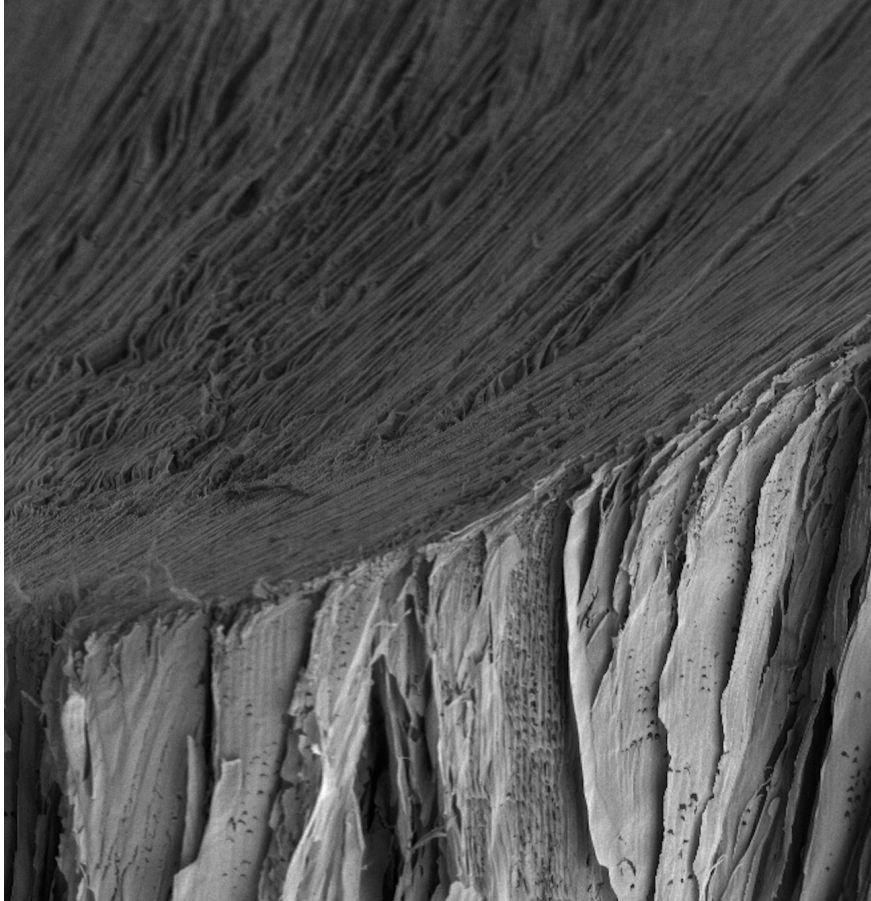




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Development of a high-performance sensor based on advanced 2D materials

Master's thesis in Materials Chemistry

Sijin Sun

DEPARTMENT OF MECHANICAL ENGINEERING

CHALMERS UNIVERSITY OF TECHNOLOGY

Gothenburg, Sweden 2026

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MASTER'S THESIS 2026

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Department of Mechanical Engineering
Division of Materials and Manufacture
CHALMERS UNIVERSITY OF TECHNOLOGY
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Supervisors: Jinhua Sun, Department of Industrial and Materials Science
& Guangliang Tian, Department of Industrial and Materials Science
Examiner: Jinhua Sun, Department of Industrial and Materials Science

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Department of Mechanical Engineering
Division of Materials and Manufacture
Chalmers University of Technology
SE-412 96 Gothenburg
Telephone +46 727724713

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Sijin Sun
Department of Mechanical Engineering
Chalmers University of Technology

Abstract

Flexible strain, pressure, and conductivity are crucial properties for real-time detection or human-body interaction sensors. However, traditional three-dimensional porous materials, such as MXene aerogel, frequently show weakness due to their weak inter-sheet connections, and randomized porous architectures lead to brittle fracture, irreversible deformation, and severe electrical signal drift under long-term cyclic loading. Further, traditional hydrophilic polymer binders such as poly (vinyl alcohol) fail to address these issues due to their high moisture sensitivity, which compromises long-term environmental and structural integrity.

To address these limitations, this thesis introduces a dual strategy that combines advanced composite chemistry with microstructural control. First, a polyamic acid triethylamine salt (PAA-TEA) solution is combined with a highly conductive MXene suspension to form a high-performance polyimide (PI) hybrid network that serves as an extrinsic structural reinforcement phase. Second, intrinsic anisotropic microstructural alignment is achieved during freeze casting by applying an alternating current (AC) electric field. This process leverages Maxwell-Wagner interfacial polarization to induce an electrical dipole moment on the MXene nanosheets, generating a rotational dielectrophoretic torque that systematically aligns them parallel to the electric field lines. Finally, subsequent thermal imidization converts the matrix into a robust aromatic polyimide framework, permanently locking the ordered pathways.

Mechanical characterization demonstrated that the 65%:35% PAA-TEA: MXene ratio provides superior performance over other investigated compositions. The application of an AC electric field substantially reinforced the material's network strength, positioning its compressive strain-stress loops consistently higher along the stress axis compared to untreated aerogel. This structural ordering allows the aligned aerogel to tolerate significantly higher strain displacements and stresses without mechanical collapse, while simultaneously optimizing contact resistance and electron-transport continuity. Ultimately, this scalable integration of high-strength polyimide chemistry and dynamic electrical alignment offers a robust framework for designing highly resilient, durable, and multifunctional flexible sensing materials that can be used in a sensor.

Keywords: aerogel, MXene, polyimide, electric field, conductivity, mechanical strength, sensor.

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Sijin Sun, Gothenburg, June 2026

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1

Introduction

1.1 Background

Aerogels are a unique class of three-dimensional porous materials characterized by ultralow density, high specific surface area, and interconnected nanoscale networks. Its name stems from the Greek word *aeros*, which means "air". Aerogels are a solid that is composed of meso- and macropores with diameters up to a few hundred nanometers and porosity of more than 90%, in which the dispersed phase is a gas, resulting in great lightness and highly compressible mechanical behavior. Owing to their tunable mechanical and electrical properties, aerogels have been widely studied for applications in thermal insulation, energy storage, and flexible detection sensors.

Alongside rapid advancements in wearable electronics and intelligent healthcare systems, flexible strain and pressure sensors have become critical components for real-time physiological monitoring, motion tracking, and human-machine interaction. Ideal sensing materials must simultaneously satisfy several demanding criteria, including low density, high mechanical resilience, stable electrical conductivity under cyclic deformation, and structural durability over long-term operation. Achieving these combined properties within a single aerogel system remains a significant materials engineering challenge.

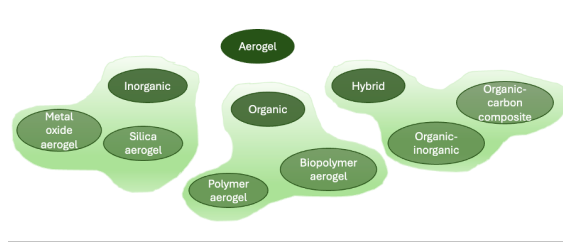


Figure 1.1: Classification of aerogels based on precursor compression

Currently, aerogels fall into three main categories, as shown in Figure 1.1: inorganic, organic, and hybrid aerogels, each containing several subtypes. Given their earlier development, inorganic aerogels were the first widely studied aerogels. The field began with Samuel Stephens Kistler's demonstration in 1931 that a gel could be dried without collapse to form an aerogel, and for decades it was dominated by classical sol-gel oxide aerogels, especially silica, due to their high porosity and large surface area [1]. However, followed by the disadvantage of inorganic aerogels, which have low mechanical strength and a hydrophilic nature, making them unstable in

processing and handling, has activated researchers to discover more.

Organic aerogels emerged as an extension of sol-gel chemistry to polymeric systems, particularly with the development of phenolic networks in the late 20th century. To meet market demand, organic aerogel, first described by Pekala et al. in 1989, is used for targeted applications due to its lower density, greater thermal insulation, and higher mechanical strength than inorganic silica aerogel [2]. A major shift came in 2004, when Brock and his co-workers showed that pre-formed colloidal nanoparticles could be controllably destabilized and assembled into gels, providing a source for nanoparticle-based aerogels beyond classical sol-gel chemistry. This idea was then extended to metal chalcogenides, oxides, carbon nanostructures, and hybrid systems, making inorganic aerogels a broad materials platform with varied tunable properties. [3] Hybrid aerogels behave as a bridge between inorganic and organic systems, combining the advantages of both by integrating organic polymers with an inorganic framework. For example, in order to improve interfacial bonding and structural stability, polymeric binders such as poly (vinyl alcohol) (PVA) are commonly incorporated into MXene systems. MXene, two-dimensional transition metal carbides, and nitrides have attracted considerable attention due to their metallic-level conductivity, hydrophilic surfaces, and rich surface functional groups (-OH, -O, -F). [4] PVA provides processability and hydrogen-bonding interactions with MXene surfaces, facilitating composite formation. Crosslinking strategies and nanocomposites have been shown to enhance compactness and conductivity compared to pristine polymer aerogels. Thus, researchers achieved aerogels with enhanced mechanical strength, functional versatility, and improved processability. More recently, hybrid aerogels have evolved toward the nanoscale region, especially organic-inorganic co-assembly. And this type of aerogel is the main target of this project.

According to the previous research, organic-inorganic aerogel can be divided into two subtypes depending on the connectivity between the organic and inorganic components: subtype one is prepared by mixing two separate organic and inorganic components through the sol-gel process, resulting in the formation of weak physical bonds, such as van der Waals, hydrogen bonds, or electrostatic bonds, and subtype two involves strong chemical interactions via covalent or non-covalent chemical bonds. Thus, in this system, the component undergoes intercalation, insertion, impregnation, trapping, encapsulation, or templating at the nanoscale [5], [6].

Further research on organic-inorganic aerogels is shown below: Carbon nanotubes exhibit exceptional mechanical, thermal, and electrical properties, but CNT-based structures are primarily governed by van der Waals forces, resulting in low surface area and low elasticity of the individual tubes. Beyond that, adding a binder can be used to structurally reinforce low-density CNT assemblies, but it tends to decrease electrical conductivity. So, according to this phenomenon, Marcus (2009) devised a way to produce ultralow-density foam by using carbon nanoparticles to crosslink randomly oriented bundles of single-walled CNTs, resulting in a high mechanical stiffness, large elastic strains, and high electrical conductivity [7].

Another type of aerogel is graphene oxide (GO)-based graphene aerogels, which are three-dimensional porous materials typically derived from GO via methods such as chemical reduction or chemical vapor deposition (CVD). These materials are designed for high-performance energy storage applications due to their large surface area, good electrical conductivity, and hierarchical pore structure, which enable efficient electron and ion transport. As a result, they are widely considered for use in supercapacitors, batteries, and solar cells [8].

However, the oxidation-reduction processes commonly employed in their fabrication tend to introduce defects and disorder within the graphene sheets, leading to a loss of mechanical strength. Mao et al emphasized that strategies such as cross-linking, composite reinforcement, and the design of controlled three-dimensional architectures are effective in enhancing the mechanical robustness of graphene aerogels while maintaining their functional properties [9].

Besides, Woo (2020) reported that using boric acid to produce crosslinked MXene/PVA nanocomposites yields a more compact morphology under SEM, indicating that boric acid can enhance the interfacial energy between MXene and PVA [10]. Tan et al. (2021) further showed that PVA-MXene composites can significantly improve the aerogel's conductivity compared to pure PVA, owing to hydrogen bonding between MXene and PVA and to defects introduced by MXene [11].

Aside from chemical additives, which can modify the aerogel's intrinsic properties, the mold design plays a critical role in directional crystal growth during freezing. As illustrated in Figures 1.2 and 1.3, four typical freezing configurations have been employed in the lab currently. In all cases, the orange region represents copper (high thermal conductivity), while the gray region represents PTFE (thermal insulation)

(a) In unidirectional freezing, solidification initiates at the copper base and progresses upward, generating a single, well-defined temperature gradient. This results in anisotropic, lamellar structures aligned parallel to the freezing direction, with pore channels oriented perpendicular to the copper bottom. (b) In double-sided freezing, the temperature is controlled at both top and bottom copper interfaces, leading to two opposing freezing fronts that advance toward the center. Consequently, the structure consists of two sets of aligned lamellae growing inward from both ends. (c) Bidirectional freezing maintains a similar overall solidification direction to unidirectional freezing. However, the lateral temperature gradient is more uniformly distributed. This produces a highly ordered and uniformly aligned lamellar structure across the transverse section, minimizing defects and enhancing structural continuity. Such alignment is beneficial for improving mechanical toughness and electrical conductivity along the preferred direction. (d) In magnetic freezing, an external magnetic field is applied by placing magnets on both sides of the mold. For magnetically responsive systems, this field induces the alignment of particles or structural units during solidification, resulting in a structure that exhibits magnetically guided orientation, often appearing as wavy or periodically aligned channels following the magnetic field lines, enabling tailored anisotropy in the material. Yao et al. (2022)

reported that adding Fe_3O_4 enables magnetic-field-assisted alignment of MXene nanosheets via directional freezing, producing an aerogel with a strongly anisotropic structure and decent electromagnetic performance [12], [13].

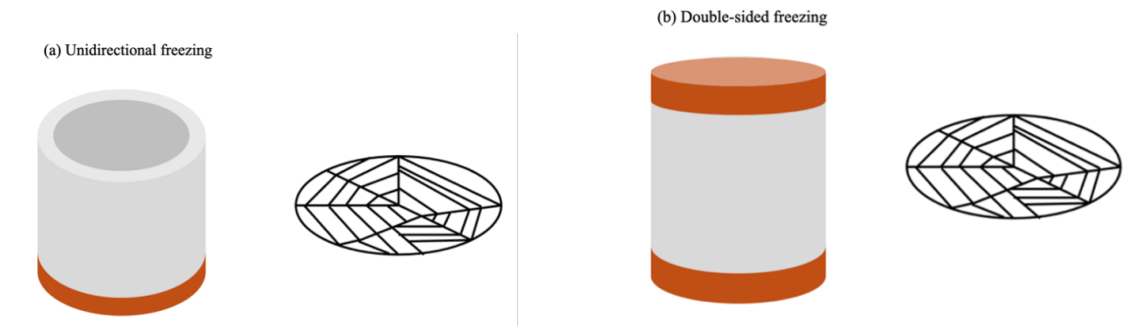


Figure 1.2: Freezing configurations

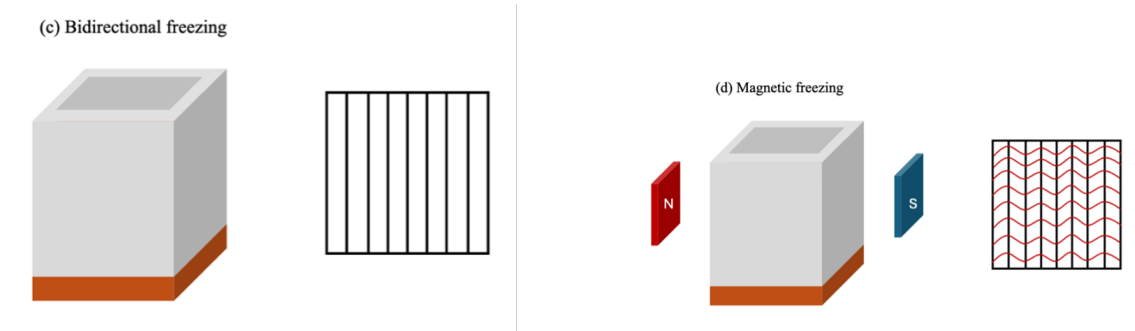


Figure 1.3: Freezing configurations

1.2 Limitations

Despite these advancements, several fundamental challenges remain unresolved. First, the mechanical durability of MXene-based aerogel remains limited. The random orientation of nanosheets and heterogeneous pore structures (not magnetic response material) generated during conventional freeze-drying often leads to brittle fracture, irreversible deformation, or structural collapse under cyclic compression. Stress transfer between nanosheets is inefficient due to weak inter-sheet connections, resulting in gradual degradation of sensing performance [14]. Second, electrical stability under repeated deformation remains problematic: electrical conductivity in MXene aerogels strongly depends on continuous sheet-to-sheet electron transport pathways. Structural separation, nanosheet slippage, restacking, or microcrack formation during cyclic loading can disrupt conductive networks, causing signal drift and reduced sensing reliability [15]. Third, while polymer reinforcement improves flexibility, commonly used binders such as PVA exhibit high hydrophilicity and moisture sensitivity due to abundant hydroxyl groups. This compromises long-term mechanical integrity and environmental stability.

