous ceramic coatings on substrates (graphite, glass, titanium, aluminum, and so on) under spark-discharge conditions. The α -Al₂O₃ films formed have good thermal stability, corrosion resistance, mechanical strength, and controlled pore structure. This article provides a summary of the principles of ASD, pore formation mechanism, process parameters, and the resulting microstructural characteristics. Special focus on the application of porous alumina produced from ASD in capacitive humidity and moisture sensors. The effects of pore-size distribution, surface morphology, adsorption/desorption kinetics, response speed, sensitivity, and long-term stability are considered. Hybrid α -Al₂O₃/SiO₂ dielectric structures have also been reviewed recently.

Keywords: Anodic Spark Deposition, Alpha Alumina, Humidity Sensor, Moisture Sensor, Porous Ceramics,

1. Introduction

A research group led by Professor Dr. Zhi David Chen, who is a Professor in the Department of Electrical and Computer Engineering at the University of Kentucky, has been working on the fabrication of moisture sensors since the 1990s. Anodic Spark Deposition (ASD) is a major step in the fabrication of low moisture sensors [1-3]. Measuring humidity is an important need in modern industry and science. Water vapour monitoring is essential in natural gas processing, semiconductor production, pharmaceutical manufacturing, food processing, fuel cell applications, and environmental monitoring, among other compressed air applications [3-12]. Depending on the sensor's humidity range, humidity sensors can be classified as either absolute or relative. Figure (1) lists applications. Robotics and assistive technology can make use of humidity sensors. When integrating sensors in different locations, these moisture sensors are helpful. Enhancement of Quick Reaction Humidity sensors is crucial for wearable sensing applications and assistive robotics. Natural gas processing, food processing, fuel cell applications, environmental monitoring, compressed air applications, next-generation assistive robotics, wearable assistive technologies, semiconductor manufacturing, pharmaceutical manufacturing, and intelligent sensor systems operating in dynamic, low-humidity environments all depend on accurate, rapid environmental sensing [1-9].



Figure 1: Applications of moisture sensors

In regions with relatively high moisture concentrations, relative humidity sensors are the most commonly utilized sensors, as in figure 2, on right left side there is more humidity where RH sensors for measurement are used, where as for low humidity (right side of figure 2), trace moisture sensors are used. However, industrial applications often require low moisture levels, typically less than 100 ppmv. Monitoring changes in water vapour concentration requires moisture sensors with extremely high sensitivity.



Figure 2: Use of Relative Humidity and Absolute Humidity Sensors.

The sensing performance of humidity sensors is primarily determined by the dielectric material in the sensing layer. Among the numerous advantages of porous metal oxides are their tremendous surface area, hydrophilic properties, chemical stability, and compatibility with microfabrication processes. Aluminum oxide is one of the most popular sensing materials.

The alumina formed by traditional anodizing of aluminum oxide is mostly in the amorphous (or gamma) state, as shown in Figure 3. These materials offer good sensitivity but suffer from drift, slow response, and environmental instability. To overcome these limitations, researchers have explored the use of alpha-phase alumina made using Anodic Spark Deposition (ASD).

ASD is a unique method to fabricate porous α - Al_2O_3 films with high physical and chemical properties. The films have shown great performance as humidity and moisture sensors.

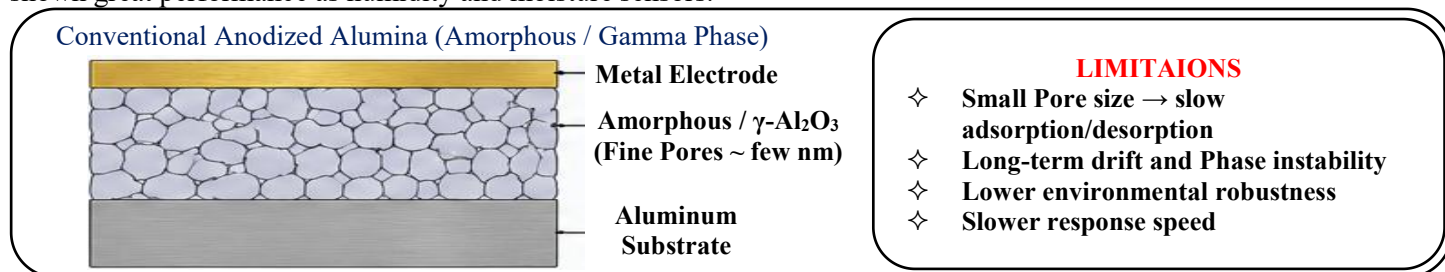


Figure 3: Conventional Anodized Alumina

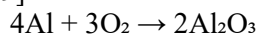
1.1. Research Gap and Motivation

ASD-derived porous α - Al_2O_3 films have several drawbacks for humidity sensing, yet they exhibit excellent properties. Firstly, if the processing parameters are not optimized, non-uniform pore distribution or local defects may be generated by the micro-arc discharge process. Second, the electrochemical reaction of the electrolyte, the discharge behavior, and the pore morphology are not yet fully predictable [1-3]. Third, producing large-area nanoscale pore architectures reproducibly in an industrial manufacturing process remains difficult. Last, the hybrid α - $\text{Al}_2\text{O}_3/\text{SiO}_2$ structures provide better insulation and stability, but add more processing steps to the fabrication process, making it more expensive and complicated. Future studies should thus be directed towards reproducibility of the process, in situ diagnostics of the discharge, predictive modeling, and scalable manufacturing methods [5,9].

1.2. Anodic Spark Deposition Fundamentals

Anodic Spark Deposition is an electrochemical surface modification method for applying a ceramic coating to a metallic substrate. Unlike conventional anodization methods, the anodization process of ASD involves the micro-arc discharge or spark phenomena at the metal-electrolyte interface. The aluminum substrate and a counter electrode in an electrolyte are subjected to a high voltage during the procedure. The breakdown occurs in the expanding oxide film when the voltage exceeds a critical threshold. Localized plasma channels with extremely high temperatures and pressures are produced as a result [13–17]. Aluminum is transformed into dense ceramic structures, and a porous microstructure is simultaneously created during the plasma-assisted oxidation process [1–9].

The overall anodic oxidation reaction is:



Under appropriate process conditions the generated oxide film is mainly composed of alpha-alumina phase (Figure 4).

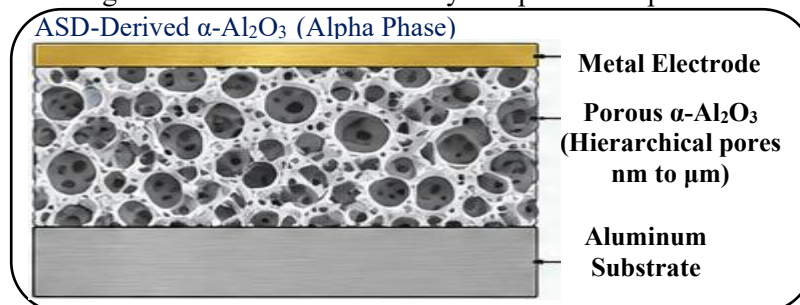


Figure 4: Alpha-alumina phase

1.3. The formation mechanism of the α -Al₂O₃ during ASD

ASD differs substantially from conventional anodization processes in the formation of α -Al₂O₃ [1-6]. In normal anodization, the aluminum oxide is usually amorphous or gamma phase. Typically, conversion to alpha alumina involves thermal treatment above 1000°C. Localized plasma discharges, however, in ASD can produce brief temperatures well into the thousands of degrees centigrade. The extremely high temperatures enable direct crystallization of alpha alumina during deposition. Steps in the ASD process are shown in Figure 5 [17-23].

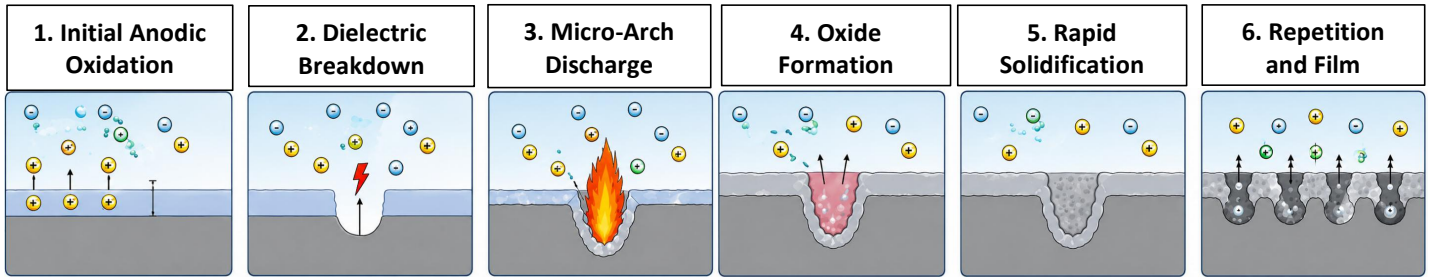


Figure 5: Schematic illustration of Anodic Spark Deposition showing the sequence of micro-arch discharge events at the anode, and the resultant hierarchical alpha-alumina coating [1-6].