Although previous studies have independently explored polymer reinforcement and related field-induced alignment, Lee et al. (2022) reported that electric fields can promote the alignment of MXene nanosheets in aqueous solution under high-frequency conditions by tuning the applied field conditions [16]. A systematic integration of PI-based structural reinforcement with electric-field-induced MXene alignment in freeze-dried aerogels has not been comprehensively investigated. The synergistic effects of composite chemistry and microstructural ordering on simultaneous mechanical and electrical stability remain insufficiently understood.

1.3 Purpose

This thesis hypothesizes that combining a mechanically robust network, a high-performance polyimide, and electric-field-induced anisotropic alignment of MXene nanosheets will synergistically enhance the mechanical resilience, fracture toughness, and electrical stability of freeze-dried MXene aerogels during cyclic deformation.

To validate this hypothesis, two complementary strategies are proposed which are Extrinsic structural reinforcement via polyimide hybrid network and Intrinsic performance enhancement via AC electric-field-induced alignment:

1. Extrinsic structural reinforcement via polyimide hybrid network. Instead of relying on conventional hydrophilic polymer binders such as Poly (vinyl alcohol), in this study, polyimide (PI) will be incorporated as a high-performance polymer reinforcement phase. Polyimide is well known for its exceptional mechanical strength, thermal stability, chemical resistance, and toughness. Unlike a hydrophilic polymer, polyimide exhibits low moisture sensitivity and strong intermolecular interactions, making it particularly suitable for humid or physiological environments. The incorporation of polyimide into the MXene composite system is expected to enhance fracture toughness through energy dissipation mechanisms, improve compressive resilience and elastic recovery, and strengthen interfacial adhesion between inorganic and conductive components. The resulting organic-inorganic hybrid network is designed by combining MXene with the toughness of polyimide, forming a mechanically robust, flexible, conductive aerogel.

2. Intrinsic performance enhancement via AC electric-field-induced alignment. The second strategy focuses on microstructural control during freeze-drying. An alternating current (AC) electric field will be applied to induce dipole polarization, rotation, and end-to-end chaining of MXene nanosheets in suspension before solidification. This field-assisted assembly is expected to generate an anisotropic, quasi-ordered architecture characterized by directionally aligned conductive pathways, stabilized electron transport, and reduced inter-sheet slippage. Such anisotropic alignment is anticipated to significantly improve both mechanical robustness and conductivity retention under cyclic compression [17]. Therefore, to enable electric-field-induced alignment during freeze casting, a custom-designed mold with conductive copper electrodes and electrically insulating PTFE walls was developed to provide con-

trolled field distribution and directional freezing conditions.

By integrating composite chemistry optimization with controlled microstructural alignment, this thesis seeks to overcome the longstanding trade-off between mechanical resilience and electrical conductivity in MXene-based aerogels. The outcome of this work may provide a scalable strategy for designing multifunctional, durable, and high-performance sensing materials.

2

Theory

2.1 Electric field alignment

2.1.1 MAXwell-Wagner polarization

Maxwell-Wagner polarization is a type of dielectric polarization that occurs at heterogeneous interfaces between phases with different conductivities and dielectric constants when an electric field is applied. Mobile charge carriers migrate into the high-conductivity phase and accumulate at the phase boundary where a less conductive phase hinders transport. This interfacial charge accumulation produces localized dipoles and generates a frequency-dependent dielectric response [18]. The characteristic timescale associated with this interfacial charge accumulation is described by the Maxwell-Wagner relaxation time:

$$\tau_{MW} \approx \frac{\varepsilon_{eff}}{\sigma_{eff}} \quad (2.1)$$

Here, ε_{eff} and σ_{eff} are the effective permittivity and conductivity that depend on the phase properties and microstructure. A useful microscopic picture is that each interface behaves like a tiny capacitor that charges/discharges at a rate set by τ_{MW} . (The permittivity ε is related to the relative permittivity (dielectric constant ε_r) by $\varepsilon = \varepsilon_r \varepsilon_0$)

For a two-phase system, the interface consists of Phase 1, characterized by high conductivity and dielectric constants, and Phase 2, characterized by low conductivity and dielectric constants. Thereafter, under an alternating current field, free charges move quickly along the high-conductivity phase to follow the applied AC field, meanwhile, it acts as a source of mobile charges. In contrast, in the low-conductivity phase, the charges move slowly, which cannot catch up with the AC field totally, thus acting as a barrier. This mismatch leads to charge accumulation at the interface due to different relaxation times [19].

2.1.2 Dipole-field interaction

In the concentrated MXene suspension, alignment is governed by MW interfacial polarization caused by the conductivity mismatch between MXene nanosheets and water molecules (the surrounding solvent). Under an applied AC field, charges accumulating at the MXene-water interface induce dipole moments that generate rotational torque, rotating the sheets into alignment.

$$\tau = pE \sin \theta \quad (2.2)$$

where τ denotes the torque acting on the dipole, p is the electric dipole moment, E is the applied electric field strength, and θ is the angle between the dipole moment vector and the electric field vector.

According to this mechanism, charges accumulate at opposite sides of the MXene nanosheets, leading to one side becoming relatively negative and the other side relatively positive, thereby creating an electric dipole moment. When this induced dipole interacts with an external electric field, a torque is generated that drives rotational alignment.

The magnitude of this torque depends on the angle between the dipole moment and the electric field. When the angle is 90 degrees, the torque is maximized, corresponding to a highly misaligned configuration and a relatively high energy state. In contrast, when the angle is 0 degrees, the torque becomes zero, indicating that the dipole is fully aligned with the field and the system has reached its minimum energy state [20].

Consequently, the system naturally has a tendency to minimize its energy. This drives the gradual rotation of the dipole toward alignment with the electric field. The process continues until the dipole moment is parallel to the field direction, corresponding to the equilibrium configuration with minimum electrostatic energy. And the alignment process can be seen as follows:

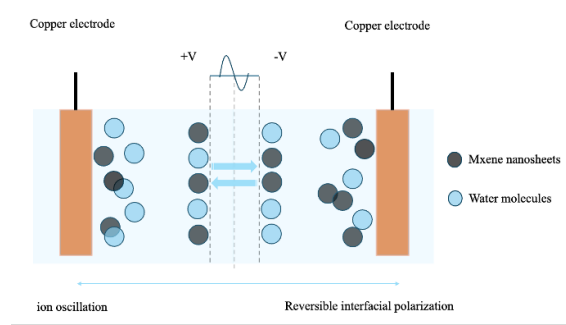


Figure 2.1: Alignment before electric field

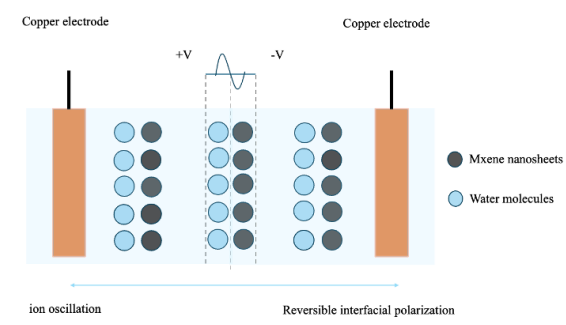


Figure 2.2: Alignment after electric field

The alignment of MXene nanosheets in an aqueous medium is governed by the interaction between an external alternating current (AC) field and the electrical double layer (EDL) of the particles. As illustrated, the application of a periodic potential (+V to -V) between the copper electrodes generates a high-frequency oscillating electric field. This process facilitates a transition from an isotropic dispersion to a highly anisotropic ordered state.

The primary driving force is reversible interfacial polarization (Maxwell-Wagner effect), where the difference in complex permittivity between the conductive MXene nanosheets and the polar water molecules induces a strong dipole moment. Because the nanosheets possess a high aspect ratio and surface charge, the resulting dielectrophoretic (DEP) torque overcomes Brownian motion, rotating the sheets into alignment parallel to the electric field lines.

2.1.3 Frequency dependence and critical frequency

Different frequencies activate different physical mechanisms, thus modulating the orientation of MXene sheets differently [16]. Interfacial polarization varies with the frequency of the applied electric field because it can affect the charge carriers from migrating and accumulate at the interfaces of the materials. So, the previous equation can be changed into:

$$f_c = \frac{1}{2\pi\tau_{MW}} \sim \frac{\sigma_{\text{eff}}}{2\pi\varepsilon_{\text{eff}}} \quad (2.3)$$

Basically, if the applied experimental frequency (f) is lower than the critical frequency (f_c), interfacial charges have sufficient time to migrate and accumulate at the interfaces, resulting in strong Maxwell–Wagner (MW) polarization and a high effective dielectric constant. In contrast, when the applied experimental frequency (f) exceeds the critical frequency (f_c), the charges can no longer follow the alternating electric field within a cycle. Consequently, MW polarization is suppressed, and the dielectric constant approaches the high-frequency limit governed primarily by electronic and atomic polarizability.

Because MXene sheets are highly conductive, with typical in-plane conductivities of $10^3\text{--}10^5 \text{ S m}^{-1}$, whereas aqueous phases typically have σ conductivities smaller than 1 S m^{-1} depending on ion concentration, the conductivity contrast drives strong MW effects at comparatively low frequencies. Therefore, the operating frequency depends on the σ and ε values of both phases, and the critical frequency (f_c) can be estimated using the relation above. This justifies the selection of the low-frequency AC range used experimentally, which is sufficiently low to enable charge accumulation while remaining high enough to avoid the formation of electrochemical products.

2.1.4 Intrinsic electronic and surface properties of MXene

MXene nanosheets (Mn+1XnTx) possess metallic/semi-metallic conduction due to delocalized transition-metal d-electrons combined with surface termination groups

(-O, -OH, and -F) that introduce net surface charge and a strong surface dipole. So, the high in-plane conductivity and charged surfaces make MXene both an efficient conductor and a source of interfacial polarization in electrolytes. Furthermore, MXene exhibits pronounced electrical anisotropy, the in-plane conductivity of individual nanosheets is several orders of magnitude higher than the through-plane conductivity. This anisotropy originates from the two-dimensional metallic bonding within the Ti-C layers, which enables highly efficient lateral electron delocalization, whereas interlayer spacing, surface terminations, and contact resistance between adjacent flakes limit charge transport perpendicular to the sheets.

This intrinsic anisotropy is essential to the electric-field alignment. In a randomly oriented network, electron transport is dominated by interflake junctions, tunneling gaps, and poorly connected edge contacts, which significantly increase resistance, the effective macroscopic conductivity is therefore constrained not by the intrinsic conductivity of MXene itself, but by the quality and orientation of inter-sheet contacts.

When an external electric field induces rotation and alignment of nanosheets, they tend to touch each other face-to-face, thus increasing the contact area, lowering the resistance, and creating more continuous paths for electrons in one direction; the distance electrons need to jump between sheets becomes smaller, and the resistance at the connection points between sheets becomes lower [21]. As a result, electricity can travel through the material more easily and smoothly, so alignment does not just change how the sheets are arranged, it makes the whole structure much better at conducting electricity.

In short, the alignment of MXene nanosheets in aqueous media under an applied electric field arises from interfacial polarization due to the mismatch in electrical conditions between MXene and water molecules. This Maxwell-Wagner polarization induces dipole moments on the nanosheets, which experience a torque in the external field, leading to rotational alignment. The efficiency of this alignment depends on frequency, ionic screening, and interfacial charge dynamics.

Researchers have traditionally employed a DC electric field to induce alignment of conductive nanosheets. However, instead of producing a well-defined aerogel structure, DC fields often lead to ion migration and electrochemical reactions, resulting in irreversible aggregation or asymmetric deposition on one side of the electrode. To overcome these limitations, an AC electric field is preferred, as its periodic reversal suppresses net ion transport and enables effective alignment of nanosheets.

In this project, due to limitations in available AC equipment, a Large Amplitude Sinusoidal Voltage (LASV) was applied using an electrochemical workstation to mimic AC conditions. LASV provides a time-dependent oscillating electric field that periodically reverses direction, enabling dynamic reorientation of MXene flakes while minimizing Faradaic reactions. And in this way, LASV reproduces the essential function of an AC field in promoting nanosheet alignment [22].

To implement this approach, which mimics an AC by LASV, a three-electrode electrochemical system was employed, which contains a working electrode (WE), a counter electrode (CE), and a reference electrode (RE), along with an electrolyte solution. The electrochemical reaction occurs on the surface of the WE, where the MXene dispersion is influenced by the applied field. The CE completes the electrical circuit by allowing current flow, while the RE maintains a stable potential in the cell, ensuring precise control of the applied voltage.

Under these conditions, the oscillating electric field induces dipoles within the MXene nanosheets, the periodic reversal of the field prevents permanent aggregation, while the controlled potential minimizes side reactions. As a result, the MXene flakes gradually align along the field direction of the electrical field due to repeated torque induced by the oscillating electric field.

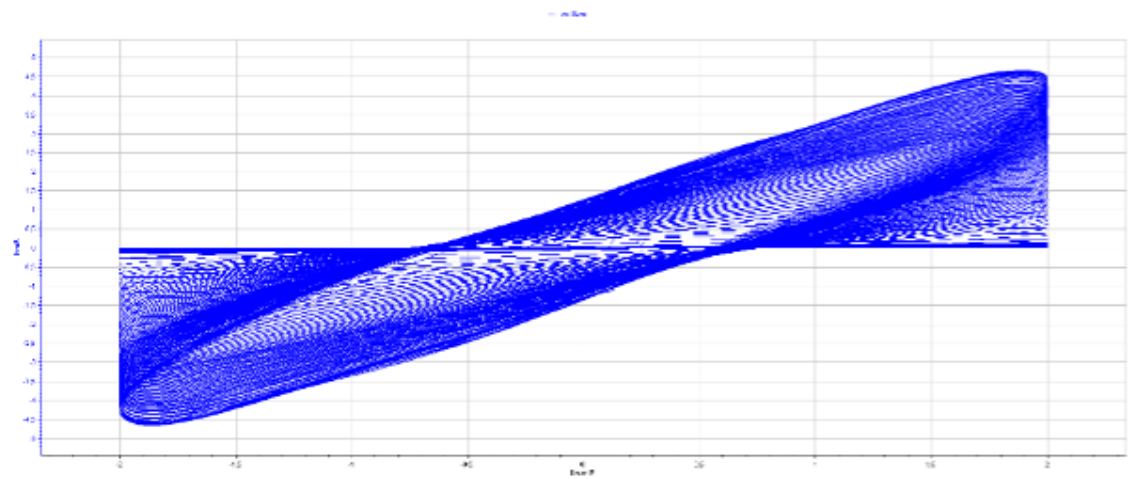


Figure 2.3: LASV technique

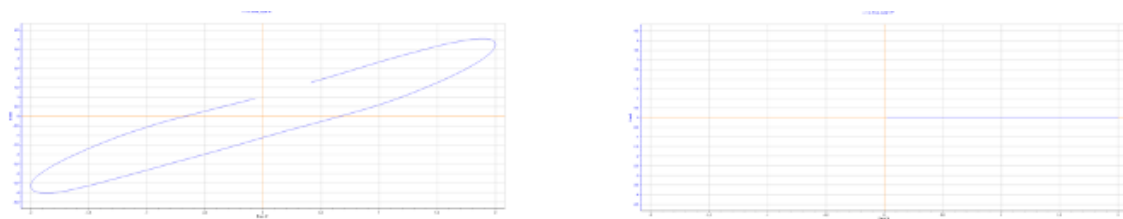


Figure 2.4: During LASV

To fully understand how this technique works, the following three figures provide a step-by-step illustration. By selecting appropriate parameters according to the condition of MXene, the applied voltage is kept high enough to align the MXene nanosheets, yet low enough to avoid water hydrolysis, as will be explained in detail in Chapter 3.