The mechanism of formation is:

1. Oxidation of aluminum in an electrolytic cell.
2. Failure of the oxide film because of its dielectric breakdown.
3. Generation of plasma at discharge channels.
4. Local melting of oxide material.
5. Quick freezing and crystallization.
6. Formation of porous ceramic structures.

These events repeat continuously and lead to the formation of a thick, porous α -Al₂O₃ layer.

1.4. ASD Electrolyte Systems and Processing Conditions

It is the choice of researchers, experts, and scientists to select a particular substrate (graphite, glass, titanium, aluminum, to name a few) based on its applications and use in a particular industry. Electrolyte composition and processing parameters play a crucial role for the quality of the ASD coatings. The most common electrolyte system used is a mixture of salts.

The typical preparation procedure is as follows:

- 450 – 600 g of any salt
- 340 – 580 g of different types of salt
- 600 – 1000 mL reaction vessel

The electrolyte temperature is slowly increased from low to 360°C.

The heating process has several functions: removal of moisture, removal of gas bubbles, stabilization of the electrolytic composition, and improvement of ionic conductivity. Once the substrate is stabilized, a set of anodic sparking conditions is applied to it. Voltage, Current density, Deposition time, Electrolyte temperature, Electrode spacing, and Electrolyte composition are important parameters of ASD. These parameters can be changed slightly and greatly affect the resulting surface morphology [24-30].

1.5. The microstructure and surface morphology of ASD films.

One of the most crucial characteristics of ASD coatings is their porousness. Studies using scanning electron microscopy (SEM) have revealed a hierarchical pore structure with large, medium, and microscopic pores. Large pores serve as transport routes and range in diameter from 1 to 10 μ m. These medium pores, with diameters of 100 nm to 1 μ m, facilitate moisture transport. These tiny holes, with diameters of 40–200 nm, create active adsorption sites. Figure 6 [4-6, 31-44] lists the many types of pores.

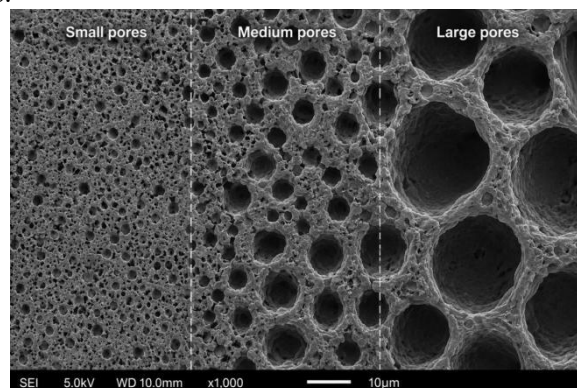


Figure 6: Categories of pores created after Anodic Spark Deposition

These pores are distributed in a way that significantly affects the sensor's performance. The films produced on ASD are frequently on the order of thousands of nanometers in size, with nanoscales embedded in larger ones. This hierarchical structure greatly improves the effective surface area for water adsorption. Adsorption (reason for high humidity) and desorption (reason for low humidity)(Fig.7) [3-12, 31-39].

1.6. Importance of the ASD in the fabrication of Humidity Sensors.

Humidity sensors rely on adsorption and desorption at the surface of the sensing material, which can significantly impact sensor performance. For optimum sensing, a sensors must have sensing material requirements. The ideal sensing material requirements are high porosity, large surface area, chemical stability, mechanical robustness, and hydrophilic properties that are illustrated in figure 7. The ideal sensing material should have high porosity, large surface area, chemical stability, mechanical robustness, and hydrophilic properties.