Figure 2.3 shows all the cycles applied to align the MXene nanosheets, although the overall trend is not clearly defined. Figure 2.4 (left) illustrates how the voltage

generates a torque to align the nanosheets. Notably, each cycle is gradually smaller than the previous one, eventually approaching zero. This behavior can be attributed to the fixed volume of the mold, within which the fraction of mobile solution diminishes continuously due to the freezing action of liquid nitrogen.

As a result, the effect of the electric field diminishes as the solution volume shrinks, which is further demonstrated in Figure 2.4 (right). Once the solution is completely frozen, the previously cyclical pattern transitions into a short, nearly linear response.

2.2 Critical frequency and field responsiveness of MXene

Previous studies have demonstrated that external electric fields can effectively modulate the charge distribution at MXene surfaces, underscoring the dominant role of interfacial effects in MXene-field interactions. The critical frequency governing polarization in aqueous MXene systems can also be interpreted via the RC model:

$$f_c \approx \frac{\sigma}{2\pi\epsilon} \approx \frac{1}{2\pi RC} \quad (2.4)$$

where f_c is the characteristic (or cutoff) frequency, σ is the electrical conductivity, ϵ is the permittivity, R is the ionic resistance across the electrode spacing, and C is the interfacial capacitance.

As discussed in the literature, ϵ measures the ability to store electric field energy, while σ reflects charge mobility. In aqueous MXene dispersions, the combination of high permittivity, moderate ionic conductivity, and significant EDL capacitance produces a relatively low critical frequency, favoring low-frequency AC electric fields for effective dipole polarization and alignment. Further demonstrate that external electric fields can redistribute surface electron density and modulate magnetic anisotropy in MXene. In solid-state systems, this response arises from electronic redistribution rather than ionic motion [23].

Therefore, the pronounced surface sensitivity of MXenes to applied electric fields further underscores the importance of interfacial charge-accumulation mechanisms. In a liquid system, MW polarization and EDL formation dominate, while in a solid-state system, electronic structure modulation dominates. This confirms the strong surface sensitivity of MXene to applied electric fields.

2.3 The purpose of using polyimide

Polyimide is prepared from the polycondensation of pyromellitic dianhydride (PMDA) and 4,4'-oxydianiline (ODA) to form polyamic acid (PAA), which is then neutralized (with triethylamine to form an ammonium salt) and thermally imidized to produce the aromatic imide backbone. Before delving into the formation of polyimide, the

properties of the two main chemicals needed to be clarified.

PMDA consists of a central benzene ring fused with two anhydride rings, and this rigid, planar structure gives the polymer high thermal stability. When it encounters a diamine, the anhydride rings open up to form covalent bonds, creating a polymer chain. ODA is an aromatic diamine containing two amine groups (-NH₂) connected by an ether oxygen (-O-) bridge. The ether oxygen acts like a hinge, allowing the aromatic rings to rotate, thus preventing the silica skeleton from being too brittle and allowing the material to withstand the stresses of freeze-drying without collapsing.

For the synthesis of polyimide, there are three main steps, which are the formation of PAA, PAA-TEA, and PI:

Poly(pyromellitic dianhydride-co-4,4'-oxydianiline), amic acid solution has been used in this paper, which is synthesized by polycondensation of the long pair of electrons on the nitrogen atom of the ODA, acting as the nucleophile. It attacks one of the electrophilic carbonyl carbons in the PMDA ring. This attachment creates a tetrahedral intermediate. Because NMP is a polar aprotic solvent, it stabilizes this transition state by solvating the molecules without donating protons that might interfere with the reaction. Then, the oxygen within the anhydride ring acts as a leaving group, resulting in an amide bond and a carboxylic acid group [24], [25].

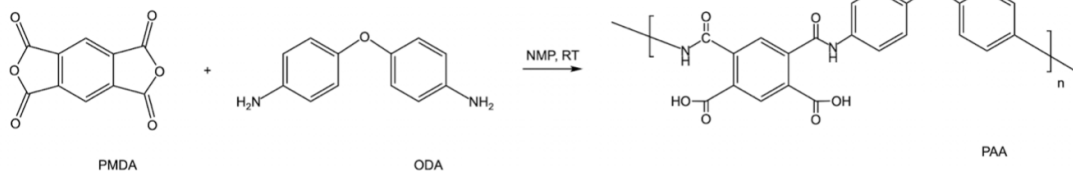


Figure 2.5: Formation of PAA

Thereafter, in order to get polyamic acid tertiary amine salt, a neutralization reaction is necessary by adding triethylamine. The long pair of electrons on the nitrogen in the TEA attacks the acidic proton of the carboxylic acid group on the PAA backbone, the proton is then transferred to the TEA, creating a positively charged triethylammonium cation and a negatively charged carboxylate anion on the polymer chain.



(R is ethyl group, C₂H₅)

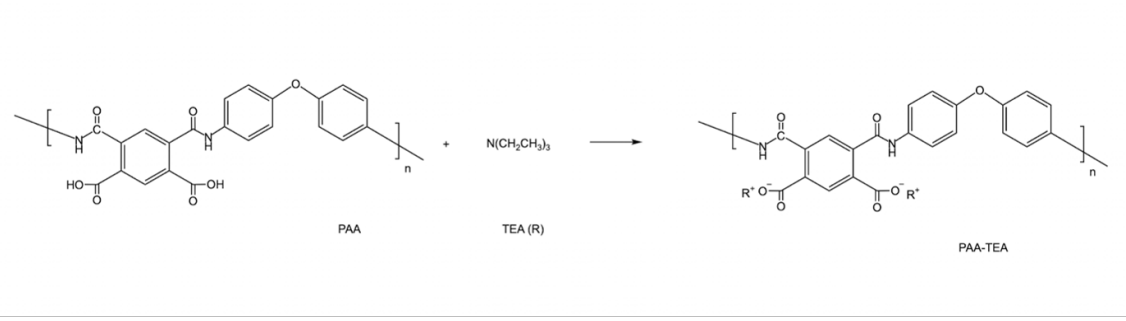


Figure 2.6: Formation of PAA-TEA

The final step is thermal imidization. The heat provides the activation energy for the oxygen in the amide group to attack the carbonyl carbon of the neighboring carboxylic acid, resulting in ring closure in which the amide nitrogen bonds to the carboxylic carbon and water is removed, thereby making the PAA chain a rigid aromatic heterocyclic polyimide.

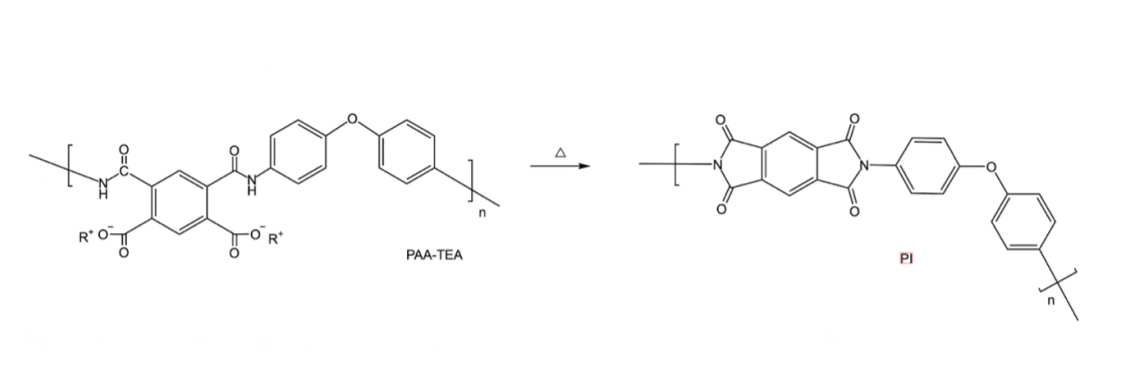
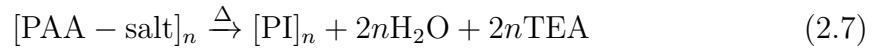


Figure 2.7: Formation of PI

The final product obtained from imidization is polyimide. Compared with traditional aerogel, polyimide replaces the fragile inorganic bonds of silica with a cross-linked aromatic polymer backbone, and a high glass transition temperature gives PI structural robustness and thermal stability. PAA/PIs permeate the silica skeleton, and MXene intercalates, forming strong covalent secondary interactions that prevent sheet drift and preserve the aligned structures formed under the field, especially after thermal curing. Besides, the aromatic, high-bond-energy backbone resists degradation and helps the aerogel tolerate elevated processing temperatures and electric fields without structural collapse [26], [27].

2.4 Hierarchical cross-linked network, conductivity, and microwave behaviour

The process begins with the formation of either a PAA-TEA matrix capable of stabilizing MXene nanosheets. Without these structural frameworks, the components would undergo phase separation.

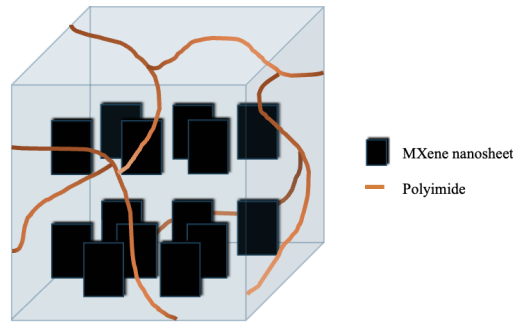


Figure 2.8: Hierarchical cross-linked network with an electric field

However, the main emphasis of this project's goal is to determine the optimal internal and external conditions. Based on these findings, the choice of additives remains flexible and can be adjusted to further enhance performance, provided they can align with the identified optimal conditions.

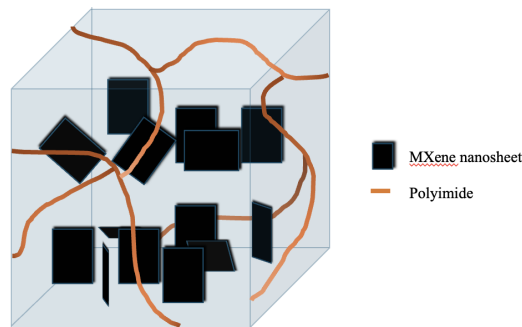


Figure 2.9: Hierarchical cross-linked network without an electric field

Thus, the process begins with the formation of poly (amic acid) (PAA) through the polycondensation of Poly(pyromellitic dianhydride-co-4,4'-oxydianiline), amic acid solution with triethylamine (TEA). PAA is partially neutralized to form the PAA-TEA salt, introducing carboxylate ($-\text{COO}^-$) groups and protonated triethylammonium (TEA^+). The negatively charged surface of MXene, terminated with $-\text{O}$, $-\text{OH}$, and $-\text{F}$ groups, strongly interacts with PAA-TEA through electrostatic attraction between MXene nanosheets and TEA^+ . Simultaneously, abundant hydrogen bonding forms between MXene $-\text{OH}$ groups and the amide ($-\text{CONH}-$) and

carboxyl groups of PAA, creating a physically cross-linked network. Owing to the high specific surface area of MXene nanosheets, PAA chains can wrap around the anchor onto their surfaces, further stabilizing the hybrid structure.

When an alternating electric field (AC field) is applied during gelation, the MXene nanosheets experience dipole-moment-driven torque and induced polarization. As a result, the two-dimensional sheets rotate and preferentially align along the electric field direction. This field-induced alignment promotes the formation of anisotropic conductive pathways, reducing the inter-sheet tunneling distance and enhancing charge-transport efficiency along the alignment direction. Consequently, the electrical conductivity of the aerogel becomes directional and is significantly improved compared to a randomly distributed system.

Simultaneously, the polyimide (PI) matrix, derived from the PAA-TEA precursor, permeates the aligned MXene framework. During thermal imidization, the polymer chains interlock within the interlayer spaces and form a covalently bonded network. This interpenetrating structure stabilizes the aligned MXene architecture, preventing sheet slippage or collapse after removal of the external field. The flexible ether linkages (-O-) in the PI backbone enable stress redistribution and energy dissipation under mechanical loading, thereby preserving mechanical robustness despite enhanced alignment.

In contrast, in the absence of an applied electric field, the MXene nanosheets remain randomly oriented within the polymer matrix. Due to their intrinsic anisotropies, this random distribution limits the formation of continuous conductive networks, resulting in lower overall electrical conductivity. Charge transport becomes dependent on stochastic intersheet contacts and increased tunneling resistance.

By summarizing all the possibilities, there are two major group need to be tested out. Group A, with an electric field, can be seen in the illustration Figure 2.8, aligns the MXene nanosheets into organized pathways, dramatically increasing conductivity. It behaves like aligned MXene layers wrapped in the PMDA-ODA polyimide web, preventing the structure from collapsing under stress.

Group B without an electric field can be seen in Figure 2.9. The tangled PMDA-ODA chains create more traps for microwaves to get in. Besides, because the MXene sheets are randomly tilted, the microwaves are bound in every direction inside the pores, leading to higher energy loss.

2.5 Mechanisms of experiments

2.5.1 Dialysis

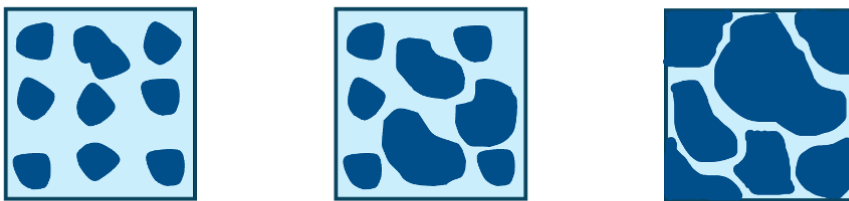
The project utilizes a pore-flow dialysis for the dialysis process. This system operates on a mechanism driven by a concentration gradient between molecules, specifically,

molecules naturally migrate from an area of high concentration to an area of low concentration until equilibrium is approached. To ensure precision in the dialysis processes, the specific pore size for the tubing is essential, which allows the target molecules to be removed because they can pass through the pores, while the useful molecules can be saved in the tubing by their larger size [28].