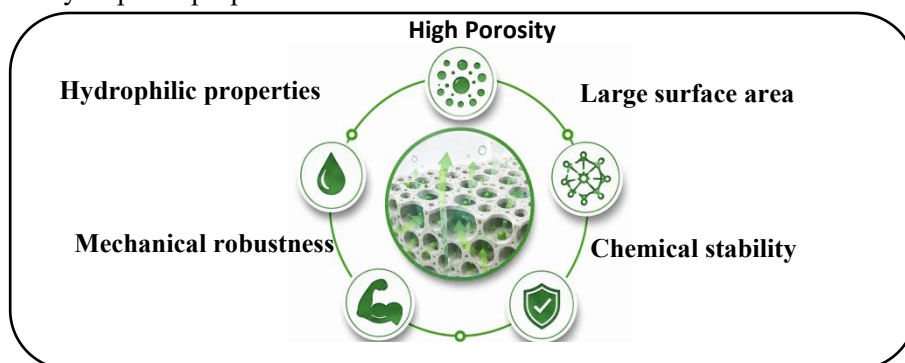


Figure 7: Ideal Sensing Material Requirements

α -Al₂O₃ produced by ASD meets all these requirements. The porous structure facilitates the easy diffusion of water molecules into the sensing layer and provides numerous adsorption sites. Therefore, humidity sensors based on ASDs exhibit high sensitivity, fast response and recovery, excellent repeatability, and long operational lifetime (Fig. 8). These properties make ASDs highly desirable for industrial moisture-sensing applications. ASD-derived porous α -Al₂O₃ has a range of RH-sensing and low-moisture applications, with Fast response reported in hierarchical porous structures. Process optimization and pore control remain challenging [1-13] (Table 1).

Table 1: Comparative Performance of Humidity Sensors

Sensor material	Typical RH range	Response/recovery	Key limitation
Polymer-based [2-3]	10–95%	1–30 s	Temperature and aging sensitivity
Conventional anodized alumina [1, 3, 5]	0–100%	seconds to minutes	Drift and environmental instability
Metal-oxide ceramics (general) [1-4]	0–100%	seconds to minutes	Often require elevated temperature or complex processing
ASD-derived porous α -Al ₂ O ₃ [5-13]	RH sensing and low-moisture applications	Fast response reported in porous hierarchical structures	Process optimization and pore control remain challenging

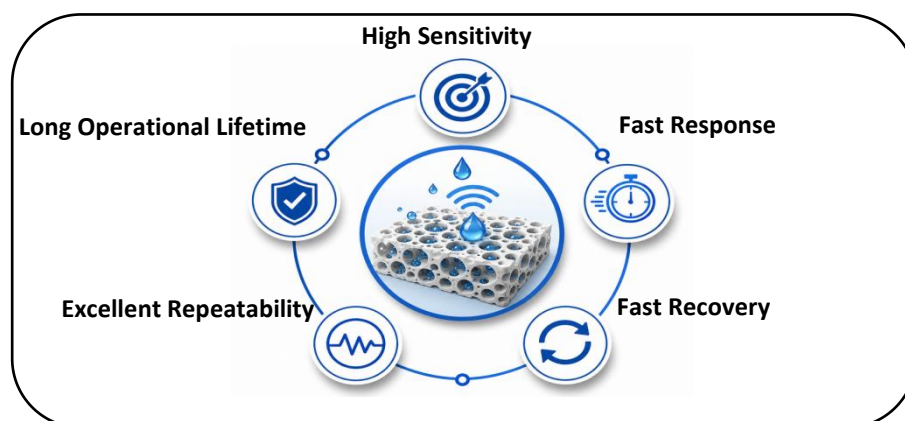


Figure 8: Advantages in the Humidity Sensor's performance

In the adsorption and desorption mechanisms of low moisture sensors, the aforementioned characteristics, high sensitivity, fast response and recovery, excellent repeatability, and long operational lifetime play a crucial role. For example, during adsorption, the response rate depends on how quickly hydroxyl groups attach to the sensing material's surface. Similarly, during desorption, the recovery rate depends on how rapidly these hydroxyl groups detach from the sensing material's surface (Fig. 9).