2.5.2 Freeze casting

The freeze casting process can be described as a directional solidification technique in which a solution or particle slurry is frozen under a controlled temperature gradient. Typically induced using liquid nitrogen. This process enables the formation of anisotropic structures through the controlled growth of solvent crystals. The process generally consists of two main steps: the first step is solvent crystal nucleation, phase separation, and directional solidification. The solution or particle slurry undergoes directional solidification, during which the solvent crystallizes along the imposed temperature gradient, segregating solutes or suspended particles into the intracrystalline regions. Additionally, the freezing rate affects the nucleation and size of pores in the aerogel. Technically, the fast-freezing rate leads to a high nucleation rate, which causes many nuclei to form in a short time. Due to the sudden dropping temperature, the water molecules don't have enough time to grow bigger and diffuse, leaving behind a structure with numerous small ice crystals. Then, the second step is the removal of the ice crystal via sublimation, typically via freeze-drying, leaving a matrix with a small, uniform, fine pore structure [13]. In short, fast freezing results in a high nucleation rate, leaving numerous small nuclei (ice crystals) and small pores. Slow freezing represents low nucleation, leading to few nuclei and larger pores after sublimation. Thus, depending on the aerogel structure, different freezing rates can be chosen and applied [29].

Slow freezing



Fast freezing

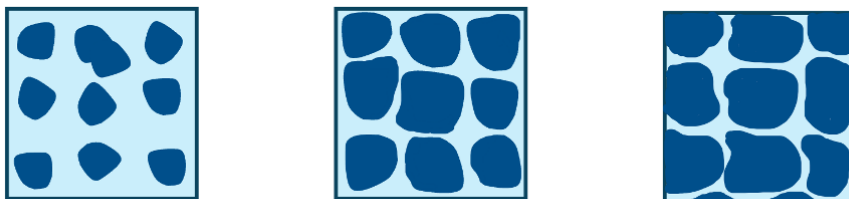


Figure 2.10: Slow freezing and fast freezing

2.5.3 Strain-stress hysteresis loop

Mechanical characterization of aerogel is commonly performed using compressive stress-strain analysis, which provides insight into stiffness, elasticity, deformation mechanisms, and structural recovery ability, and will be further discussed at the end of this thesis. Within these mechanisms, there are two main concepts, which are stress and strain, meaning applied force per unit area and relative deformation, respectively.

So, when an aerogel has been tested under cyclic loading and unloading, the loading and unloading stress-strain paths do not completely overlap. This phenomenon is known as hysteresis and reflects the irreversible internal processes and energy dissipation within the material. Further, the area between the loading and unloading curves, reflecting the dissipated mechanical energy, is called a hysteresis loop, and in porous aerogel systems, it is possibly due to the pore collapse or pore deformation, friction between network structures, or partial structural recovery during loading [30],[31]. Analysis of the hysteresis loop provides insight into the elasticity, recoverability, and mechanical stability of the aerogel for repeated loading applications.

2.5.4 Piezoresistive effect

When a conductive material changes its shape or volume due to external pressure, it will trigger the piezoresistive effect. Technically, the piezoresistive effect refers to the change in the electrical resistance of a conductive material when it is subjected to mechanical stress or strain. External force, such as pressure or compression, can change the material's geometry (shape or dimensions), and in some cases, it will change the intrinsic electrical properties.

As a result, the electrical resistance may either increase or decrease depending on the materials and the nature of the applied stress. This change in resistance can subsequently affect the voltage or current in an electrical circuit, making the piezoresistive effect more important to explain in sensors [32].

3

Methods

3.1 Molds

A custom mold was designed to enable simultaneous electric-field-assisted alignment and directional freeze casting. The mold consisted of PTFE sidewalls to provide electrical insulation and chemical stability. A copper bottom plate served as both the lower electrode and thermal conduction base to promote controlled freezing. Parallel copper plates were positioned to generate a uniform AC electric field across the suspension. Polypropylene plates were inserted between conductive components to prevent electrical short-circuiting while maintaining structural stability.

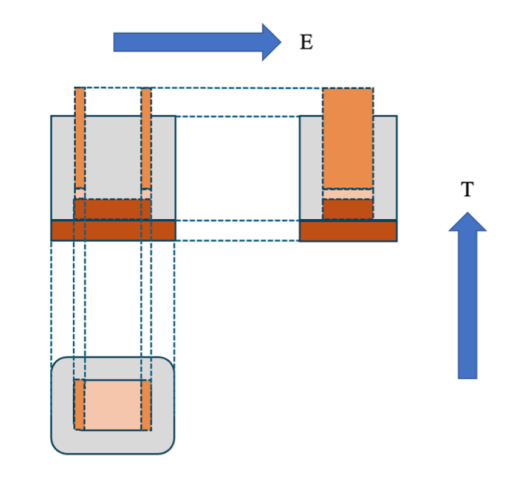


Figure 3.1: The applied electric field crossed with the temperature field

Other than that, another type of mold has been applied as a control group. In this case, the copper plate is added parallel to the copper bottom plate, which makes the electric field's direction the same as the temperature field, which is the opposite of the first mold.

By comparing these two molds, one with the applied electric field crossed with the temperature field, and one with the applied electric field parallel to the direction of the temperature field, to discover which direction can enhance the aerogel's structure.

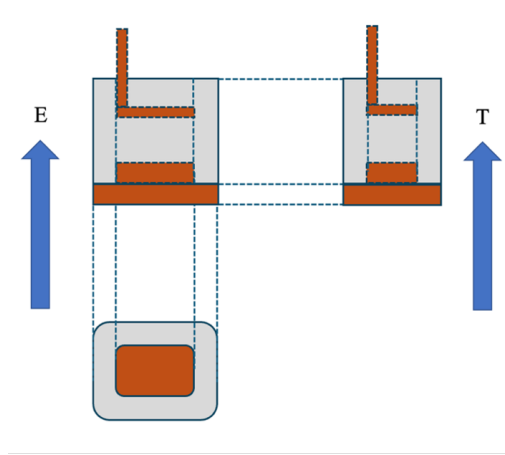


Figure 3.2: The applied electric field parallel to the temperature field

3.2 MXene synthesis

MXene ($\text{Ti}_3\text{C}_2\text{T}_x$) was synthesized by etching the Ti_3AlC_2 MAX phase. First, concentrated HCL (37) was slowly added to the distilled water under stirring. Then, LiF was added to the solution and stirred until it fully dissolved to generate the etching medium. Subsequently, Ti_3AlC_2 powder was gradually added, and the mixture was continuously stirred for 24-36 hours to complete the etching process. It is worth noting that the ratio between HCl, distilled water, LiF, and Ti_3AlC_2 is 7.5ml: 2.5ml: 0.8g: 0.5g.

After etching, the suspension was washed away and purified by sequential centrifugation: low-speed centrifugation at 4000 rpm for 5 min (4 cycles) followed by medium-speed centrifugation at 10000 rpm for 30 min (1 cycle), and high-speed centrifugation at 18000 rpm for 30 minutes to 1 hour (3 cycles). The product was then centrifuged at 2000 rpm for 2 min, and the supernatant was collected while the sediment was discarded [33], [34].

To obtain a higher MXene concentration, the supernatant was further centrifuged at 18000 rpm for 30 minutes to 1 hour, and the resulting material was collected and redispersed in 8-10 ml of distilled water to get a uniform, well-dispersed MXene dispersion.

3.3 Alternating current electric field

Continuing the unfinished explanation from Chapter 2, first, the setting voltage is around 2 volts, because theoretically, the water will have thermodynamic decomposition at 298 K and 1 to 1.23 volts. However, because of the reaction overpotentials and resistance losses, the actual voltage that will decompose water will be a bit higher, around 1.8 to 2.6 volts. So, consider the amount of the solution, voltage 2 will be applied in this project [35].

The experiment process can then be summarized as follows: connect the copper plates to the mold using the banana plug cables and set up the parameters for the alignment experiment. During the alignment, short circuits must be avoided, and the process lasts 5 minutes.

3.4 Liquid nitrogen vapor freezing

After the samples have been subjected to an AC electric field in the PTFE-copper mode for 5 to 10 min, the MXenes solution is placed above the liquid nitrogen to initiate freezing for 30 min, keeping it frozen continuously without contacting the liquid nitrogen directly. Then, post-freezing is maintained for 1-2 hours without an AC electric field. After the sample has been fully frozen, it is moved to a freeze-dryer machine and dried under vacuum for 2 days [16].

3.5 Processes in short

3.5.1 Neutralization of polyamic acid

The synthesis is initiated by neutralizing polyamic acid, also called salt formation, to convert polyamic acid (PAA) into the triethylammonium salt (PAA-TEA), rendering the polymer water-soluble while maintaining chain integrity before gel formation. Then, weigh 4.6 g (3.95 ml) of a 15 wt PAA solution in NMP into a 20 ml glass beaker. (Important: keep the beaker open, because the acid-base neutralization between the carboxylic acid groups of PAA and triethylamine is exothermic. Heat generation can cause expansion of air and volatile TEA, leading to dangerous internal pressure if sealed), followed by adding 0.39 g (530 μ L) of triethylamine (TEA) slowly to the PAA solution. Thereafter, stir the mixture under an ice-water bath for 2 hours. (Notes: during neutralization, the solution will be slightly increased in viscosity and change color to light yellow). Before moving to the next step, the solution must be perfectly homogeneous, with no visible phase separation or undissolved regions [36].

3.5.2 NMP extraction by dialysis

NMP is one of the hazards prevent aerogel from forming a good structure, instead, it will cause structure failure because it has extremely low vapor pressure. If NMP is still inside the mixture, it might not freeze solidly at standard freezer temperature. During the vacuum stage, the NMP will stay liquid and cause the ice crystal to melt, leading to a structural collapse. Other than that, NMP can sit at the surface of the MXene nanosheets acting as an insulating coating, decreasing the conductivity, resulting in less sensitivity to pressure or touch [37].

The solvent exchange process is designed to remove N-methyl-2-pyrrolidone (NMP), which acts as a strong plasticizer and interfaces with subsequent gelation and imidization processes. Dialysis is employed to gradually replace NMP with deionized (DI)

water under controlled conditions. The procedure begins by filling a 500 to 800-mL beaker with DI water. The PAA-TEA solution is then transferred into dialysis tubing, which is suspended at the top of the beaker. A gentle vortex is maintained in the surrounding water to promote uniform mass transfer while allowing sufficient time for the polymer chains to extend and orient.

Driven by the concentration gradient across the semipermeable membrane (with a pore size of approximately 3.5 mm), NMP diffuses out of the tubing into the DI water, while water simultaneously diffuses inward due to osmotic pressure. This bidirectional mass transfer facilitates gradual solvent exchange without abrupt structural disruption of the polymer network.

To ensure efficient removal of NMP, the external DI water is replaced periodically. On the first day, the water is refreshed every 2 hours to accelerate the initial extraction phases, during which most of the NMP is removed. On the second day, the replacement interval is extended to every 8 hours. Subsequently, the water is changed every 12 hours until no residual fishy odor is detected in the surrounding solution, indicating minimal remaining NMP.

During dialysis, several observable changes provide insight into the process. Initially, the PAA-TEA in NMP appears as a clear yellow, highly viscous liquid. As solvent exchange progresses, the solution should transition to a transparent light yellow or translucent milky yellow appearance. Deviations from this behavior, such as the development of a dark brown or opaque yellow, may indicate premature imidization or degradation, or excessively rapid NMP diffusion, which can hinder proper chain reorientation and lead to aggregation or clumping. Additionally, the solution’s viscosity is expected to decrease significantly as NMP is replaced with water. Also, because of osmotic pressure, the volume inside the dialysis tubing will likely increase, so if the tubing is tight at the end, it means water has entered the solvent-rich environment to push NMP out [24], [36], [38], [39].

3.5.3 Final mixing, freeze drying, and thermal curing

Combine the PAA-salt solution and MXene dispersion, then stir and sonicate for 30 to 60 minutes in total. First, 15 minutes at room temperature for uniform MXene distribution; next, 15-60 minutes at around 40 degrees to increase viscosity due to siloxane condensation [40].

Gel precursor solutions were cast into 4 mL molds and frozen under liquid nitrogen vapor freezing. The resulting samples were then subjected to a vacuum treatment following a stepped thermal profile, first held at 80 degrees for 2 hours to gradually remove residual solvents, then heated to 120 degrees for 2 hours to initiate imide ring closure, and finally maintained at 200 degrees for 10 hours to maximize imidization and ensure complete polymer network formation [41].

3.6 Experimental setup

The aerogel’s density was targeted at approximately 0.08 g/cm³, which is considered optimal for balancing structural integrity and functional performance. The mold volume was fixed at 4 ml to ensure consistency across all experimental conditions. Based on these parameters, the control variable method was adopted to systematically investigate the effect of processing conditions on the aerogel properties. Specifically, two types of molds were employed, one with an applied electric field and one without, along with four different solution ratios and compositions.

As summarized in the table below, the primary objective of this study is to determine the optimal aerogel composition and internal structure that can simultaneously achieve high mechanical strength and high electrical conductivity. These two properties are often in competition, making it essential to carefully tune both the material formulation and processing conditions. The reason the final ratios are between these three will be explained in the results [42], [43], [44], [45].

	Mold 1		Mold 2	
	With E	Without E	With E	Without E
PAA-TEA: MXene (65%: 35%)				
PAA-TEA: MXene (70%: 30%)				
PAA-TEA: MXene (80%: 20%)				

To identify the optimal ratio between PAA-TEA and MXene, the most direct and reliable approach is to first conduct experiments using mold 2 with an applied electric field. Under these conditions, the cooling process is isotropic, meaning that heat transfer occurs uniformly in all directions. This eliminates directional influences and allows for an unbiased evaluation of the intrinsic effects of composition on the aerogel structure and properties.

Once the optimal PAA-TEA to MXene ratio is determined under these baseline conditions, this ratio can then be applied to experiments involving molds with an applied electric field. By comparing the results obtained with and without the electric field, it becomes possible to isolate and analyze the influence of the electric field on the microstructure, mechanical performance, and electrical conductivity of the aerogels. This stepwise approach ensures a clear understanding of both compositional and processing effects, ultimately guiding the design of high-performance aerogel materials.

And in order to further prove the effectiveness of the effect from the electric field, based on the previous study, this project introduces, additionally, direct liquid nitrogen immersion freezing to freeze the structure before it is affected by the force from the cooling direction.

So, in short, this project uses liquid nitrogen vapor freezing to figure out the op-

	Mold 2	
	With E	Without E
PAA-TEA: MXene (65%: 35%)		
PAA-TEA: MXene (70%: 30%)		

timal ratio between PAA-TEA and MXene to produce an aerogel that is good in mechanical strength by adding an electric field, while using direct liquid nitrogen immersion freezing to dig into the morphology of the aerogel from the electric field. Usually, liquid nitrogen vapor freezing has a relatively slower cooling rate than direct liquid immersion freezing, which leads to a relatively larger pore size and a more interconnected structure, compared with direct liquid immersion freezing, even it has a very fast cooling rate and results out in a smaller pore size and relatively uniform structure, but it can cause sample deformation and cracking, which is not optimal in making aerogel.

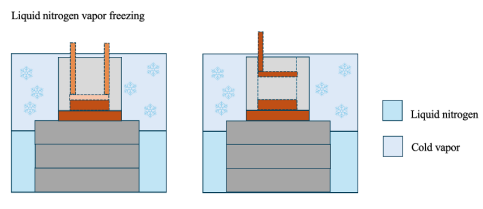


Figure 3.3: Two types of freezing speed mode

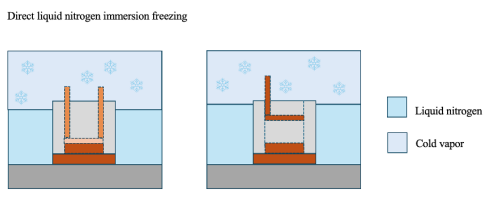


Figure 3.4: Two types of freezing speed mode

4

Results and discussions

4.1 Compressive modulus

Originally, this paper aims to discuss aerogel's structure in two types of molds, since the result is unexpected, as mold 1 would cause the failure of aerogel's structure, as seen in Figure 4.1 (left), it created a mountain-like structure in the middle, which was affected by the temperature field and formed another structure that perpendicular to the mountain-like structure which was affected by electric field. And this crossed structure makes aerogel properties testing harder. Thereafter, the samples tested in the following section are prepared from mold 2.

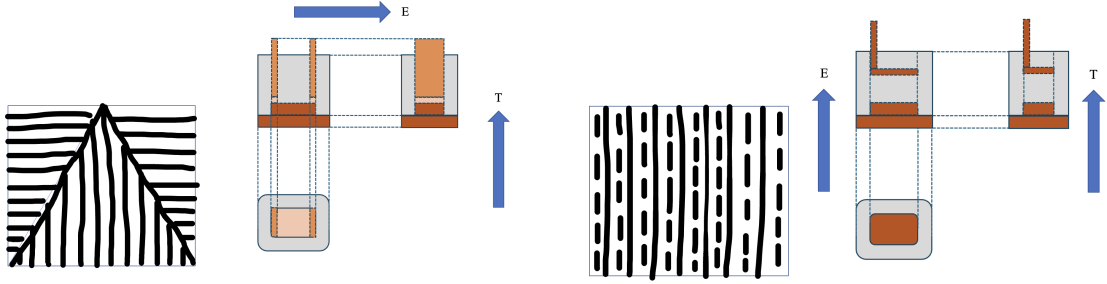


Figure 4.1: Preliminary results from two types of molds

The compressive modulus of aerogel specimens was obtained from compressive tests using a universal tensile testing machine, and four experimental groups were introduced. Before presenting the final results, several observations from the pre-synthesis stage should be clarified. Firstly, all the samples that have been tested in this thesis are prepared by mold 2, because compared with mold 1, mold 2 has a regular shape to test aerogel's properties. Secondly, during the preliminary experiments, additional PAA-TEA: MXene ratios, including 30%: 70%, 50%: 50%, and 60%: 40%, were prepared and evaluated. However, all of these aerogels exhibited brittle and loosely connected structures. In some cases, the samples were fragmented into small pieces even without external compression. These observations indicate that the PAA-TEA content must exceed the MXene content to sufficiently encapsulate the MXene nanosheets and effectively bridge the separated nanosheets into a stable three-dimensional network. Thirdly, the only variable is the concentration of MXene and the ratio between MXene and PAA-TEA, which means the concentration of PAA-TEA is constant.

Groups	Performance
1	MXene (32.5 mg/ml) without an electric field in PAA-TEA: MXene ratio of 65%: 35%
2	MXene (15.4 mg/ml) without an electric field in PAA-TEA: MXene ratio of 65%: 35%
3	MXene (15.4 mg/ml) with an electric field in PAA-TEA: MXene ratio of 65%:35%
4	Different displacement of MXene (15.4 mg/ml) with and without an electric field in PAA-TEA: MXene ratio of 65%: 35%
5	Different displacement of MXene (25 mg/ml) with and without electric field in PAA-TEA: MXene ratio of 65%: 35%
6	Different displacement of MXene (25 mg/ml) with and without electric field in PAA-TEA: MXene ratio of 65%: 35% in direct liquid immersion freezing

Based on the characterization results of the aerogel's superplastic behavior in groups 1 and 2, which contained different MXene concentrations but identical PAA-TEA concentrations, the ratios of 65%: 35% and 70%: 30% were identified as the most promising compositions from a macroscopic perspective. These samples maintained good structural integrity after freeze-drying. In contrast, the 80%:20% ratio exhibited obvious defects extending from the bottom to the top of the aerogel in both groups. This suggests that although sufficient PAA-TEA is necessary to fully coat the MXene nanosheets and stabilize the structure, excessive polymer content may exceed the structural tolerance of the system, resulting in visible defects. Therefore, the 80%: 20% ratio can be regarded as suboptimal.

To further evaluate the optimal composition, preliminary mechanical tests were conducted to investigate the relationship between elasticity and MXene concentration. The aerogel with a higher MXene concentration (32.5 mg/ml) could not fully recover its original shape after 70% compression, indicating irreversible deformation. By comparison, the aerogel with a lower MXene concentration (15.4 mg/ml) was able to recover after a displacement corresponding to 70% of its original height. The detailed analysis of this behavior is discussed in the following sections.

As discussed in Chapter 2, the hysteresis loop in the cyclic compression testing represents dissipated mechanical energy. A larger hysteresis loop area corresponds to greater energy dissipation, whereas a smaller hysteresis loop area indicates more reversible elastic deformation and lower internal energy loss during cyclic loading. Therefore, aerogels with smaller hysteresis loops exhibit more elastic behavior and improved recovery capability.

In addition to the loop area, the position of the hysteresis loop on the stress axis also provides important information. When two aerogels are compressed to the same strain, the sample with the higher stress response requires a greater force to deform. This indicates a stiffer material with a stronger, more effectively cross-linked network structure.

From the stress-strain plots of Group 1, shown in Figures 4.2, 4.3, 4.4, all three ratios were able to withstand at least 40% displacement. However, the 65%: 35% ratio exhibited both a smaller hysteresis loop area and a higher stress response compared with the 70%: 30% and 80%: 20% ratios. This suggests that, under an MXene concentration of 32.5 mg/ml, the 65%: 35% composition demonstrates superior elastic recovery and mechanical stability. A similar trend was observed in group 2, shown in Figures 4.5, 4.6, 4.7, where the maximum sustainable displacement increased to 50%. Once again, the 65%: 35% ratio displayed a smaller hysteresis loop and a higher stress response, confirming that the 80%: 20% ratio was mechanically less favorable. Further comparison between the 65%: 35% and 70%: 30% samples under 55% strain showed that although the hysteresis loop area was relatively similar, the 65%: 35% aerogel withstood higher stress at the same strain level. Consequently, the 65%: 35% ratio can be considered the optimal composition, despite the presence of slight plastic deformation observed during cyclic compression. Overall, these results indicate that an aerogel with an MXene concentration of approximately 15.4 mg/ml and a PAA-TEA: MXene ratio of 65%: 35% provides the most favorable balance between elasticity, structural stability, and mechanical strength. Based on this conclusion, subsequent experiments were designed using this optimized composition.

Group 1-MXene (32.5 mg/ml) without an electric field PAA-TEA: MXene ratio of 65%: 35% (Compressive stress versus strain curves during loading-unloading cycles with increasing strain amplitude)

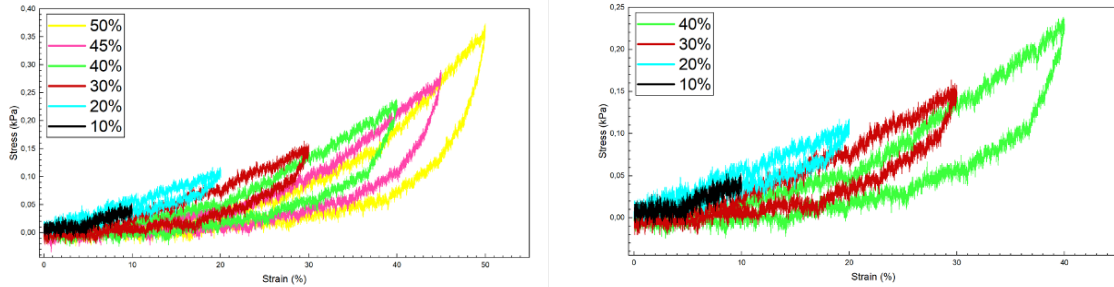


Figure 4.2: Strain-stress curves between ratio of 65%: 35%

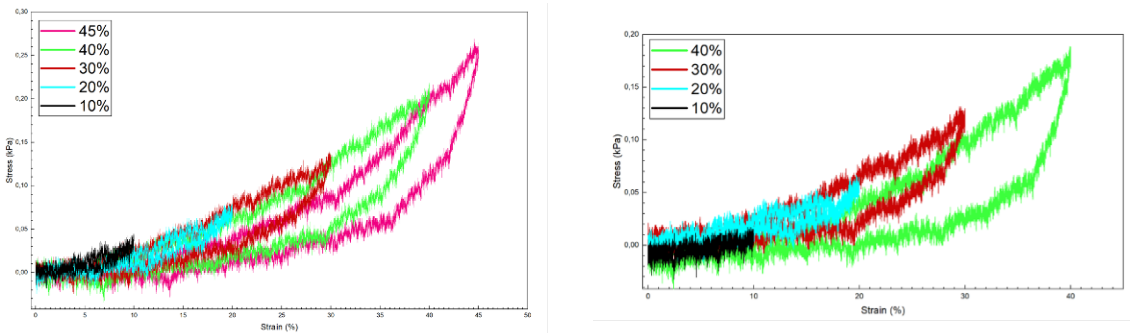


Figure 4.3: Strain-stress curves between ratio of 70% and 30%.

Figure 4.4: Strain-stress curves between ratio of 80% and 20%.

Group 2-MXene (15.4 mg/ml) without an electric field

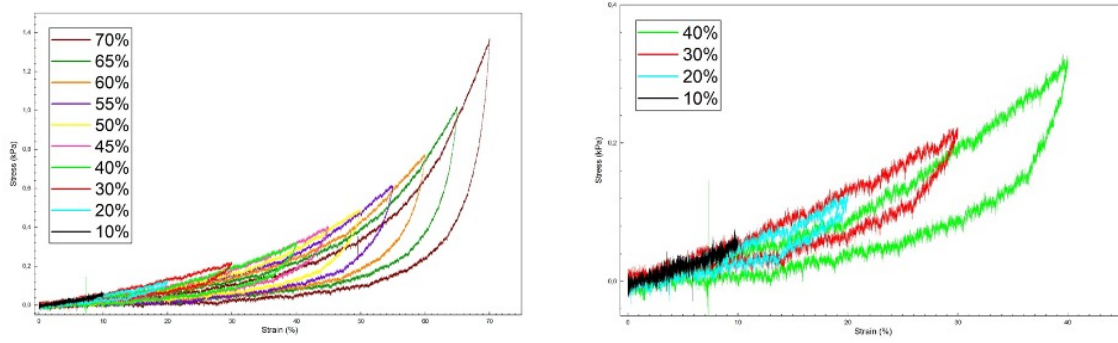


Figure 4.5: Strain-stress curves between ratio of 65%: 35%

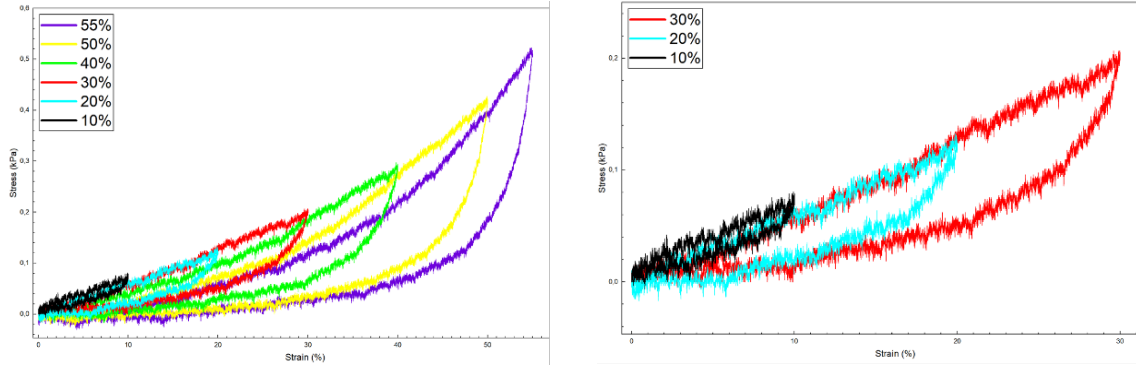


Figure 4.6: Strain-stress curves between ratio of 70%: 30%

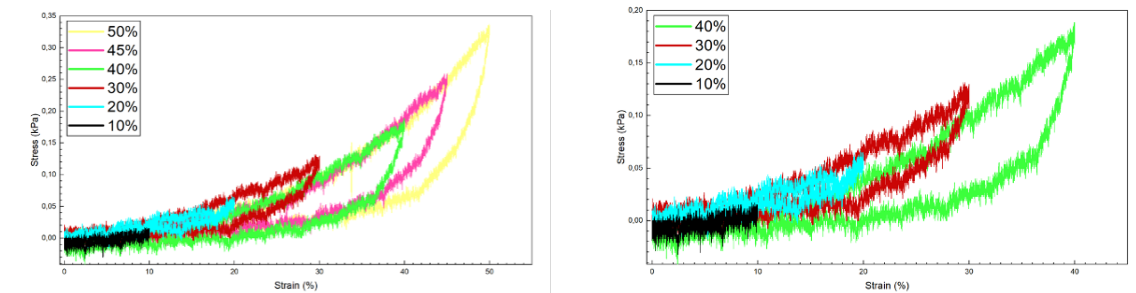


Figure 4.7: Strain-stress curves between ratio of 80%: 20%

To investigate the primary objective of this project, whether an applied electric field could align the MXene nanosheets and thereby strengthen the aerogel structure. Groups 3 and 4 were introduced. Group 3 was prepared using the optimized composition while incorporating an external elastic field during possession. Group 4 was designed specifically to compare aerogels with identical MXene concentration and composition, with and without the application of the electric field.

The results from Group 3 in Figures 4.8, 4.9 show that the 65%: 35% ratio consistently exhibited a higher stress response than the 70%: 30% ratio under the

same strain conditions, although the differences in hysteresis loop area were relatively small. This indicates that the 65%: 35% aerogel possessed a stronger network structure and could withstand greater stress. Furthermore, comparison between Groups 2 and 3 demonstrated that while the 65%: 35% ratio remained largely unchanged, the maximum strain displacement of the 70%: 30% ratio increased from 55% to 65% after applying the electric field. These findings suggest that the electric field contributes to structural reinforcement.

Additional evidence can be observed from the group 4 comparison between the 65%: 35% aerogels prepared with and without the electrical field (seen in Figures 4.10, 4.11, 4.12, 4.13). The stress-strain loop of the electrically treated sample was consistently positioned higher on the stress axis than that of the untreated sample, indicating that the electrically aligned aerogel could withstand greater stress under identical strain conditions.

In summary, the mechanical characterization demonstrates that the 65%: 35% PAA-TEA: MXene ratio provides superior performance compared with the other investigated compositions. Moreover, applying an electric field further enhances the aerogel's structural strength, enabling it to withstand higher compressive stress.