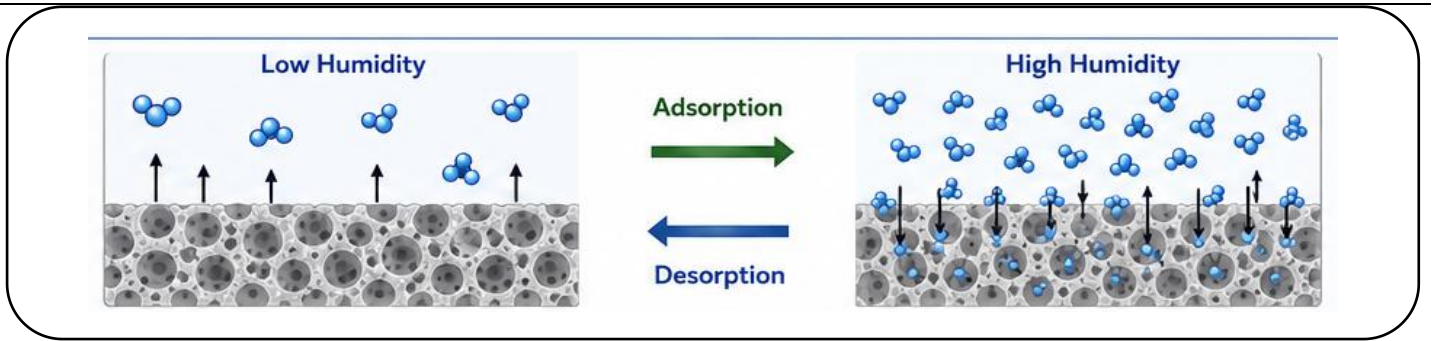


Figure 9: Illustration of Adsorption (High Humidity) and Desorption (Low Humidity)

1.7. The surface morphology and its effect on the performance of the sensors

The surface morphology directly impacts the sensor's response behavior. When humidity shifts, water molecules are either adsorbed onto or desorbed from the pore surfaces. Pore geometry has a significant impact on this process. Larger active surface area, greater adsorption capacity, quicker moisture exchange, and enhanced sensitivity are all characteristics of sensors with more nanoscale pores. The fastest response times are found in sensors with the greatest number of holes smaller than 100 nm, according to an experimental study. This enhancement is especially noticeable in ultra-low-humidity conditions, when adsorption kinetics govern the sensor's behavior [31–44].

1.8. Hybrid α - $\text{Al}_2\text{O}_3/\text{SiO}_2$ Structures

ASD films have very good porosity; however, when metal electrodes are deposited directly on the porous alumina, the continuous pore pathways sometimes make it difficult to avoid an electrical short circuit. For this purpose, an atomic layer deposition (ALD) process of SiO_2 is used to address this problem, while Figure 10 shows the hybrid structure [5].

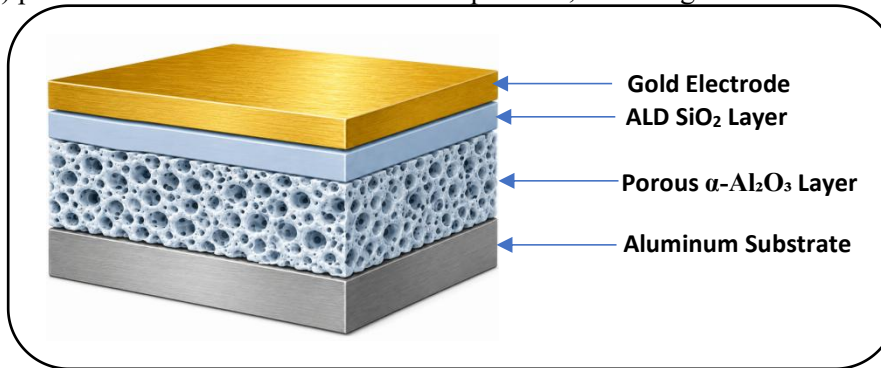


Figure 10: Schematic illustration of a hybrid humidity sensor structure

The ALD SiO_2 layer serves several roles, including electrical insulation, surface passivation, enhanced stability, and controlled pore accessibility. This new hybrid dielectric architecture has the best of both worlds, with high sensitivity to the sensor [45–48].

1.9. The advantages of ASD over the conventional techniques

ASD offers several advantages over conventional anodization and ceramic fabrication techniques. Why ASD-derived α - Al_2O_3 is distinct

1.9.1. Direct α -phase formation

Unlike conventional anodization, ASD can produce α - Al_2O_3 during deposition through localized plasma-assisted crystallization, reducing the need for high-temperature post-annealing. [1,3,4]

1.9.2. Hierarchical porosity

ASD naturally generates interconnected macro-, meso-, and nanopores that enhance water-vapor transport and adsorption [4,24–30]. Complex lithographic processes are removed naturally with the formation of porous structures.

1.9.3. Strong substrate adhesion

The oxide grows from the substrate itself, providing better adhesion than many deposited ceramic coatings. [4,25,26]

1.9.4. Chemical and thermal stability

α - Al_2O_3 offers superior stability in harsh environments compared with many polymeric sensing materials [1,3,45]. The films' resistance to acids, alkalis, and corrosive environments is outstanding.