Group 3-MXene (15.4 mg/ml) with an electric field PAA-TEA: MXene ratio of 65%: 35%

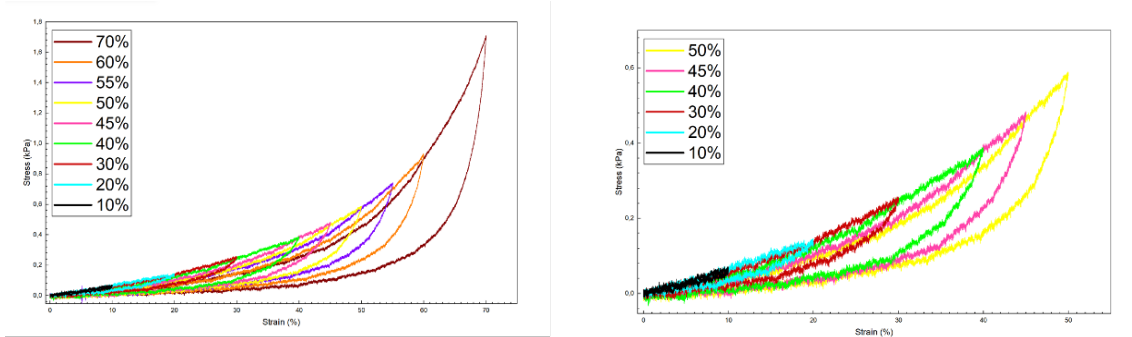


Figure 4.8: Strain-stress curves between ratio of 65%: 35%

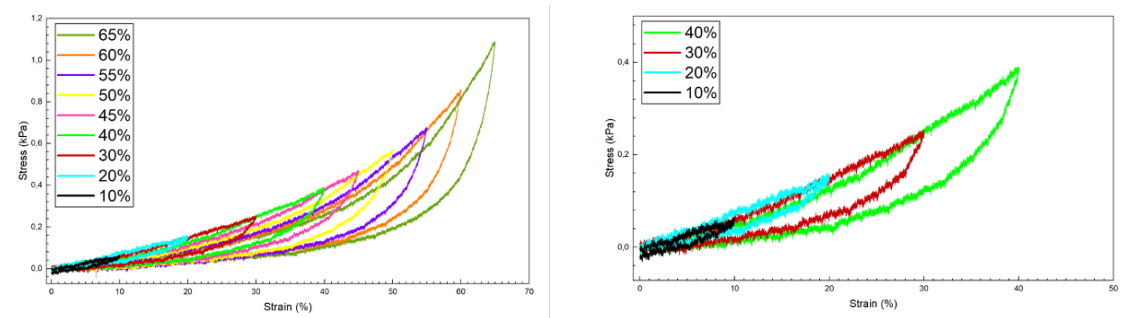


Figure 4.9: Strain-stress curves between ratio of 70%: 30%

4. Results and discussions

Group 4-different displacement of MXene (15.4 mg/ml) with and without an electric field in PAA-TEA: MXene ratio of 65%: 35%

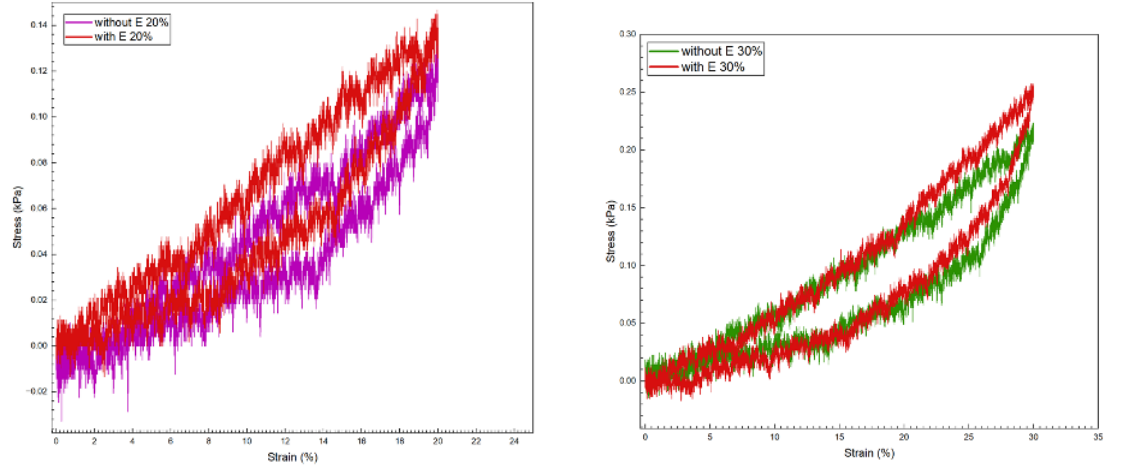


Figure 4.10: Displacement between 20% and 30%

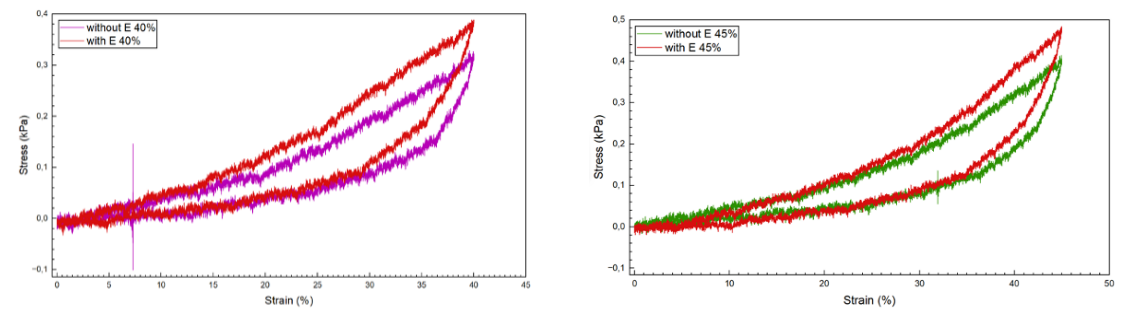


Figure 4.11: Displacement between 40% and 45%

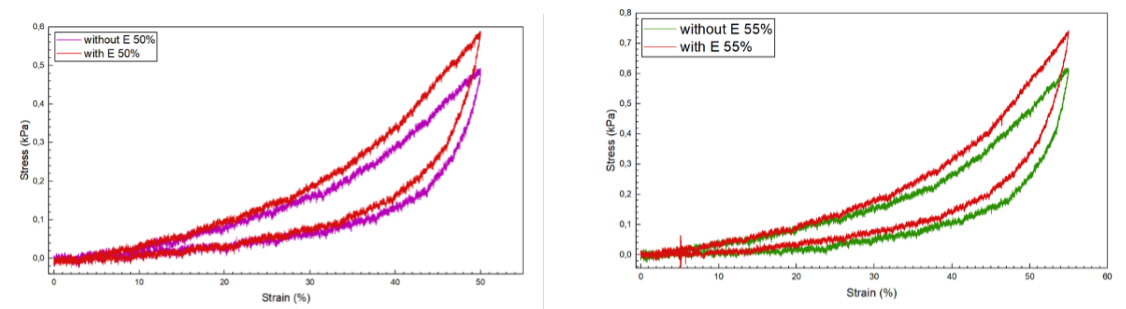


Figure 4.12: Displacement between 50% and 55%

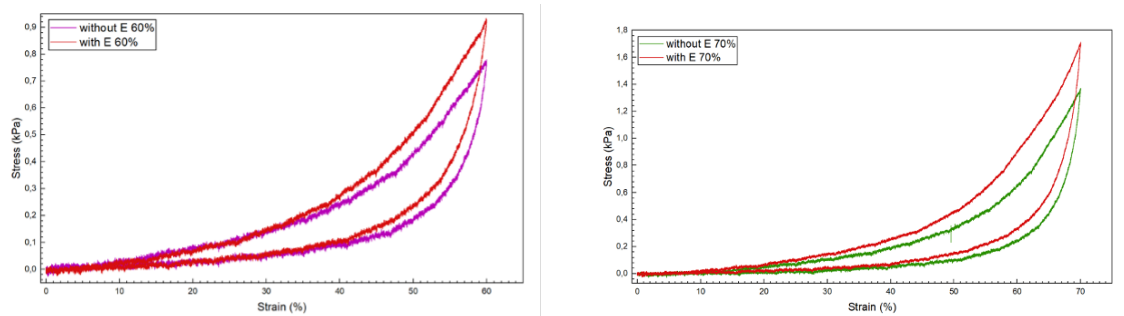


Figure 4.13: Displacement between 60% and 70%

However, high mechanical strength alone is insufficient for a material to function effectively as a sensor. Electrical conductivity is equally important for sensor functions. Therefore, resistance measurements were conducted on all aerogels to evaluate their electrical performance. The results showed that, although the aerogel with an MXene concentration of 15.4 mg/ml exhibited excellent mechanical strength, no measurable electrical response was detected during resistance testing. This suggests that the concentration of MXene naosheets was insufficient to establish continuous conductive pathways throughout the aerogel structure. In contrast, aerogel with higher MXene concentrations exhibited clear resistance responses, indicating the formation of conductive networks. Therefore, in addition to the two MXene concentrations discussed previously, a concentration of 25mg/ml was introduced to achieve a better balance between mechanical strength and electrical conductivity. The corresponding aerogels' results are presented as groups 5 and 6 in the previous chapter. Further, the effect of liquid nitrogen vapor freezing and direct liquid immersion freezing was investigated in the following context.

As shown in Figure 4.14 4.15, the aerogel fabricated under an applied electric field can undergo larger strain deformation without obvious irreversible deformation. In addition, the energy dissipation during compression is lower than that of the aerogel prepared without an electric field, which is consistent with the observations discussed in the previous section. Thereafter, based on this conclusion, the aerogel with MXene concentration at 25 mg/ml achieved mechanical properties comparable to those of the 15.4 mg/ml sample while maintaining sufficient electrical conductivity. Then, to further evaluate the influence of the freezing method, Figures 4.14 and 4.16 from Groups 5 and 6, respectively, were compared. Both with prepared under an electric field, with the only difference being the freezing process: liquid nitrogen vapor freezing versus direct liquid immersion freezing. The results indicate that the aerogel prepared by liquid nitrogen vapor freezing can withstand higher compressive stress at the same strain level. For example, at a strain of 60% (the orange line), the liquid nitrogen vapor frozen aerogel sustained a stress of around 1.6 kPa, whereas the directly immersed aerogel reached only about 1.1 kPa. Similar trends were observed at strain levels of 55%, 50%, and 45%, etc., demonstrating the superior mechanical performance of the vapor-frozen structure.

Another important conclusion can be drawn regarding the effect of MXene con-

centration. Even though MXene concentration at 15.4 mg/ml can achieve a high mechanical strength, no measurable resistance response was observed during electrical testing, indicating that conductive pathways were not sufficiently developed within the structure. In contrast, the aerogel with MXene concentration at 32.5 mg/ml provided adequate conductivity for sensing applications but exhibited reduced mechanical strength. The aerogel containing 25 mg/ml MXene achieved the most balanced performance, combining high mechanical strength with a clear electrical response, making it the most suitable candidate for sensor fabrication.

Group 5-Different displacement of MXene (25 mg/ml) with and without electric field in PAA-TEA: MXene ratio of 65%: 35%

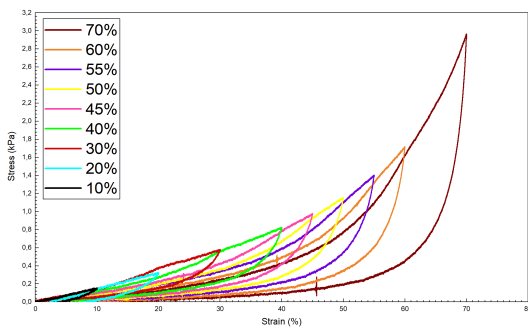


Figure 4.14: Strain-stress curves between ratio of 65% and 35% with an electric field.

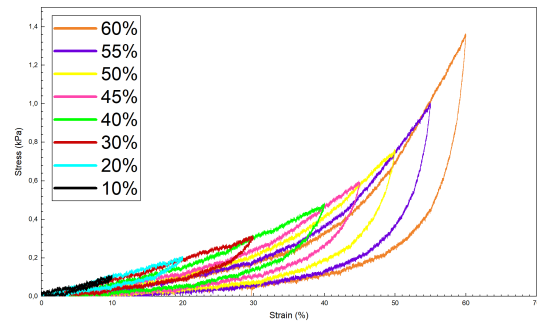


Figure 4.15: Strain-stress curves between ratio of 65% and 35% without an electric field.

Moreover, the superior mechanical properties of the liquid nitrogen vapor-frozen aerogel can be attributed to the ice crystal growth process. During vapor freezing, ice crystals grow more gradually and have sufficient time to develop an interconnected and well-organized porous network. In contrast, direct liquid immersion freezing causes rapid solidification, which tends to produce less ordered and more isolated pore structures. Consequently, the interconnected architecture formed during vapor freezing contributes to enhanced structural integrity and improved mechanical performance. Therefore, among all the investigated aerogels, the aerogel prepared with MXene concentration at 25 mg/ml under an applied electric field and subjected to liquid nitrogen vapor freezing demonstrated the most promising combination of mechanical strength and electrical conductivity, making it the optimal candidate for flexible sensor applications.

Group 6-different displacement of MXene (25 mg/ml) with and without electric field in PAA-TEA: MXene ratio of 65%: 35% in direct liquid immersion freezing

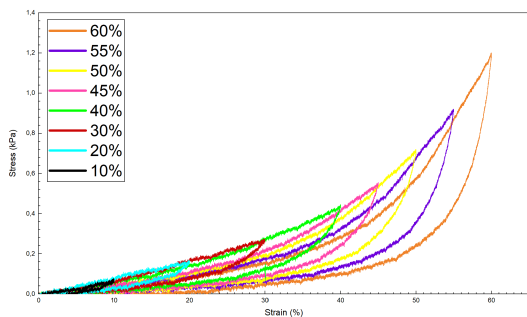


Figure 4.16: Strain-stress curves between ratios of 65% and 35% with an electric field in direct liquid immersion freezing.

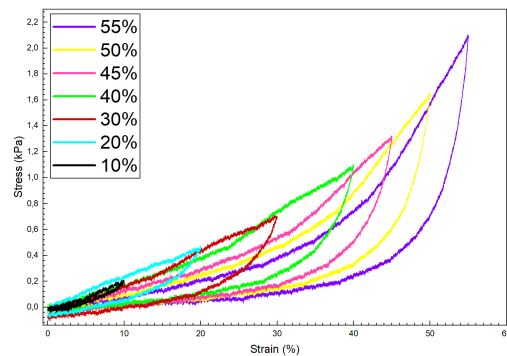
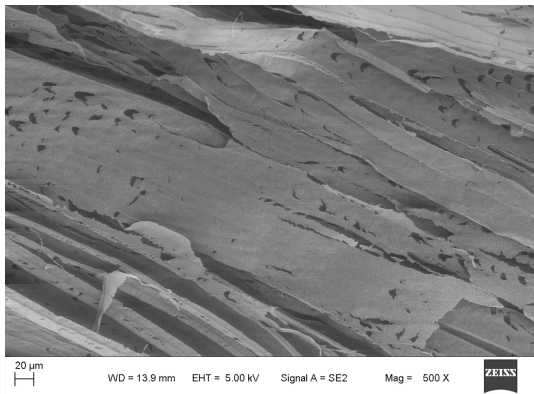


Figure 4.17: Strain-stress curves between ratios of 65% and 35% without an electric field in direct liquid immersion freezing.

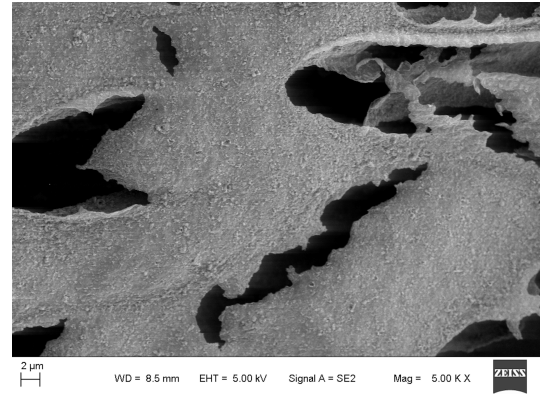
4.2 Microscopic view-SEM

The following section will focus on the SEM images. SEM images of the MXene/PAA-TEA aerogels prepared with and without an applied electric field are shown in Figure 4.18 and Figure 4.19. The obvious morphological difference between these two types of aerogels is first observed at low magnification. The sample prepared under an electric field shows a more aligned and continuous lamellar structure, whereas the aerogel prepared without an electric field shows a less ordered structure. This observation implies that the applied electric field promotes the orientation of the structural components during aerogel formation. This conclusion connects and supports the previous mechanical strength section that the electric field strength of the aerogel structure facilitates more efficient stress transfer and directional transport pathways within the aerogel, potentially contributing to enhanced mechanical performance.

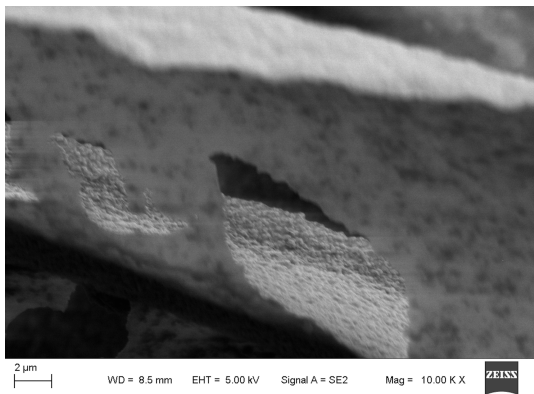
At higher magnifications between each Figures (c) and (d) that shows more details of each sample, both samples display a rough surface morphology consisting of a continuous PAA-TEA matrix with dispersed nanoscale features. These features are attributed to MXene sheets distributed throughout the polymer network. However, there are some substantial differences in the morphology of the MXene spreading domains observed between the two samples. For example, starting from figure 4.19 (c), it looks like MXene nanosheets are accumulated along one line of the side, other than that, MXene nanosheets are also spreading randomly along this piece of facade, making lots of small groups gather on different surfaces. And this situation also happens in image (d). On the contrary, Figure 4.18, through the hole, analyzes the spreading of the MXene nanosheets, showing that they are relatively covering the surface regularly and with less random stacking. Therefore, the preliminary effect of the electric field appears to be the enhancement of structural alignment at the microscale, enabling them be able to withstand more stress or any external force.



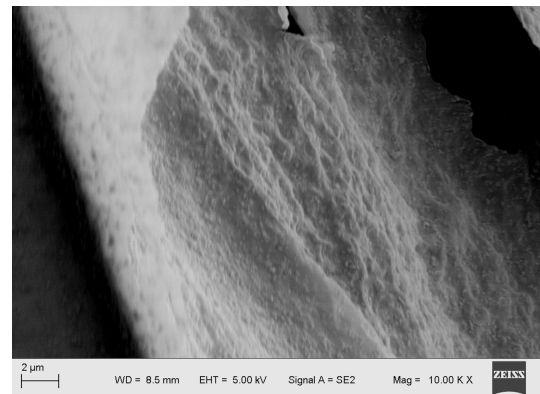
(a) Mag:500 X



(b) Mag:5000 X

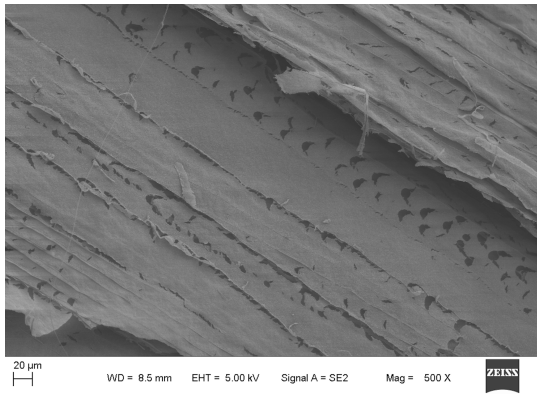


(c) Mag:10000 X

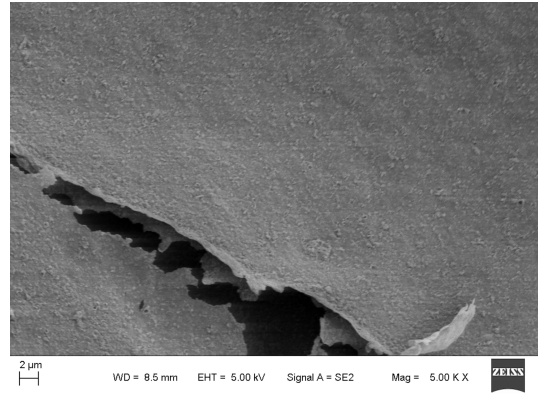


(d) Mag:10000 X

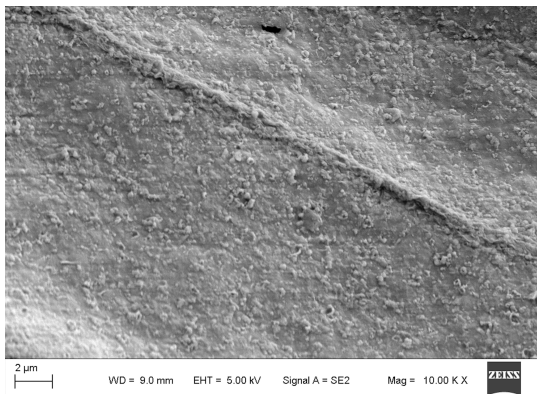
Figure 4.18: Scanning electron microscopy of aerogel prepared with an electric field



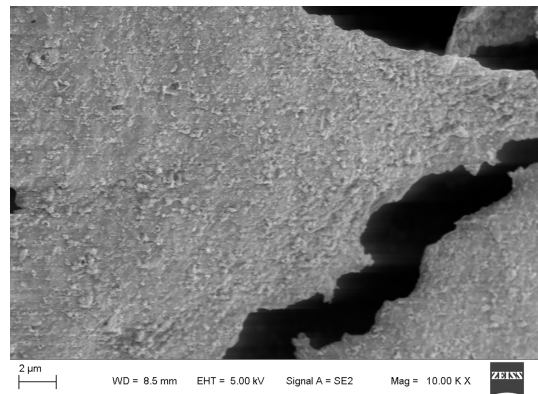
(a) Mag:500 X



(b) Mag:5000 X



(c) Mag:10000 X



(d) Mag:10000 X

Figure 4.19: Scanning electron microscopy of aerogel prepared without an electric field

4.3 Sensor testing

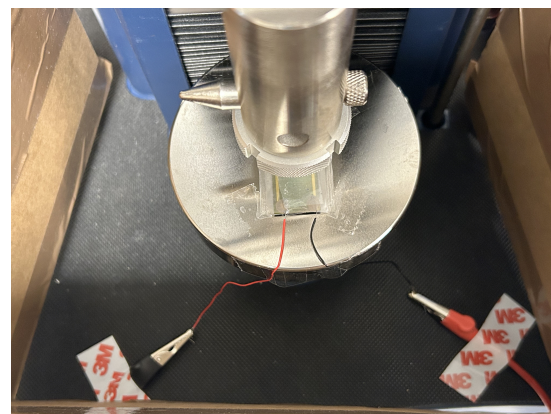
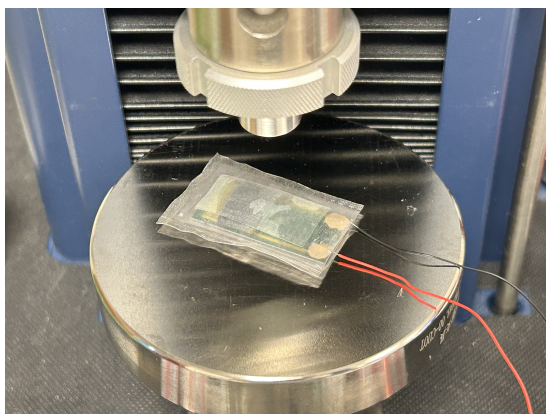


Figure 4.20: Testing setup

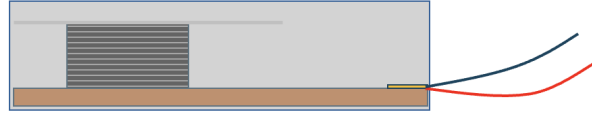


Figure 4.21: Schematic illustration of the fabricated sensor structure

The fabricated sensor consists of a multilayer structure designed for mechanical stability and signal transduction. The device is built on a bottom electrode, which serves as the primary electrical contact for signal collection. A porous aerogel layer is placed directly on top of the bottom electrode, acting as the active sensing material due to its high surface area and sensitivity to mechanical deformation.

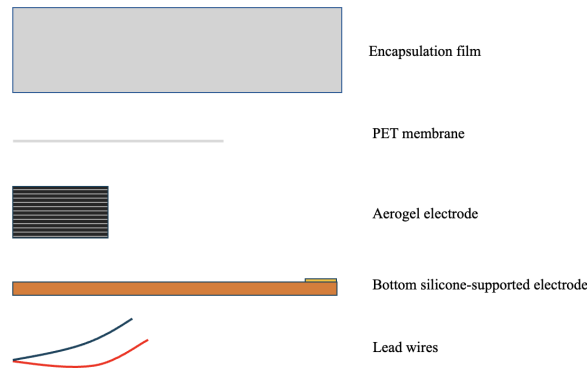


Figure 4.22: Schematic illustration of the fabricated sensor structure in details

To enhance mechanical durability and prevent structural collapse of the aerogel, a flexible PET membrane is placed above the aerogel as a supporting layer. Finally, the entire structure is encapsulated within a PET-based pocket, which provides overall mechanical protection and maintains device stability during repeated deformation.

This study investigated the pressure-sensing behavior of a parameter sensor. The mechanical properties of the conductive aerogel determined the performance of the pressure sensor, as shown in Figure 4.20. Pressure sensing depends on the piezoresistive effect, meaning that when the sample's geometric dimensions or shape change under applied pressure, its resistivity also changes accordingly. When no pressure is applied, the resistance of the conductive aerogel remains constant, and the sample produces no voltage. If there is no voltage, the current is zero. Thereafter, the current through the sample responds linearly to the applied voltage. In short, when pressure is applied to the sample, changes in its shape and volume alter the conductive pathways in the porous structure, and the resistance changes accordingly, based on Ohm's Law:

$$V = IR \quad (4.1)$$

Here V is the electrical potential difference, I is the electric current, and R is the

electrical resistance, which represents the opposition to the flow of electric charge.

Thus, pressure deforms pores in the porous aerogel, increasing the contact area, creating more conductive pathways, decreasing resistance, and increasing current (Voltage is stable). In order to evaluate the pressure sensitivity of this sensor, to directly reflect the sensor's performance, pressure sensitivity has been introduced:

$$S = \frac{\Delta I / I_0}{\Delta P} \quad (4.2)$$

where I_0 is the initial current of the sensor, ΔI is the change in current induced by the applied pressure, and ΔP is the change in the applied pressure.

Figures 4.23(left) and (right) present the relative current change (I) as a function of applied pressure for displacement values of 0.6 mm and 0.65 mm, respectively, under no electric field. Linear fitting of the experimental data yields pressure sensitivities of 0.54252 kPa^{-1} and 0.28247 kPa^{-1} , with corresponding coefficients of determination R^2 of 0.9911 and 0.9913. These high R^2 values indicate excellent linearity between pressure and relative current change over the measured pressure range.

In contrast, the sample under an electric field, Figure 4.24, shows the pressure-response characteristics of the sensor when an electric field is applied. The calculated sensitivities increase to 0.74414 kPa^{-1} and 0.56447 kPa^{-1} for displacements of 0.6. and 0.65 mm, respectively. The corresponding R^2 values are 0.9730 and 0.9590, indicating that the sensor response remains strongly linear despite a slight increase in data scatter [46] [47]. The coefficient of determination (R^2) is used to evaluate how well the fitted linear model represents the experimental data. Usually, if the R^2 value is higher than 0.95 will be considered to be accurate enough to describe a linear model over the investigated pressure range [48].

Comparing the results obtained with and without the electric field shows that the application of an electric field significantly enhances the pressure sensitivity of the sensor. For the 0.6 mm displacement case, the sensitivity increases from 0.54252 kPa^{-1} to 0.74414 kPa^{-1} , corresponding to an improvement of approximately 36.5%. For the 0.65 mm displacement case, the sensitivity increases from 0.28247 kPa^{-1} to 0.56447 kPa^{-1} , representing an enhancement of around 99.8%. These results suggest that the electric field effectively amplifies the electromechanical response of the sensing material, leading to larger current variations under the same applied pressure.

4. Results and discussions

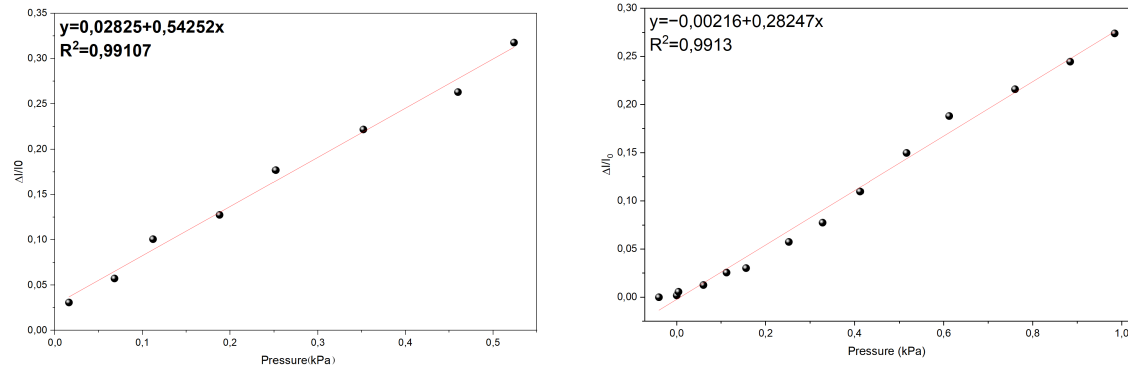


Figure 4.23: Relative changes in current corresponding to different applied pressures without electric field

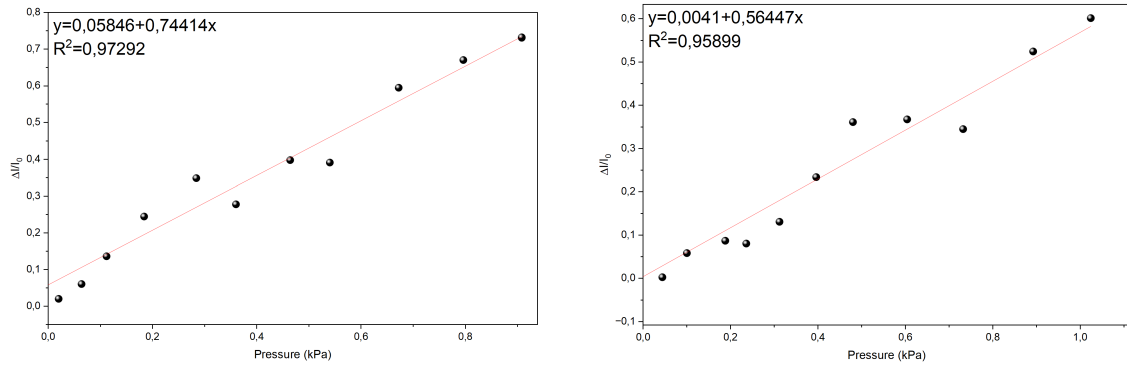


Figure 4.24: Relative changes in current corresponding to different applied pressures with electric field

Then, comparing the long-term cyclic stability test of these two types of samples, Figure 4.25 and Figure 4.26, shows that the sample with an electric field can almost keep the same high level from beginning to end, while the sample without an electric field keeps going down over time. Thus, it can be said that the sample with an applied electric field is more stable than the sample without an applied electric field when they are pressed in the same displacement.