1.9.5. Long-Term Reliability

Alpha alumina is more structurally stable than the amorphous and gamma-phase alumina. Again, it must be noted that it depends on researchers, experts, and scientists to choose a substrate appropriate for their applications and use in a particular industry; substrates can be graphite, glass, titanium, or aluminum, to name a few.

1.9.6. Scalability

Several samples can be processed concurrently in a common electrolyte system.

1.10. Industrial Applications

- ✓ ASD sensors for humidity and moisture work well for a wide variety of applications.
- ✓ Compressed Air Monitoring
- ✓ Moisture detection in an industrial pneumatic system
- ✓ Natural Gas Processing
- ✓ Tracking water vapor and dew point
- ✓ Semiconductor Manufacturing
- ✓ Control of very low humidity in fabrication plants
- ✓ Pharmaceutical Production
- ✓ Environmental monitoring in cleanrooms
- ✓ Transformer and Lubrication Oils
- ✓ Water contamination detection
- ✓ Aerospace Systems
- ✓ Humidity measurement in critical areas
- ✓ Hydrogen and Fuel Cell Systems
- ✓ Optimization of moisture management

1.11. Critical Assessment of Advantages and Limitations

Although porous alumina sensors have been studied for several decades, most review articles focus on conventional anodized alumina, polymer-based sensors, or general humidity-sensing materials. Anodic Spark Deposition (ASD) has been shown to be a unique process for directly growing porous α -Al₂O₃ coatings with a hierarchical pore structure and high chemical stability, yet it has received comparatively little attention in reviews. Moreover, the correlation among the processing conditions of ASD, pore morphology, adsorption/desorption kinetics, and sensor performance has not yet been systematically discussed. This review fills the void by critically discussing the application of porous α -Al₂O₃ films derived from ASD for humidity and moisture sensing, pore-structure engineering, response mechanisms, hybrid α -Al₂O₃/SiO₂ architectures, and possible future directions for intelligent and IoT-enabled sensing systems.

4.0 Conclusion

One of the most promising methods for creating porous α -Al₂O₃ films for sophisticated humidity and moisture sensors is anodic spark deposition. On substrates (graphite, glass, titanium, and aluminum), the technique enables the direct creation of chemically stable, mechanically strong, and extremely porous ceramic structures. The unique hierarchical pore structure created by ASD confers high sensitivity and reaction rate due to its large active surface area for moisture absorption and desorption. When combined with ALD-deposited SiO₂, hybrid dielectric structures with superior electrical insulation and long-term stability are produced, which further enhances sensor performance. According to experimental studies, the distribution and density of nanoscale pores play a crucial role in determining sensor response properties. Humidity and moisture sensors will continue to expand in functionality and capabilities through advancements in process control, nanomaterial integration, and intelligent sensing systems. Humidity sensors can be utilized in assistive and robotics technologies.

Future Research Directions

Process Optimization: ASD parameters can be correlated with the pore morphology using machine learning techniques.

Nanostructure Engineering: Accurate control of nanoscale pore formation can further enhance the response rate.

Advanced Hybrid Materials: New composites (α -Al₂O₃, graphene, metal-organic frameworks, and functional nanomaterials) could be used to further increase the sensitivity. **Artificial Intelligence Integration:** AI-driven signal processing can enhance calibration and drift compensation. **IoT Connectivity:** Seamless integration with wireless sensor networks will allow for real-time monitoring and predictive maintenance. **Sustainable Manufacturing:** Starting from a study of the compatibility of these materials, the development of environmentally friendly ASD electrolytes, and energy-efficient processing methods.

Acknowledgment

The authors would like to express their gratitude to the University of Kentucky, the Benazir Bhutto Shaheed University of Technology and Skill Development, Khairpur Mirs, and the co-authors' institutions.

Author's Contribution: Ronak Ali (First Author and Corresponding Author): Dr. Ronak Ali had the idea for the review, performed the literature search and wrote the first draft and then subsequent drafts, Shakil Ahmed Jiskani (technical guidance), Safdar Ali Abro (investigated review articles that helped in initial write up), Abdul Shakoor Shaikh (helped with resources to complete study), Kaneez Fatima: (validation of writing the right content under each heading), Ghulam E Mustafa Abro (review of literature and its quality), Madad Ali Shah (Professor Dr. Madad Ali Shah did overall supervision)

Conflict of Interest: The authors have no conflict of interest.

Funding Resource: This work was not funded by any external grants

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Received: April 18th 2026

Accepted: June 6th 2026