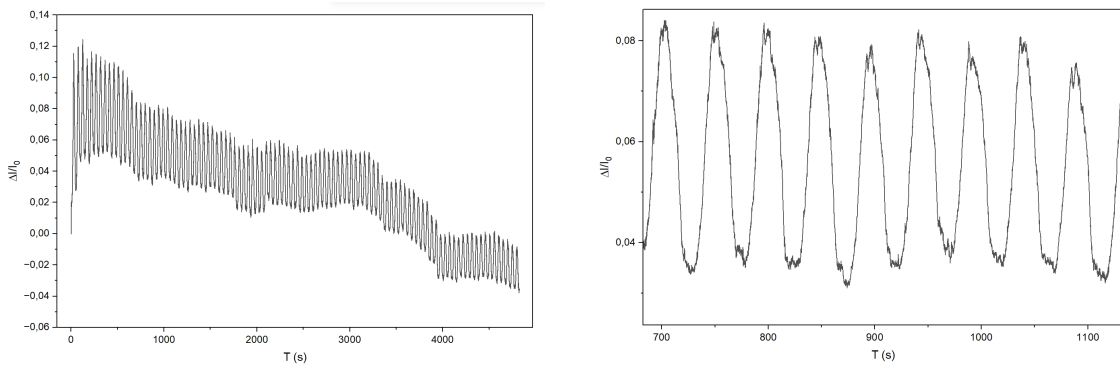


Figure 4.25: The long-term cyclic stability test of 0.4 mm displacement for 100 cycles without an electric field

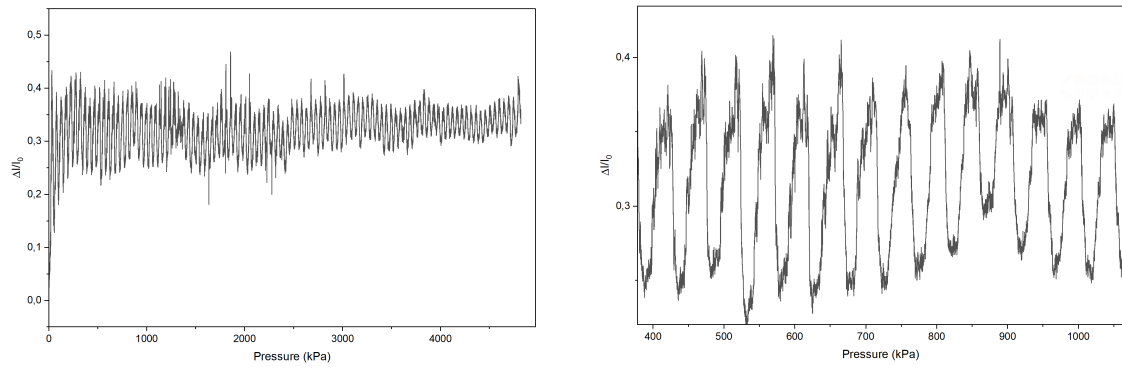


Figure 4.26: The long-term cyclic stability test of 0.4 mm displacement for 100 cycles with an electric field

5

Conclusions

Based on the results obtained in this study, the aerogel sensor fabricated using PAA-TEA: MXene ratio of 65%: 35%, with a MXene concentration of 25 mg/ml, and formed under liquid nitrogen vapor freezing process, demonstrated excellent performance. The resulting aerogel exhibited high mechanical strength, high pressure sensitivity, and good long-term cyclic stability, indicating its suitability for flexible pressure-sensing applications. Furthermore, the electric field applied during the fabrication contributed to the formation of an anisotropic porous structure, which enhanced the sensing performance of the material.

This study only focused on the development and characterization of polyimide/MXene composite aerogel fabricated under an electric field. Future work may explore the incorporation of additional polymer components or reinforcing agents to further improve the mechanical robustness and durability of the aerogel while still maintaining its sensing performance. Thereafter, sensor development may reach a new level.

Bibliography

- [1] [Puttavva Meti et al. “Evolutionary Progress of Silica Aerogels and Their Classification Based on Composition: An Overview.” *Nanomaterials*, vol. 13, no. 9, 27 Apr. 2023.]
- [2] [Shahriar Ghaffari-Mosanenzadeh, et al. “Recent Advances in Tailoring and Improving the Properties of Polyimide Aerogels and Their Application.” *Advances in Colloid and Interface Science*, vol. 304, 1 June 2022.]
- [3] [Ziegler, Christoph, et al. “Modern Inorganic Aerogels.” *Angewandte Chemie International Edition*, vol. 56, no. 43, 22 Sept. 2017.]
- [4] [Cai, Z., Kim, H. Recent advances in MXene gas sensors: synthesis, composites, and mechanisms. *npj 2D Mater Appl* 9, 66 (2025).]
- [5] [Faustini, Marco, et al. “History of Organic-Inorganic Hybrid Materials: Prehistory, Art, Science, and Advanced Applications.” *Advanced Functional Materials*, vol. 28, no. 27, 14 Apr. 2018.]
- [6] [Meti, Puttavva, et al. “Overview of Organic–Inorganic Hybrid Silica Aerogels: Progress and Perspectives.” *Materials and Design*, vol. 222, Oct. 2022.]
- [7] [Worsley, M. A.; Kucheyev, S. O.; Satcher, J. H.; Hamza, A. V.; Baumann, T. F. Mechanically Robust and Electrically Conductive Carbon Nanotube Foams. *Appl. Phys. Lett.* 2009, 94, 073115.]
- [8] [Worsley, Marcus A, et al. “Synthesis and Characterization of Highly Crystalline Graphene Aerogels.” *ACS Nano*, vol. 8, no. 10, 8 Oct. 2014.]
- [9] [Mao, Jiajun, et al. “Graphene Aerogels for Efficient Energy Storage and Conversion.” *Energy and Environmental Science*, vol. 11, no. 4, 2018.]
- [10] [Woo, Jeong Hyeon, et al. “Effects of the Addition of Boric Acid on the Physical Properties of MXene/Polyvinyl Alcohol (PVA) Nanocomposite.” *Composites Part B: Engineering*, vol. 199, Oct. 2020.]
- [11] [Tan, K.H., et al. “Optical and Conductivity Studies of Polyvinyl Alcohol-MXene (PVA-MXene) Nanocomposite Thin Films for Electronic Applications.”

- Optics and Laser Technology, vol. 136, Apr. 2021.]
- [12] [Yao, Yuanyuan, et al. “MXene Hybrid Polyvinyl Alcohol Flexible Composite Films for Electromagnetic Interference Shielding.” *Applied Surface Science*, vol. 578, 24 Nov. 2021.]
 - [13] [Wegst, Ulrike G. K., et al. “Freeze Casting.” *Nature Reviews Methods Primers*, vol. 4, no. 1, 25 Apr. 2024.]
 - [14] [Li, Wenjie, et al. “Electric Field-Induced Ordered-Structural Aerogels Enable Superinsulation and Multifunctionality.” *Small*, vol. 20, no. 51, 14 Oct. 2024.]
 - [15] [Wei, Chunxiang, et al. “Recent Advances in MXene-Based Aerogels: Fabrication, Performance and Application.” *Advanced Functional Materials*, vol. 33, no. 9, 26 Dec.2022.]
 - [16] [Lee, Changjae, et al. “Field-Induced Orientational Switching Produces Vertically Aligned Ti₃C₂T_x MXene Nanosheets.” *Nature Communications*, vol. 13, no. 1, 24 Sept. 2022.]
 - [17] [Ravindran, Anil R, et al. “Synergistic Delamination Toughening of Composites Using Multi-Scale Carbon Reinforcements.” *Composites. Part B, Engineering*, vol. 161, 1 Mar. 2019, pp. 18–28.]
 - [18] [Li, Changsheng, et al. “A Direct Proof for Maxwell–Wagner Effect of Heterogeneous Interface.” *AIP Advances*, vol. 11, no. 6, 1 June 2021.]
 - [19] [Juarez-Martinez, Gabriela, et al. “Maxwell–Wagner Effect.” *Encyclopedia of Nanotechnology*, 2012, pp. 1276–1285.]
 - [20] Vijay, Sudarshan, et al. “Dipole-Field Interactions Determine the CO₂ Reduction Activity of 2D Fe–N–c Single-Atom Catalysts.” *ACS Catalysis*, vol. 10, no. 14, 5 June 2020, pp. 7826–7835.
 - [21] [Okubo, Masashi, et al. “MXene as a Charge Storage Host.” *Accounts of Chemical Research*, vol. 51, no. 3, 22 Feb. 2018, pp. 591–599.]
 - [22] [Bell, Christopher G., et al. “Theory of High Frequency, Large-Amplitude Sinusoidal Voltammetry for Ideal Surface-Confined Redox Systems.” *Electrochimica Acta*, vol. 56, no. 22, 6 July 2011, pp. 7569–7579.]
 - [23] [Frey, Nathan C., et al. “Surface-Engineered MXenes: Electric Field Control of Magnetism and Enhanced Magnetic Anisotropy.” *ACS Nano*, vol. 13, no. 3, 17 Jan. 2019, pp. 2831–2839.]

- [24] [Woo, Youngtai, et al. "Synthesis and Characterization of Sulfonated Polyimide Membranes for Direct Methanol Fuel Cell." *Journal of Membrane Science*, vol. 220, no. 1-2, Aug. 2003, pp. 31–45.]
- [25] [Tyan, Horng-Long, et al. "Mechanical Properties of Clay-Polyimide (BTDA-ODA) Nanocomposites via ODA-Modified Organoclay." *Journal of Polymer Science Part B Polymer Physics*, vol. 38, no. 22, 1 Jan. 2000, pp. 2873–2878.]
- [26] [Meador, Mary Ann B., et al. "Polyimide Aerogels with Amide Cross-Links: A Low-Cost Alternative for Mechanically Strong Polymer Aerogels." *ACS Applied Materials and Interfaces*, vol. 7, no. 2, 7 Jan. 2015, pp. 1240–1249.]
- [27] [Shi, Baolu, et al. "Fabrication and Applications of Polyimide Nano-Aerogels." *Composites Part A: Applied Science and Manufacturing*, vol. 143, 21 Jan. 2021.]
- [28] [Baker, Richard W. *Membrane Technology and Applications*. 15 Mar. 2004, p 16.]
- [29] [Xie, Xiao, et al. Large-Range Control of the Microstructures and Properties of Three-Dimensional Porous Graphene. Vol. 3, no. 1, 2 July 2013.]
- [30] [Zhu, Jiaying, et al. "Superelastic and Ultralight Aerogel Assembled from Hemp Microfibers." *Advanced Functional Materials*, vol. 33, no. 22, 14 Mar. 2023.]
- [31] [Zhong, Xian-Ci, et al. "Stress–Strain Hysteresis Loop-Based Machine Learning Models for Predicting Metal Fatigue Life under Uncertainty." *Materials*, vol. 18, no. 18, 16 Sept. 2025.]
- [32] [Phan, Hoang-Phuong, et al. "The Piezoresistive Effect of SiC for MEMS Sensors at High Temperatures: A Review." *Journal of Microelectromechanical Systems*, vol. 24, no. 6, 4 Sept. 2015.]
- [33] [Alhabeb, Mohamed, et al. "Guidelines for Synthesis and Processing of Two-Dimensional Titanium Carbide (Ti₃C₂T_x MXene)." *Chemistry of Materials*, vol. 29, no. 18, 6 Sept. 2017, pp. 7633–7644.]
- [34] [Murali, G., et al. "A Review on MXene Synthesis, Stability, and Photocatalytic Applications." *ACS Nano*, vol. 16, no. 9, 12 Sept. 2022, pp. 13370–13429.]
- [35] [Dobó, Zsolt, and Árpád Bence Palotás. "Impact of the Voltage Fluctuation of the Power Supply on the Efficiency of Alkaline Water Electrolysis." *International Journal of Hydrogen Energy*, vol. 41, no. 28, 27 July 2016.]
- [36] [Meador, Mary Ann B., et al. "Mechanically Strong, Flexible Polyimide Aerogels Cross-Linked with Aromatic Triamine." *ACS Applied Materials and*

- Interfaces, vol. 4, no. 2, 10 Jan. 2012, pp. 536–544.]
- [37] [LEE, K. “Toxicity of N-Methyl-2-Pyrrolidone (NMP): Teratogenic, Sub-chronic, and Two-Year Inhalation Studies.” *Fundamental and Applied Toxicology*, vol. 9, no. 2, Aug. 1987, pp. 222–235.]
- [38] [Yang, Wenke, et al. “Robust and Superelastic Spider Web-like Polyimide Fiber-Based Conductive Composite Aerogel for Extreme Temperature-Tolerant Linear Pressure Sensor.” *Science China Materials*, vol. 66, no. 7, 26 Apr. 2023, pp. 2829–2842.]
- [39] [Jia, Gaoen, et al. “Biomimetic Gradient Fibrous Aerogel Pressure Sensor Featuring Ultrawide Sensitive Range and Extraordinary Pressure Resolution for Machine Learning Enabled Posture Recognition.” *Advanced Fiber Materials*, vol. 7, no. 5, 18 June 2025, pp. 1632–1647.]
- [40] [Tafreshi, Omid Aghababaei, et al. “Flexible, Thermally Stable, and Ultralightweight Polyimide-CNT Aerogel Composite Films for Energy Storage Applications.” *ACS Applied Materials and Interfaces*, vol. 15, no. 43, 17 Oct. 2023.]
- [41] [Kawakami, Hiroyoshi, et al. “Gas Transport Properties in Thermally Cured Aromatic Polyimide Membranes.” *Journal of Membrane Science*, vol. 118, no. 2, Sept. 1996, pp. 223–230.]
- [42] [Tabata, M., et al. “Development of Silica Aerogel with Any Density.” *IEEE Nuclear Science Symposium Conference Record*, 2005, vol. 2, 2026, pp. 816–818.]
- [43] [Lan, Xiaolin, et al. “All-Ceramic SiC Aerogel for Wide Temperature Range Electromagnetic Wave Attenuation.” *ACS Applied Materials and Interfaces*, vol. 14, no. 13, 22 Mar. 2022.]
- [44] [Dong, Xinyu, et al. “Highly Robust and Hydrophobic Aerogel Beads with Dandelion-like Structure for Water Treatment.” *Chemical Engineering Journal*, vol. 456, 20 Dec. 2022.]
- [45] [Chen, Junyong, and Defang Pan. “Robust and Hydrophobic Silica/Polyimide Aerogel with Pomegranate-like Structure for Thermal Insulation and Flame Retardancy up to 1300 °C.” *Molecules*, vol. 30, no. 8, 11 Apr. 2025, p. 1709.]
- [46] [Zhao, Yi, et al. “Ultra-Sensitive and Durable Strain Sensor with Sandwich Structure and Excellent Anti-Interference Ability for Wearable Electronic Skins.” *Composites Science and Technology*, vol. 200, 1 Nov. 2020.]

- [47] [He, Song, et al. “Ultrasensitive and Self-Powered Multiparameter Pressure–Temperature–Humidity Sensor Based on Ultra-Flexible Conductive Silica Aerogel.” *Gels*, vol. 9, no. 2, 17 Feb. 2023, p. 162.]
- [48] [Nau, Robert. “What’s a Good Value for R-Squared?” Duke University, 2019, people.duke.edu/~rnau/rsquared.htm.]

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